TMS2003

132nd Annual International Meeting & Exhibition

The Minerals, Metals & Materials Society welcomes you to the TECHNICAL PROGRAM for the 132nd TMS Annual Meeting & Exhibition, to be held March 2–6, 2003, in San Diego, California.

This document comprises

THE COMPLETE TECHNICAL PROGRAM

Including fully text-searchable paper titles, abstracts, and author names with affiliations

For your convenience, we have also included details on

- Meeting Activities and Registration
- Conference Proceedings
- Our Exhibition
- TMS Membership
- Additional On-line Resources that You May Utilize

All designed to help you prepare for— and optimally benefit from— one of the world's premier metals and materials events.

See you in San Diego!

http://www.tms.org/AnnualMeeting.html

AN INTERNATIONAL EVENT IN SCIENCE AND ENGINEERING

During the week of March 2–6, the 2003 TMS Annual Meeting & Exhibition will host approximately 4,000 science and engineering professionals, representing more than 70 different countries. They are convening at the San Diego Convention Center and the San Diego Marriott Hotel & Marina to attend a field-spanning array of metals and materials symposia containing more than 200 sessions and 1,900 individual technical presentations.

This year's meeting will feature programming by

- TMS Electronic, Magnetic & Photonic Materials Division
- TMS Extraction & Processing Division
- TMS Light Metals Division
- TMS Materials Processing & Manufacturing Division
- TMS Structural Materials Division
- TMS Education Committee
- TMS Young Leaders Committee
- ASM International's Materials Science Critical Technologies Sector
- Aluminum Association
- International Magnesium Association

In addition to the technical programming featured on the following pages, attendees will have the opportunity to

- **Tour** the Exhibition of Approximately 200 Companies Displaying New Products and Services
- **Attend** Special Lectures and Tutorials
- **Participate** in Short Courses on Sulfide Smelting, Magnesium Metallurgy, Heat Treating Aluminum Alloys, Process Heating, Pumping Systems, and Computational Materials
- **Enjoy** Special Luncheons, Dinners, and Social Functions, including events honoring Ronald Armstrong, Michael Messhi, and Akira Yazawa
- **Network** Extensively
- **Experience** the Charm and Amenities of Family-Friendly San Diego

Extensive details about these and all conference-related activities can be found on the 2003 TMS Annual Meeting Web Site.

WANT TO BE PART OF THE ACTION?

Registration is easy.

Just complete and mail or fax the Annual Meeting Registration Form that appears in this document. Or, visit the meeting web site to register immediately (and securely) on-line.

To register in advance, your submission must reach TMS not later than **February 3, 2003.** After this date, it will be necessary to register at the meeting site.

The **San Diego Marriott Hotel & Marina** is the TMS headquarters hotel. Special conference rates have been contracted with this hotel and others in the area surrounding the **San Diego Convention Center.** To receive special rates, use the TMS 2003 Housing Reservation Form that appears in this document and that can be found on the meeting web site.

Special Opportunity for TMS Nonmember Registrants: All nonmember registrants automatically receive a one-year introductory associate membership in TMS for 2003. Membership benefits include a subscription to *JOM* (print and on-line versions) and significant discounts on TMS products and services.

More on the benefits of membership appears on the TMS Membership Web Pages.

INTERESTED IN BUSINESS OPPORTUNITIES?

The 2003 TMS Annual Meeting & Exhibition presents businesses, universities, institutions, agencies, consultants, and others with myriad opportunities to partner in effective marketing communication. Such opportunities to reach thousands of meeting attendees include:

- Placing a **Booth** in the Exhibition
- Placing an **Ad** in the Official Conference Publication and At-Meeting Program: *JOM*
- Sponsoring High-Profile **Attendee Services,** such as the CyberCenter, Coffee Breaks, Signage, and Prize Drawings.
- Hosting a **Hospitality Suite**

More information on these opportunities is available on the 2003 TMS Annual Meeting Sponsorship Web Pages.

CONFERENCE PROCEEDINGS: THE RECORDS OF EVENTS

The technical program of each TMS Annual Meeting yields numerous conference proceedings that document many presentations delivered in session rooms. Such publications can be ordered both before and after the meeting via the meeting registration form and/or the TMS Document Center.

The following symposium proceedings will be available in tandem with the meeting.

- ***Electron Microscopy: Its Role in Materials Science***: The Mike Meshii Symposium on Electron Microscopy
- ***EPD Congress 2003*** (Documenting the Symposia Global Development of Copper and Gold Deposits; Mercury Management; Recycling, General; Residue Handling in Metals Processing; Sensors and Control in Materials Processing; Waste from Metal Plating Industries)
- ***Friction Stir Welding and Processing II***
- ***High Temperature Alloys: Processing for Properties***
- ***Hot Deformation of Aluminum Alloys 2003***
- ***Light Metals 2003*** (Documenting the Symposia Alumina and Bauxite; Aluminum Reduction Technology; Carbon Technology; Cast Shop Technology; Reactive Metals; Recycling, Aluminum)
- ***Magnesium Technology 2003***
- ***Materials Lifetime Science and Engineering***
- ***Metallurgical and Materials Processing Principles and Technologies: The Yazawa International Symposium***
  - Volume 1: *Fundamentals and New Technologies*
  - Volume 2: *High Temperature Metal Production*
  - Volume 3: *Aqueous and Electrochemical Process*
- ***MPMD Fourth Global Innovations Symposium: Energy Efficient Manufacturing Processes***
- ***Surface Engineering in Materials Science II***

The following proceedings volumes will be released shortly after the meeting.

- ***Aluminum Reduction: Potroom Operations***
- ***Aluminum 2003*** (Documenting the Symposia Automotive Alloys 2003; Universities Servicing Education, Research, and Technology Internationally for the Aluminum and Light Metals Industries; Increasing Energy Efficiency in Aluminum)
- ***Gamma Titanium Aluminides 2003***

The following symposia will be documented in upcoming TMS periodicals.

- Advances in MEMS and Optical Packaging (*Journal of Electronic Materials*)
- Applications and Processing of Powder Metallurgy Refractory Metals and Alloys (*JOM*)
- Computational Methods in Materials Education (*JOM*)
- Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Prof. Ronald W. Armstrong (*Metallurgical and Materials Transactions A*)
- Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging (*Journal of Electronic Materials*)
- Materials and Processes for Submicron Technologies III (*Journal of Electronic Materials*)
- Phase Stability, Phase Transformations, and Reactive Phase Formation in Electronic Materials (*Journal of Electronic Materials*)
- Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties (*Metallurgical and Materials Transactions A*)

Detailed information about these publications, and many others, can be found in the TMS Document Center.

ADDITIONAL RESOURCES

On-line answers to any of your 2003 TMS Annual Meeting & Exhibition questions can be found at

- **2003 TMS Annual Meeting & Exhibition Web Site:** Get up-to-the-minute meeting details and complete registration materials at http://www.tms.org/AnnualMeeting.html
- **TMS Personal Conference Scheduler:** Review the most-up-to-date version of the technical program, examine the calendar of events, and create your own personalized itinerary by visiting http://pcs.tms.org
- **TMS Document Center:** Review the complete tables of contents for conference proceedings and order publications by visiting http://doc.tms.org
- **TMS Membership:** Learn more about the benefits of membership by touring http://www.tms.org/Society/membership.html
- **TMS Business-to-Business Partnering:** Learn how TMS can help your organization maximize its impact by viewing http://www.tms.org/Meetings/Annual-03/Annual03-exhibit.html

If you want to contact a person, more details are available at

TMS Meetings Department
The Minerals, Metals & Materials Society
184 Thorn Hill Road, Warrendale, PA 15086 USA
Telephone: (724) 776-9000, ext. 243
Fax: (724) 776-3770
E-mail: mtgserv@tms.org

TMS2003

ADVANCE REGISTRATION FORM

Advance Registration Deadline: February 3, 2003

Payment must accompany form.
Forms received past this date will be processed at the on-site fee.

AM03-TECH

WEB — http://www.tms.org
Web registration requires credit card payment.

FAX — USA: 724-776-3770
Fax registration requires credit card payment.

MAIL — **Return with payment to:** TMS, Meeting Services, 184 Thorn Hill Road, Warrendale, PA 15086

1. ☐ Dr. ☐ Prof. ☐ Mr. ☐ Mrs. ☐ Ms. Member of: ☐ TMS ☐ ISS ☐ SME ☐ SPE Member Number:________________

Ms. ______________________________________
Last Name First Name Middle Initial

Employer/Affiliation:______________________ Title:______________________

Address: ☐ Business ☐ Home ______________________________________

City:______________ State/Province:__________ Zip/Postal Code:__________ Country:__________

Telephone:______________ Fax:______________ E-Mail:______________

Guest/Spouse Name:______________________ *Guests do not receive admission to technical sessions.*

2. Registration Fees:

	Advance Fees until February 3, 2003		On-Site Fees after February 3, 2003	
☐ Member	$400	M	$500	ML
☐ Non-member Author*	$455	NMA	$555	NMAL
☐ Non-member *	$525	NM	$625	NML
☐ Student Member ##	$0	STU	$0	STUL
☐ Student Non-member ## *	$25	STUN	$25	STUNL
☐ TMS Senior Member	$225	RM	$225	RML
☐ Exhibit Booth Personnel	$0	E	$0	EL
☐ Exhibit Only	$35	EO	$35	EOL

Registration TOTAL $ ________

** Includes TMS membership for 2003*
Students must attach a copy of their school's student identification card.

3. Social Function Tickets:

	Event	Fee	Quantity	Total	
Monday 3/3/03	Michael Meshii Honorary Dinner	$60	____	$______	MD
	Akira Yazawa Honorary Dinner	$60	____	$______	YD
	Ronald Armstrong Honorary Dinner	$60	____	$______	RD
Tuesday 3/4/03	TMS Banquet	$60	____	$______	AD
	Tables of 8	$480	____	$______	AD8
	Table Sign to Read ______________				
	Extraction & Processing Division Luncheon	$30	____	$______	EP
	Tables of 8	$240	____	$______	EP8
	Table Sign to Read ______________				
Wednesday 3/5/03	Light Metals Division Luncheon	$30	____	$______	LM
	Tables of 8	$240	____	$______	LM8
	Table Sign to Read ______________				
	Structural Materials Division Luncheon	$30	____	$______	SM
	Tables of 8	$240	____	$______	SM8
	Table Sign to Read ______________				
	Gamma Titanium Aluminides Dinner	$60	____	$______	GTD

Social Function TOTAL $ ________

4. Tutorial Luncheon Tickets:

Monday 3/3/03	Fee	Quantity	Total	
The Young Leader Tutorial Lecture is free. *You may purchase the optional box lunch for*	$25	_____	$______	EM

5. Publication Orders: *All orders that are not indicated for shipment on this form must be picked-up at the meeting.*

Order Number	Title	Shipping Weight	Quantity	SubTotal Weight	Member Price	Non-member Price	Sub-Total Price
03-5352	Electron Microscopy (Meshii)	3	______	______	$96	$137	$ ________
03-5344	Metallurgical & Materials Processing (Yazawa)—Three-volume Set	11	______	______	$337	$538	$ ________
03-5468	Vol. 1: Fundamentals & New Technologies *(only)*	4	______	______	$154	$220	$ ________
03-5476	Vol. 2: High-Temp. Metal Production *(only)*	4	______	______	$154	$220	$ ________
03-5484	Vol. 3: Aqueous & Electrochemical Process *(only)*	3	______	______	$112	$159	$ ________
03-5328	EPD Congress 2003	3	______	______	$87	$123	$ ________
03-531X-G	Light Metals 2003 *(book and CD set)*	7	______	______	$158	$226	$ ________
03-5360	Friction Stir Welding and Processing II	3	______	______	$110	$157	$ ________
03-5336-G	Magnesium Technology 2003 *(book and CD set)*	3	______	______	$113	$161	$ ________
03-5395	High Temperature Alloys	2	______	______	$75	$107	$ ________
03-5387	Hot Deformation of Aluminum Alloys 2003	4	______	______	$123	$175	$ ________
03-5441	Materials Lifetime Science & Engineering	2	______	______	$91	$130	$ ________
03-5379	Surface Engineering in Materials Science II	2	______	______	$124	$177	$ ________

SubTotal $ ________

WEIGHT AND ZONE CHART

Weight	USA	Canada	Mexico	Western Europe	Eastern Europe	Australia, New Zealand	Central, South America	Pac. Rim, Asia	Middle East, India, Africa
1	$2.50	$4.00	$4.50	$4.00	$5.00	$4.50	$5.00	$5.00	$6.50
2	$3.00	$5.00	$9.00	$8.00	$10.00	$9.00	$10.00	$10.00	$13.00
3	$3.50	$6.00	$13.50	$12.00	$15.00	$13.50	$15.00	$15.00	$19.50
4	$4.00	$7.00	$18.00	$16.00	$20.00	$18.00	$20.00	$20.00	$26.00
5	$4.50	$8.00	$22.50	$20.00	$25.00	$22.50	$25.00	$25.00	$32.50
6	$5.00	$9.00	$27.00	$24.00	$30.00	$27.00	$30.00	$30.00	$39.00
7	$5.50	$10.00	$31.50	$28.00	$35.00	$31.50	$35.00	$35.00	$45.50
8	$6.00	$11.00	$36.00	$32.00	$40.00	$36.00	$40.00	$40.00	$52.00
9	$6.50	$12.00	$40.50	$36.00	$45.00	$40.50	$45.00	$45.00	$58.50
10	$7.00	$13.00	$45.00	$40.00	$50.00	$45.00	$50.00	$50.00	$65.00
11	$7.50	$14.00	$49.50	$44.00	$55.00	$49.50	$55.00	$55.00	$71.50
12	$8.00	$15.00	$54.00	$48.00	$60.00	$54.00	$60.00	$60.00	$78.00

If books are to be shipped, please complete the following.

Total Weight ______ Calculate shipping fees from the chart (at left) $ ________

$5 handling fee per order shipped. $ ________

NOTE: *If your order exceeds 12 pounds, add the amount that it is over from the chart (at the left) to reach the total weight of your order. [Example: 16 lbs. (delivered in U.S.A.) would be 12 lbs. ($8.00) + 4 lbs ($4.00) = 16 lbs. ($12.00)]*

Publications TOTAL $ ________

6. Continuing Education Short Courses: *Saturday & Sunday, March 1-2, 2003*

	Advance Fees until February 3, 2003 Member	Non-member	On-Site Fees after February 3, 2003 Member	Non-member
☐ 1. Sulfide Smelting: Principles, Technologies, and Environmental Considerations (Sat./Sun.)	$645	$735	$695	$785
☐ 2. Magnesium Metallurgy—Processing and Industrial Applications (Sat./Sun.)	$645	$735	$695	$785
☐ 3. Heat Treatment of Wrought and Cast Aluminum Alloys (Sat./Sun.)	$645	$735	$695	$785
☐ 4. Process Heating Workshop—Jt. TMS/DOE (Sat.)	$475	$560	$525	$610
☐ 5. Pumping Systems Assessment Workshop - Jt. TMS/DOE (Sun.)	$475	$560	$525	$610
☐ 6. Computational Materials Design (Sun.)	$475	$560	$525	$610

Short Course TOTAL $ ______ $ ______

7. 2003 Membership Dues: *For current TMS members only*

☐ Full Member	$90	FM
☐ Junior Member	$55	JM
☐ ASM/TMS Joint Student Member	$25	ST

8. Payment enclosed:

☐ Check, Bank Draft, Money Order

Make checks payable to TMS. Payment shall be made in US dollars drawn on a US bank.

☐ Credit Card Expiration Date: ______________________

Card No.: ______________________

☐ Visa ☐ MasterCard ☐ Diners Club ☐ American Express

Cardholder Name: ______________________

Signature: ______________________

9. TOTAL FEES PAID .... $__________

Refund policy: Written requests must be mailed to TMS, post-marked no later than February 3, 2003. A $50 processing fee will be charged for all cancellations. No refunds will be processed after February 3, 2003.

TMS2003

132nd Annual International Meeting & Exhibition
March 2-6, 2003 • San Diego, California, USA

HOUSING RESERVATION FORM

Mail or fax this housing form to:
Travel Planners, Inc., 381 Park Ave. South,New York, NY 10016
FAX: 212-779-6128 • PHONE: 800-221-3531, ext. 1
(in 212, 718, 516, 914, 631 or international call 212-532-1660, ext. 1)
(CHOOSE ONLY ONE OPTION)

Making your reservation is easier than ever through Travel Planners' real-time Internet reservation system! Just log on to **www.tms.org**, and follow the link to Travel Planners. You will be able to view actual availability, learn about your hotel's features and services, and obtain local city and sightseeing information. Most importantly, you will receive instant confirmation of your reservation!

Reservations must be received at Travel Planners by: Monday, February 3, 2003

Arrival Date ______________________ Departure Date ______________________

Last Name ______________________ First Name ______________________ MI __________

Company ______________________

Street ______________________ Address ______________________

City ______________ State/County ______________ Zip/Postal Code ______________ Country ______________

Daytime Phone ______________________ Fax ______________________

Additional Room Occupants ______________________

E-mail ______________________ (confirmation will be sent via e-mail if address is provided)

Non-Smoking Room Requested ______________________ Special Needs ______________________

Indicate 1st, 2nd, & 3rd hotel choice:

1. ______________________
2. ______________________
3. ______________________

Type of Accomodations: (check one)

☐ Single 1 person/1bed ☐ Double 2 people/1bed ☐ Twin 2 people/2 beds
☐ Triple 3 people/2 beds ☐ Quad 4 people/2 beds

If all three (3) requested hotels are unavailable, please process this reservation according to: (check one) ☐ ROOM RATE ☐ LOCATION

HEADQUARTERS

1. **San Diego Marriott Hotel & Marina**
$212/s/d • $232/bayview

2. **Clarion Hotel**
$125/single • $125/double

3. **Westgate Hotel**
$160/single • $160/double

4. **The Horton Grand Hotel**
$169/single • $169/double

5. **Hyatt Regency San Diego**
$239/single • $239/double

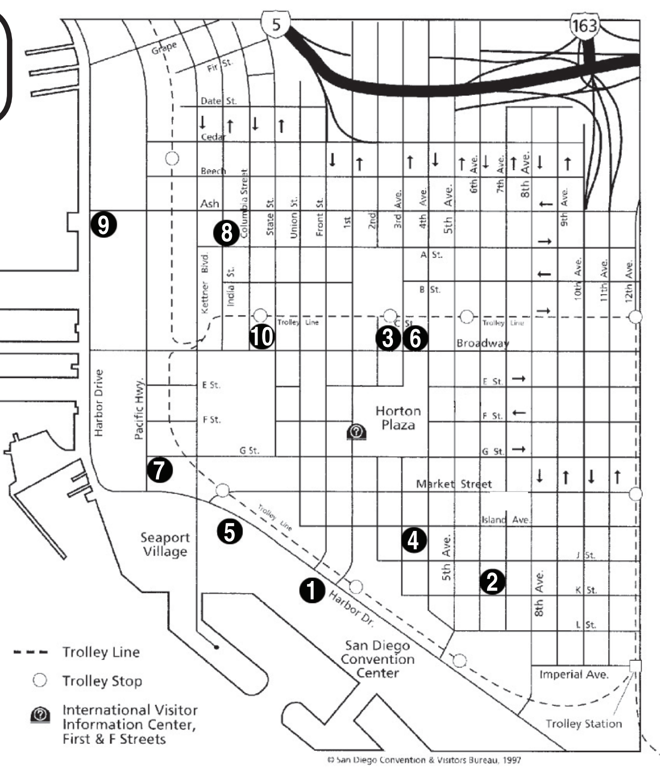
6. **U.S. Grant Hotel**
$179/single • $179/double

7. **Embassy Suites Hotel**
$190/single • $210/double

8. **Best Western Bayside Inn**
$124/single • $124/double

9. **Holiday Inn – on The Bay**
$165/single • $165/double

10. **Wyndham Emerald Plaza Hotel**
$179/single • $179/double



TMS has contracted a block of rooms at the headquarters hotel, San Diego Marriott Hotel & Marina, along with each of the hotels, and therefore has assumed a financial liability for any and all rooms in that block that are not reserved. You are strongly encouraged to reserve your room(s) at the hotels listed to limit our financial liability. Please help TMS achieve overall success with the 132nd TMS Annual Meeting & Exhibition by making your reservation at one of the listed hotels prior to the advance housing deadline. Thank you.

Confirmations: Confirmations will be e-mailed, faxed or mailed to you from Travel Planners, Inc. once your reservation has been secured with a deposit. You will not receive a confirmation from your hotel. If you do not receive a confirmation within 7 days, please call Travel Planners, Inc.

Changes/Cancellations: All changes and cancellations in hotel reservations must be made with Travel Planners, Inc. up until 3 business days prior to arrival and are subject to the individual hotel's cancellation policies. Cancellations and changes within 3 days of arrival MUST be made with your hotel directly. (If canceling or changing a room at the Hyatt after February 20, 2003, you must call the Hyatt directly.) Many hotels are now imposing fees for early departure. This rate is set by each hotel and may vary accordingly. Please reconfirm your departure date at the time of check-in.

Reservations/Deposits: All reservations are being coordinated by Travel Planners, Inc. Arrangements for housing must be made through Travel Planners, Inc. and NOT with the hotel directly. Reservations via Internet, phone or fax will be accepted with a major credit card only. Housing forms and written requests will be accepted with a major credit card or deposit of one night's room and tax payable to Travel Planners, Inc. Check must be drawn in US funds on a US bank. No wire transfers will be accepted. Deposit policies are set by each hotel, and are outlined on your hotel confirmation.

Please read all hotel information prior to completing and submitting this form to Travel Planners, Inc. Keep a copy of this form. Use one form per room required. Make additional copies if needed.

Deposit Payment: ☐ Check ☐ American Express ☐ MasterCard ☐ VISA ☐ Discover ☐ Diners

Account Number ______________________ Expiration Date ______________

Card Holder Name ______________________ Authorized Signature ______________________

Room	Monday-March 3 AM	Monday-March 3 PM	Tuesday-March 4 AM	Tuesday-March 4 PM	Wednesday-March 5 AM	Wednesday-March 5 PM	Thursday-March 6 AM
1A		Sensors and Control in Materials Processing - I	Materials Processing Fundamentals: Powder Synthesis and Processing	Materials Processing Fundamentals: Solidification and Forming Processes	Materials Processing Fundamentals: Heat and Fluid Flow: Modeling and Property Effects	Materials Processing Fundamentals: Thermodynamics and Reaction Kinetics	
1B	Waste from Metal Plating Industries - I	Residue Handling in Metals Processing - I	Global Development of Copper and Gold Deposits - I	Global Development of Copper and Gold Deposits - II	Mercury Management: Treatment Technologies	Mercury Management: Mining Operations	Mercury Management: Remediation and Fundamentals
2	Magnesium Technology 2003: Magnesium Melt Protection and Recycling	Magnesium Technology 2003: Magnesium Casting Properties	Magnesium Technology 2003: Magnesium Casting and Solidification and Simulations	Magnesium Technology 2003: Magnesium Casting Properties and Welding	Magnesium Technology 2003: Magnesium Alloy Development-Mechanical Properties - I	Magnesium Technology 2003: Magnesium Alloy Development-Mechanical Properties - II and Magnesium Wrought Alloys	Magnesium Technology 2003: Magnesium General Sessions
3	Alumina and Bauxite: Bayer Process Chemistry	Alumina and Bauxite: Precipitation and Alumina Products	Alumina and Bauxite: Red Mud Operations	Alumina and Bauxite: Bauxite and Alumina Extraction	Recycling - General Sessions: Aluminum	Recycling - General Sessions: General Recycling	
4		Computational Phase Transformations: Microstructural Evolution and Interdiffusion (parallel session)	Actinide Materials: Processing, Characterization, and Behavior: Plutonium	Actinide Materials: Processing, Characterization, and Behavior: Advanced Fuel Cycles I	Actinide Materials: Processing, Characterization, and Behavior: Advanced Fuels and Materials I	Actinide Materials: Processing, Characterization, and Behavior: Advanced Fuels and Materials II	
5A	The MPMD Fourth Global Innovations Symp: Energy Efficient Manufacturing Processes: I	The MPMD Fourth Global Innovations Symp: Energy Efficient Manufacturing Processes: II	General Abstracts III: Smelting, Melting, and Effluent Control	The MPMD Fourth Global Innovations Symp: Energy Efficient Manufacturing Processes: III	Products, Applications, and Services Showcase: Furnaces & Casting Technology	Products, Applications, and Services Showcase: Reduction Technology & Laboratory Analysis	
5B	Automotive Alloys 2003 - I	Automotive Alloys 2003 - II	Increasing Energy Efficiency in Aluminum	Universities Servicing Education, Research and Technology Internationally for the Aluminium and Light Metals Industries - I	Aluminum Reduction Technology: Fundamentals (parallel session)	Materials Research to Meet 21st Century Defense Needs	
6B	Aluminum Reduction–Potroom Operations Symposium: Potline Improvements	Aluminum Reduction–Potroom Operations Symposium II/ Aluminum Reduction Technology: Environmental - General	Aluminum Reduction Technology: Environmental - HF & PFC	Aluminum Reduction Technology: Modeling	Aluminum Reduction Technology: Modernization	Aluminum Reduction Technology: Advanced Processes	Aluminum Reduction Technology: Pot Control
6C	Cast Shop Technology: Cast Shop Safety	Cast Shop Technology: Continuous & DC Casting	Cast Shop Technology: DC Casting II	Cast Shop Technology: Filtration/Metal Treatment	Cast Shop Technology: Grain Refining	Cast Shop Technology: Solidification and Foundry Technology	Cast Shop Technology: Foundry Technology II
6D	Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Plenary	Cast Shop Technology: Melting and Alloying (parallel session)	Carbon Technology - I	Carbon Technology - II	Carbon Technology - III	Carbon Technology - IV	
6E	Hot Deformation of Aluminum Alloys: Deformation, Recrystallization and Texture - I	Hot Deformation of Aluminum Alloys: Deformation, Recrystallization and Texture - II	Hot Deformation of Aluminum Alloys: Constitutive Analysis, Modeling and Simulation	Hot Deformation of Aluminum Alloys: Superplasticity and Creep	Hot Deformation of Aluminum Alloys: Processing, Structure and Property - I	Hot Deformation of Aluminum Alloys: Processing, Structure and Property - II	Hot Deformation of Aluminum Alloys: Processing, Structure and Property - III
6F	International Symposium on Gamma Titanium Aluminides: Application	International Symposium on Gamma Titanium Aluminides: Fundamentals	International Symposium on Gamma Titanium Aluminides: Fatigue and Fracture	International Symposium on Gamma Titanium Aluminides: Environmental Effects and Applications	International Symposium on Gamma Titanium Aluminides: Processing - IM, PM, and Sheet	International Symposium on Gamma Titanium Aluminides: High Temperature Properties	International Symposium on Gamma Titanium Aluminides: Advanced Alloy Design

	Monday-March 3 AM	Monday-March 3 PM	Tuesday-March 4 AM	Tuesday-March 4 PM	Wednesday-March 5 AM	Wednesday-March 5 PM	Thursday-March 6 AM
7A	Surface Engineering in Materials Science-II: Nanotechnology and Property Evaluation	Surface Engineering in Materials Science-II: Nanotechnology and Property Evaluation II	Surface Engineering in Materials Science-II: Surface Engineering and Modifaction	Surface Engineering in Materials Science-II: Coatings and Properties	Surface Engineering in Materials Science-II: Manufacturing via Surface Engineering	Surface Engineering in Materials Science-II: High Temperature Materials and Surface Alteration	
7B	Martensitic Transformations in Low Symmetry Materials - I	Martensitic Transformations in Low Symmetry Materials II	Friction Stir Welding and Processing-II: Friction Stir Joining: Process Models	Friction Stir Welding and Processing-II: Friction Stir Joining: Corrosion & Corrosion Fatigue	Friction Stir Welding and Processing-II: Friction Stir Joining: Structures & NDE	Friction Stir Welding and Processing-II: Friction Stir Joining: Mechanical Properties	Friction Stir Welding and Processing-II: Friction Stir Processing
8	International Symposium on Intermetallic and Advanced Metallic Materials: C.T. Liu Symposium I	International Symposium on Intermetallic and Advanced Metallic Materials: C.T. Liu Symposium II	International Symposium on Intermetallic and Advanced Metallic Materials: C.T. Liu Symposium III	International Symposium on Intermetallic and Advanced Metallic Materials: C.T. Liu Symposium IV	International Symposium on Intermetallic and Advanced Metallic Materials: C.T. Liu Symposium V	International Symposium on Intermetallic and Advanced Metallic Materials: C.T. Liu Symposium VI	International Symposium on Intermetallic and Advanced Metallic Materials: C.T. Liu Symposium VII
9	The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research I	The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research II	The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research III	The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research IV	The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research V	The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research VI	
10	Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties I	Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties II	Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties III	Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties IV	15th International Symposium on Experimental Methods for Microgravity Materials Science I	15th International Symposium on Experimental Methods for Microgravity Materials Science II	15th International Symposium on Experimental Methods for Microgravity Materials Science III
11A	Microstructural Processes in Irradiated Materials: Advanced Experimental Techniques I	Microstructural Processes in Irradiated Materials: Advanced Experimental Techniques II	Microstructural Processes in Irradiated Materials: Multiscale Modeling I	Microstructural Processes in Irradiated Materials: Multiscale Modeling II	Microstructural Processes in Irradiated Materials V	Microstructural Processes in Irradiated Materials VI	Microstructural Processes in Irradiated Materials: Fusion Reactor Materials
11B	Computational Phase Transformations: Atomistic Modeling I - Phase Stability	Computational Phase Transformations: Atomistic Modeling II - Diffusion, Elastic Property and Crystal Growth	Computational Phase Transformations: Fundamental Properties of Surfaces and Interfaces	Computational Phase Transformations: Effect of Interface on Phase Transformation	Computational Phase Transformations: Effect of Internal Defects and External Fields	Computational Phase Transformations: Solidification and Crystallization	Computational Phase Transformations: Engineering Applications
12	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - I	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - II	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - III	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - IV	Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - V		
13	Computational Methods in Materials Education: Insertion of Computational Methods - I	Computational Methods in Materials Education: Insertion of Computational Methods II	Computational Methods in Materials Education: New Computational Courses	Computational Methods in Materials Education: Market Perspectives and Teaching Materials	High Temperature Alloys: Processing for Properties: Wrought Alloys	High Temperature Alloys: Processing for Properties: Cast Alloys and Machining	High Temperature Alloys: Processing for Properties: Alternate Materials
14A	Materials Processing Under the Influence of Electrical and Magnetic Fields - I	Materials Processing Under the Influence of Electrical and Magnetic Fields - II	Materials Processing Under the Influence of Electrical and Magnetic Fields - III	Materials Processing Under the Influence of Electrical and Magnetic Fields - IV	Materials Processing Under the Influence of Electrical and Magnetic Fields - V	Materials Processing Under the Influence of Electrical and Magnetic Fields - VI	
14B	International Symposium on Structures & Properties of Nanocrystalline Materials: Theory and Simulations	International Symposium on Structures & Properties of Nanocrystalline Materials: Mechanical Properties I	International Symposium on Structures & Properties of Nanocrystalline Materials: Mechanical Properties II	International Symposium on Structures & Properties of Nanocrystalline Materials: Magnetic and other Functional Properties	International Symposium on Structures & Properties of Nanocrystalline Materials: Microstructure and Properties	International Symposium on Structures & Properties of Nanocrystalline Materials: Phase Transformation	
15A	Materials and Processes for Submicron Technologies-III: I	Materials and Processes for Submicron Technologies-III II	Science and Technology of Magnetic and Electronic Nanostructures: Bionanoelectronics	Science and Technology of Magnetic and Electronic Nanostructures: Semiconductor Nanostructures	Science and Technology of Magnetic and Electronic Nanostructures: Nanostructures in Functional Materials: Magnetic	Science and Technology of Magnetic and Electronic Nanostructures: Functional Nanostructures: Ferroic Materials	Science and Technology of Magnetic and Electronic Nanostructures: Nanoscale Probes

Monday-March 3		Tuesday-March 4		Wednesday-March 5		Thursday-March 6	
AM	PM	AM	PM	AM	PM	AM	
Advances in MEMS and Optical Packaging - I	Advances in MEMS and Optical Packaging - II	Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging I	Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging II	Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging III	Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging IV	Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging V	15B
Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction I	Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction II	Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction III	Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction IV	Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction V	Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction VI		16A
Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong I	Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong II	Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong III	Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong IV	Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong V	Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong VI		16B
Defects and Deformation of Crystalline Solids–in Honor of Dr. Man H. Yoo - I	Defects and Deformation of Crystalline Solids–in Honor of Dr. Man H. Yoo - II	Defects and Deformation of Crystalline Solids–in Honor of Dr. Man H. Yoo - III	Defects and Deformation of Crystalline Solids–in Honor of Dr. Man H. Yoo - IV	Defects and Deformation of Crystalline Solids–in Honor of Dr. Man H. Yoo - V	Defects and Deformation of Crystalline Solids–in Honor of Dr. Man H. Yoo - VI		17A
Measurement and Interpretation of Internal/Residual Stresses: Polycrystalline Deformation Modes: Expt and Model	Measurement and Interpretation of Internal/Residual Stresses: Deformation Modes Part II	Measurement and Interpretation of Internal/Residual Stresses: Welding	Measurement and Interpretation of Internal/Residual Stresses: Processing	Measurement and Interpretation of Internal/Residual Stresses: Neutron and SXRD	Measurement and Interpretation of Internal/Residual Stresses: Composites	Measurement and Interpretation of Internal/Residual Stresses: SXRD on Composites	17B
Applications and Processing of Powder Metallurgy Refractory Metals and Alloys: Molybenum Base Alloys and Modeliing	Applications and Processing of Powder Metallurgy Refractory Metals and Alloys: Tungsten and Rhenium Base Alloys	Materials Lifetime Science and Engineering - I	Materials Lifetime Science and Engineering - II	Materials Lifetime Science and Engineering - III	Materials Lifetime Science and Engineering - IV		18
	General Abstracts I Nonferrous Alloys and Other Materials		General Abstracts II Steels and Non-Metals		General Abstracts IV Fatigue, Fracture, and Plastic Deformation		19
	Yazawa International Symposium– Kinetics & Thermodynamics	Yazawa International Symposium– Metals & Sulfide Systems	Yazawa International Symposium– Slags & Fluxes	Yazawa International Symposium– Elemental Losses & Distributions	Yazawa International Symposium– Alloy Properties	Yazawa International Symposium– Experimental Techniques	Solana
	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: General - I	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: General - II	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: General - III	Yazawa International Symposium– Advances in Materials Processing Technologies: General - I	Yazawa International Symposium– Advances in Materials Processing Technologies: General - II	Yazawa International Symposium– Advances in Materials Processing Technologies - III: Casting & Aluminium Processing	Point Loma
Marriott Hotel	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: Nickel	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: Lead-Zinc	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: Copper - I	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: Copper - II	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: Copper - III	Yazawa International Symposium– Advances in Non-Ferrous Production Technologies & Industrial Practice: Copper - IV	Santa Rosa
	Yazawa International Symposium– Recycling, Waste Treatment & Environmental Issues I	Yazawa International Symposium– Recycling, Waste Treatment & Environmental Issues II	Yazawa International Symposium– Recycling, Waste Treatment & Environmental Issues III	Yazawa International Symposium– Iron & Steel Making Fundamentals - I	Yazawa International Symposium– Iron & Steel Making Fundamentals - II	Yazawa International Symposium– Iron & Steel Making Fundamentals - III and Applications	Leucadia
	Yazawa International Symposium– Aqueous & Electrochemical Processing - I	Yazawa International Symposium– Aqueous & Electrochemical Processing - II	Yazawa International Symposium– Aqueous & Electrochemical Processing - III	Yazawa International Symposium– Aqueous & Electrochemical Processing - IV	Yazawa International Symposium– Aqueous & Electrochemical Processing - V		Pacific

Table of Contents

TECHNICAL PROGRAM

San Diego Convention Center; San Diego, California USA; March 2-6, 2003

MONDAY AM

Advances in MEMS and Optical Packaging - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Sungho Jin, University of California-San Diego, Department of Mechanical & Aerospace Engineering, La Jolla, CA 92093-0411 USA; Darrel R. Frear, Motorola, Tempe, AZ 85284 USA; Andrei M. Shkel, University of California-Irvine, Department of Mechanical & Aerospace Engineering, Irvine, CA 92697-3975 USA; Martin Weiser, Honeywell Electronics Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Monday AM Room: 15B
March 3, 2003 Location: San Diego Convention Center

Session Chairs: Sungho Jin, University of California-San Diego, Dept. of Mechl. & Aeros. Eng., La Jolla, CA 92093-0411 USA; Andrei Shkel, University of California-Irvine, Dept. of Mechl. & Aeros. Eng., Irvine, CA 92697-3975 USA

8:30 AM Invited
Atomic Layer Deposition-Based Packaging for Reliable MEMS: *Yung-Cheng Lee*[1]; [1]University of Colorado, Dept. of Mechl. Eng., Boulder, CO 80309-0427 USA

The number of exciting microelectromechanical devices demonstrated each year is growing exponentially. However, the reliability concerns severely limit MEMS insertion into systems today. The reliability of moving micro-sensors and micro-actuators is degraded by nano-scale interface failure mechanisms whenever there are surface-to-surface contacts. The failures are strongly affected by the contact modes and materials, and the effects can be changed significantly if there are minute environmental variations due to particles, charges, and moistures. Fortunately, with the advancement of nano-technologies, we may be able to design and fabricate nano-scale protective coatings to assure MEMS reliability. One of such technologies is atomic layer deposition (ALD). ALD has been applied to coat thin alumina layer to protect MEMS from electrical shorts. In addition, the alumina layers can be mixed with ZnO layers for a coating with specified electrical conductivities. Such a nano-scale, electrically conductive composite may solve the charge-induced reliability problem. This paper will discuss ALD-based packaging and its effects on RF and optical MEMS.

9:00 AM Invited
Organic Versus Inorganic MEMS Actuators: *Marc Madou*[1]; [1]University of California-Irvine, Dept. of Mechl. & Aeros. Eng., 4200 Eng. Gateway Bldg., Irvine, CA 92697-3975 USA

Pros and cons of MEMS actuators based on organic and inorganic materials are presented. Evaluation criteria include: power consumption, force exerted, durability, cost, biocompatibility and percentage of expansion/contraction upon cycling of the actuators. One application for both type of actuators that will be detailed is that of an in-vivo responsive drug delivery system.

9:30 AM Invited
Thermal Bonding Processes for MEMS Packaging Applications: *Liwei Lin*[1]; [1]University of California-Berkeley, Dept. of Mechl. Eng., Berkeley Sensor & Actuator Ctr., 5126 Etcheverry, Berkeley, CA 94720-1740 USA

MEMS packaging has become a major manufacturing issue for commercialization. In the past twenty years, the application of microelectronic technology to the fabrication of mechanical devices stimulated emerging research in semi-conductor microsensors and microactuators. However, research on MEMS packaging issues have been generally neglected. This talk will introduce several thermal-bonding processes for MEMS packaging in an effort to address the MEMS packaging issues. These include an integrated LPCVD nitride bonding process; localized eutectic, fusion and solder bonding processes; RTP (rapid thermal processing) bonding processes; nano-second laser welding process; ultrasonic sealing process; localized CVD sealing process and low-temperature solder bonding processes. Specific issues in MEMS packaging applications will also be discussed, including vacuum encapsulation, accelerated testing and long-term reliability of the encapsulated MEMS devices.

10:00 AM Invited
Micro-Structures with Error-Suppression and Self-Calibration Control Capabilities: *Andrei M. Shkel*[1]; [1]University of California-Irvine, Dept. of Mechl. & Aeros. Eng., 4200 Eng. Gateway Bldg., Irvine, CA 92697-3975 USA

Micro-sensors and actuators are differentiated from conventional sensors and actuators by their size, cost, and techniques used in their manufacture. The small size of MEMS is a convenience, a contribution to their potentially low cost, and a source of new conceptual challenges. Improvements in sensing structures, process technology, and circuit technology are presently addressed by the MEMS research community. Unavoidable, the next logical step in the MEMS evolution will be development of precision micro-systems by integrating a number of smart control functions including self-calibration, self-testing, structural compensation, and even communication with one or more other sensors located on the same chip. This paper gives an overview of functional blocks needed for Smart MEMS and provides several examples highlighting benefits of the paradigm.

10:30 AM Break

10:50 AM Invited
Bonding-Curvature and Charge Dissipation Issues in MEMS and Optical Packaging: *Sungho Jin*[1]; [1]University of California-San Diego, Dept. of Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92130-0411 USA

In the packaging of MEMS, optical and electronic devices, there is a need to directly bond a wide variety of inorganic materials such as oxides, nitrides, carbides, fluorides, diamond, and semiconductors. Such applications involve hermetic sealing components, three-dimensional MEMS assembly components as well as active semiconductor or optical components, dielectric layers, diffusion barriers, wave guides, and heat sinks. These materials are known to be very difficult to wet and bond with low melting point solders. In this presentation, new Sn-Ag or Au-Sn based universal solders doped with a small amount of rare-earth elements which now allow direct and powerful bonding onto the surfaces of various MEMS, optical or electronic device materials will be described. Various packaging-related structural or electrical aspects in MEMS, such as the control/maintenance of membrane curvature and charge dissipation will also be discussed.

11:20 AM
Curvature Correction Mechanism in MEMS Micro-Mirror Structures Using Ion-Beam Modification of Residual Stress: *Harley T. Johnson*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mechl. & Industl. Eng., 140 Mechl. Eng. Bldg., MC-244, 1206 W. Green St., Urbana, IL 61801 USA

Residual stress causes significant problems in the design and manufacture of free standing thin film MEMS devices. In free standing films, through thickness stress may lead to large out-of-plane curvature which is particularly problematic for optical MEMS mirrors used to reflect light. In the work described here, a new experimental post-processing method is used to reduce and repair curvature in free standing thin film micro-mirrors.[1] The method uses ion-beam induced damage and residual stress to offset the effects of process induced residual stress. A combined continuum and molecular dynamics analysis is used here to explain the mechanics of the process. The analysis shows that stress caused by ion-beam amorphization at the surface of the film reduces the curvature of the structure. At the continuum level, the model is based on the familiar approach in thin film mechanics relating in-plane mismatch stress to wafer curvature.[2] The mismatch stress distribution here is nonuniform and not well understood a priori, however, so the continuum analysis goes beyond the standard Stoneyís equation approach. Details of the ion induced stress field are found first through molecular dynamics simulations similar to previous work in the area of ion implantation modeling.[3] The molecular dynamics simulations model the transient process by which individual incident ions penetrate a crystalline Silicon thin film and create defects and damage. A statistical analysis of the resulting equilibrium microstructure leads to an estimate of the through thickness residual stress distribution on a cross section of ion-machined film. The computed stress distribution is then used in the continuum model to compute the resulting curvature change in the structure. Curvature change estimates are in reasonable agreement with experimentally observed values. The results show that MEMS structures with thicknesses in the micron range are accurately described using a multiscale approach, but within the framework of standard thin film mechanics theory. [1]T. G. Bifano, H. T. Johnson, P. Bierden, and R. Mali (2002), ìElimination of stress-induced curvature in thin-film structures,î to appear in J. MEMS. [2]L. B. Freund (1996), ìSome elementary connections between curvature and mismatch strain in compositionally graded thin films,î J. Mech. Phys. Sol. 44, 723. [3]T. Diaz de la Rubia and G. H. Gilmer (1995), ìStructural transformations and defect production in ion implanted silicon: A molecular dynamics simulation study,î Phys. Rev. Lett. 74, 2507.

11:40 AM

Materials Reliability and Packaging in a Piezoelectric MEMS Microengine: *D. F. Bahr*[1]; A. L. Olson[1]; L. M.R. Eakins[1]; M. S. Kennedy[1]; J. M. Kayner[1]; C. D. Richards[1]; R. F. Richards[1]; [1]Washington State University, Mech. & Matls. Eng., PO Box 642920, Pullman, WA 99164-2920 USA

A recently developed thermal microengine relies upon a flexing piezoelectric film to convert mechanical to electrical power. The mechanical work is provided via a novel thermodynamic cycle which approaches a Carnot cycle, and is based on the expansion of a liquid-vapor fluid when heat is cycled in the system. Each engine works over a small temperature difference; multiple wafers will need to be combined to increase the useful temperature range of the device. The reliability of the flexing piezoelectric film will be examined as a function of processing conditions, particularly the strength and adhesion of the piezoelectric film during both processing and operation. The ability to control fluid in the engine cavity through chemical modification of the surface as well as patterning capillary structures will be discussed. The issues surrounding materials compatibility for multiple die packages will be explored for this application.

Alumina and Bauxite: Bayer Process Chemistry

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: David Kirkpatrick, Kaiser Aluminum & Chemical Group, Gramercy, LA 70052-3370 USA; Steve Rosenberg, Worsley Alumina Pty, Ltd., Process Chemistry Group, Collie, West Australia 6225 Australia; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday AM — Room: 3
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: David Kirkpatrick, Kaiser Aluminum & Chemical Group, Gramercy, LA 70052-3370 USA; Steve Rosenberg, Worsley Alumina Pty Ltd., Process Chem. Grp., Collie, W. Australia 6225 Australia

8:30 AM

Experimental Study of the Dissolution of Aluminum Phases as a Function of Temperature, Caustic Concentration and Additives: *Donald A. Palmer*[1]; Pascale Benezeth[1]; David J. Wesolowski[1]; Sacha Hilic[1]; [1]Oak Ridge National Laboratory, Cheml. Scis., PO Box 2008, Bldg. 4500S, Oak Ridge, TN 37831-6110 USA

The kinetics of dissolution and equilibrium solubilities of gibbsite, boehmite, bayerite and sodalites were studied from 30 to 90∞C as functions of caustic concentration and ionic strength (sodium nitrate) in the absence and presence of triethanolamine and other additives. These results build on our existing experimental database of boehmite solubility to 300∞C and gibbsite solubility to 80∞C in sodium hydroxide and chloride solutions. The solubility constants are treated with consistent models that include appropriate data in the literature for prediction of solubilities over wide ranges of state conditions. The solubility enhancing effect (kinetics and equilibrium) of triethanolamine were also established and quantified. A new experimental approach for studying the kinetics of dissolution and precipitation of gibbsite and boehmite is also presented.

8:55 AM

The Manufacture of Tricalcium Aluminate: *Lawrence Joseph Andermann*[1]; Geoffrey Joseph Pollet[1]; [1]Kaiser Aluminum and Chemical Corporation, Commodities Techl. Services, 1111 Airline Hwy. 61, Gramercy, LA 70052 USA

In the Bayer Process, production of alumina is limited by the rate of liquor passing through Kelly Presses. Kelly Presses are utilized for secondary clarification, and filter clothes in combination with Tricalcium Aluminate (TCA) are used to trap solids after the initial clarification in gravity settlers. Desirable qualities of TCA are TCAís that allow fast liquor flow rates and to filter solids to low concentrations. Inconsistent performance of Kelly Presses are not fully understood, so this paper offers some explanation on how physical and chemical conditions in TCA production govern desirable properties. Slaking temperature, lime to alumina ratio, and caustic concentration affect the TCAís particle size distribution, crystal structure, and composition. TCAís particle size distribution, crystal structure, and composition are thought to affect the flux of liquor through Kelly Presses. Controlling slaking conditions of TCA, Kaiserís Gramercy Works has reduced the number of presses needed per day by 33%.

9:20 AM

Overview of the Behavior of Sodium Oxalate in Bayer Liquor and its Effect of the Process: *Myong Jun Kim*[1]; Seong Oh Lee[2]; [1]Chonnam National University, Dept. of Resources & Energy Eng., 300 Puk-Ku, Yong-bong-Dong, Kwanju 500-757 Korea; [2]Korea Chemical Company, Ltd., 1696-1, Nanjun-ri, Samho-myun, Youngam-gun, Chonnam Korea

Under digestion condition of the Bayer process, the organic matter is degraded into numerous low molecular weight species. Some are insoluble and go out with the mud, and some are soluble and remain in the circuit. The soluble species are generally compounds with carboxylic, phenolic and carboxylic-phenolic functional groups, that readily react with sodium hydroxide to give their respective sodium salts. The ultimate degredation products are sodium carbonate and sodium oxalate. It has been known for a long time that soudium oxalate can interfere with the Bayer process, but the nature and the size of the compound on the process are not clear, hence the numerous publication on the matter. This paper presents an overview of the numerous published reports in the literature on the behaviour of sodium oxalate in Bayer liquor and its effect on the process, based on plant data as well as laboratory experiments.

9:45 AM

Sodium Aluminosilicate Scale Formation on Steel Substrates: Experimental Design and Assessment of Fouling Behaviour: *J. Addai-Mensah*[1]; R. Jones[1]; M. Zbik[1]; A. Gerson[1]; [1]University of South Australia, Ian Wark Rsrch. Inst., Mawson Lakes, Adelaide SA 5095 Australia

The fouling of heat transfer equipment as a result of sodium aluminosilicate precipitation from SiO2-containing caustic aluminate liquors is a troublesome problem confronting alumina refining, nuclear waste treatment and pulp and paper industries. Recourse to meaningful, well-planned laboratory studies is necessary in producing reliable information required for the formulation of fouling mitigation strategies. Experimental design and assessment of the mimicking systems must be carried out to ensure good reproducibility, process/interfacial chemistry and geometric similarity with real systems, and address the hydrodynamics issues involved. In this paper, investigations carried out to establish a reliable methodology for the analysis and assessment of isothermal, batch precipitation fouling behaviour of sodium aluminosilicate at 316 stainless steel surfaces under turbulent flow conditions are reported. Studies performed using optically clear, self-nucleating liquors at 180°C in 0.6 dm3 and 4 dm3 autoclaves highlight the pivotal role of hydrodynamics and time on scale microstructure and coverage.

10:10 AM Break

10:20 AM

Effects of Temperature and Method of Solution Preparation on the Performance of a Typical Red Mud Flocculant: *Pierre Ferland*[1]; John T. Malito[2]; Everett C. Phillips[2]; [1]Alcan International Ltd., Arvida Rsrch. Dvlp. Ctr., Jonquiere, QC G7S 4K8 Canada; [2]Ondeo Nalco Company, Techl. Ctr., Naperville, IL 60563-1198 USA

Alcan International Ltd. in collaboration with Ondeo Nalco R&D have carried out a fundamental study on the dissolution and performance of a 100% anionic polymer. The effects of methods of preparation, solvent composition, temperature and time on flocculent activity under conditions relevant to both atmospheric and pressure decantation were investigated. Flocculent activity was determined using static and dynamic settling tests, and the results were correlated with the reduced specific viscosity (RSV). For any given method of preparation of the flocculent solutions (makeup/dilution) the RSV tended to decrease with increasing solution ionic strength, independent of ionic speciation. While a significant loss in flocculent activity occurred with long exposure of the solution to high temperature, only a minor loss occurred in the short time required to flocculate and settle the mud in a decanter operating at 150°C. Recent results in an actual plant pressure decanter appear to validate this conclusion.

10:45 AM

Mixer Design Optimization for High Solids Contents Media: Methodology and Application to the PECHINEYís High Density Desilication Process: *Florent Bouquet*[1]; [1]Robin Industries, Hydraulic Eng. Dept., 10 Rue du Bois Gasseau, BP 94 Samoreau, Avon, Cedex 77212 France

The most obvious aim of a mixer in slurries exhibiting high solids concentrations (around 1000 g/l) is to prevent the solids from settling at the bottom of the tank. Nevertheless, depending on process requirements, the mixer may also have to be able to keep the solids concentration homogeneous all over the height of the tank, to prevent short-circuits in the tank, to facilitate the slurry transfer from one tank to another in case of continuous processes, to achieve sufficient heat transfer performances? The list is quite long. This paper points out the important parameters to take into account for the mixer design and presents a methodology for defining an optimized mixing system for high solid contents slurries. An application of this method to PECHINEYís high density desilication process will be described through industrial examples.

11:10 AM

The Application of the Theory on Systematic Energy-Saving in the Process of Alumina Production: *Qi Lijuan*[1]; [1]China Aluminium Corporation, Zhengzhou Rsrch. Dept., Zhengzhou, Henan 450041 China

Procedure in the Alumina production is a long one that bears many processes. Traditonal energy-saving is based on the single equipment, the result of which is limited. It is meaningful that energy-saving is studied from the view point of the whole production system, especially in the production which has many and complicated processes, like alumina production. In this essay the application of the method of systematic energy-saving in the process of alumina production is deeply studied.

11:35 AM

Water Conservation in Alumina Refinery Plant: *B. K. Padhi*[1]; B. Toppo[1]; P. Vidyasagar[1]; [1]National Aluminium Company, Ltd., QC Lab., Damanjodi, Orissa 763008 India

The conservation of water is an important issue now days. To minimize the wastage of water various steps are being taken worldwide. During production of alumina from bauxite, using dilute sodium aluminate liquor and steam is required. After removal of precipitated aluminium hydroxide crystals from the sodium aluminate solution, it is then concentrated in multiple effect evaporators. Water vapors so generated are collected at about 90°C. This water is alkaline in nature with pH value in the range of 9.5-10.5 and the conductivity is of the order of 30-70 micro siemen/cm along with 30-40ppm of sodium and 4-6ppm of aluminium ions. This is due to combination of otherwise pure condensate by tiny droplets of process liquor carried forward in evaporation under vacuum conditions. Out of total generation of 380M3/hr condensate about 230M3 is used for process dilution/washing in plant and 150M3/hr has to be utilized for some other purpose or disposed off. No such activity has been initiated by any alumina industry for reutilization purpose for boiler use. The present studies are mainly emphasizing the reutilization of 150M3 dumped wastewater for boiler use, after ion exchanging by passing through suitable resins using dihydroxy and trihydroxy weak organic acids as complexing agent at 40-45°C. The exchange of aluminium and sodium by the cation resins and organic anions by anion resins were studied by using FTIR spectroscopy. The quantitative analysis of aluminium was carried out by using capillary Ion analyzer (electrophoresis method). The outlet water was having the pH 7.0 and the conductivity of 0.2 micro siemen/cm, which is suitable for high-pressure boiler feed water. The mechanism of complex formation of aluminium with dihydroxy and trihydroxy acid and exchange of aluminium complex by cation resins and energy conservation by utilizing preheated water for boiler use have been discussed in the present paper.

Aluminum Reduction–Potroom Operations Symposium: Potline Improvements

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Alton T. Tabereaux, Alcoa Inc., Process Technology, Muscle Shoals, AL 35661 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday AM — Room: 6B
March 3, 2003 — Location: San Diego Convention Center

Session Chair: Alton T. Tabereaux, Alcoa Inc., Process Tech., Muscle Shoals, AL 35661 USA

8:30 AM

Century Aluminumís Experience with Initial Operation of a New PotlineñTeamwork Resulting in Continuous Improvement: *Thomas Ray Alcorn*[1]; Joseph E. Brown[1]; Donald Wayne Shelman[1]; [1]Century Aluminum of Kentucky, 1627 State Rte. 271 N., PO Box 500, Hawesville, KY 42348 USA

A Fifth Potline was started at the Century Aluminum Hawesville, Kentucky plant during 1999. During this time the plant was in the midst of a strike as well as the plant being sold. As a result of these activities construction and initial operation was not as good as desired. Major efforts have been underway since Century acquired the plant in April, 2001 to improve the potline operation to projected performance levels. This included equipment improvements, revised operating conditions, improved computer process control and operating procedures. This team effort has resulted in a continuous performance improvement for the potline. This presentation will detail many of the areas addressed as well as performance trends to date.

8:55 AM

The Dunkirk SmelterñYears of Technology Creeping and Continuous Improvement: *Claude Vanvoren*[1]; Jerome Bos[1]; Jean-Michel Peyneau[1]; [1]Aluminium Pechiney, Rsrch. Ctr. LRF, BP 114, Saint Jean de Maurienne 73300 France

The Dunkirk smelter, located in North West of France, started late 1991 as the first Pechiney AP 30 smelter. At this early stage of the smelter life, the 264 AP 30 pots were operated at around 295 kA consistent with the original cathode design and a ìclassicalî ACD management. From progressive turnover to fully graphitized cathode blocks and continuous improvement practices to reduce ACD, the operating setpoint was progressively increased to 320 kA which was achieved early 1998. At this time, the re-engineering work carried out by the Pechiney Research Center to design a cell operating at about 350 kA was advanced enough to aim for a complete retrofitting of the plant. Combination of results obtained on test pots in St Jean de Maurienne and local results obtained on cells equipped with a booster rectifier enable a first change of anode format in May 2001. This first step leads to an operation at around 335 kA. In summer 2002, both implementation of the patented Forced Convection Network and final AP 35 anode format were completed, enabling future operation at about 344 kA. The AP 35 operating setpoint, over than 350 kA, will be achieved early 2005 with the new AP 35 lining design, which implementation started early 2002.

9:20 AM

Improvement of Potroom Operational Results by Team Work Group Organisation: *Elmar Sturm*[1]; Michael Grunwald[2]; [1]Hamburger Aluminium-Werk GmbH, Potrooms, Dradenauer Hauptdeich 15, Hamburg 21129 Germany; [2]Consultant, Hamburg Germany

Three years ago a so-called semi-autonomous team work organization was introduced at HAW. This type of work organization, consists of a flat organization structure, gives more responsibility to the workers and has already been successfully introduced in the car- and chemical industry. At HAW the first attempt was made to introduce this model into a continuous shift potroom work organization. Work team groups were installed in the potrooms, gas cleaning/maintenance, metal treatment/transport and workers were trained to carryout multi-purpose functions. Work quality standards were established and controlled by independent personnel. The results were directly communicated and visualized. Part of the waging system has been related to work quality of the teams. Within the last three years the quality of work has been tremendously improved with a significant influence on operational results.

9:45 AM
Key Aspects to Sucessfully Restart Pots After Major Power Curtailment: *Joel J.P. Camara*[1]; Nilson F. Ferraz[1]; Nilson P. Souza[1]; [1]Alumar, BR-135, Km 18 Distro Indutrial de, Sao Luis, MA 65095-050 Brazil

Aluminum plants incur power curtailment due to various reasons, mostly imposed by the country governments when struggling to cope with power demand, usually during severe draught seasons. This was the case in Brazil in the summer of 2002, when Alumar, an Alcoa & Billiton joint venture, had to reduce 161 MW of its total 610 MW. This presentation highlight the various key aspects in being successful at restarting pots, and the Alumar very successful experience in re-starting 140 Alcoa A697pots in 24 days, without a single pot loss which could be attributed to the restart process method. The restart occurred without injury incident of any kind, and with a very quick process/operational stability recover. It will describe the various key aspects of planning, maintenance/anode supply/ingot/potroom operation interactions, process control and crews/process teams organization and training needs. Also, an evaluation was made of two different restart methods currently used at various Alcoa facilities, one of which tried at Alumar, and the other effectively used in this campaign.

10:10 AM Break

10:20 AM
How to Make the Hydro Aluminium Sunndal Expansion Project Competitive with Other Profitable Projects by Using the HAL-250 Pot Technology: *KÅre ÿrbeck Vee*[1]; Jan Arve Haugan[1]; Anton Hus□y[1]; [1]Hydro Aluminium, Tech. Ctr. ≈rdal, PO Box 303, ÿvre ≈rdal NO-6882 Norway

The Brownfield expansion of 239,000 tonnes at the Sunndal plant, thereby replacing 66,000 tonnes from the old S□derberg potline, is based on Hydro Aluminiumís own HAL-250 technology. Evaluation and comparison with other pot technologies have shown that this technology is very competitive, and it is the prerequisite for the present project, both in terms of cost efficiency, volume, investment cost and profitability. The presentation explains why, in terms of: Space efficiency - two rows of pots in one building to cope with the very limited available space on site; Very low fluoride emissions - less than 0.35 kg F/t Al by use of a special gas collection system, to meet environmental regulations (both OSPAR and local conditions); Advanced computer simulations - used to design the ventilation system in the pot room to minimize heat stress for the operators; High productivity - which is necessary due to high manning costs in Norway; Possibility of a common anode configuration and equipment with the existing old prebake potline in Sunndal and in other Hydro Aluminium plants, and further development of these. This competitive position is achieved both through solutions of technical matter (equipment, design, etc.) and through solutions of operational matter (routines, organization, etc.) The present presentation will describe the elements and the progress of the ongoing modernization project in Sunndal.

10:45 AM
Reduction in Pot Fumes in the Potroom Area: *Gilles Proulx*[1]; Gilles Massicotte[1]; Michel Gendron[1]; [1]Alcoa Deschambault, 1 Blvd. Des Sources, Deschambault, Quebec GOA 1SO Canada

Fluoride emissions produced during aluminum reduction electrolysis process consist mainly of gaseous and particulate fluoride. They exit from the cell into main gas duct system and transport to the dry scrubber. Most of the fluoride emissions comes from the anodes setting operation. During this operation, covers are removed and anodes butts are placed into a tray. Other emissions are also observed during metal and bath tapping, pot dressing, etc. These operations generate emissions: from the pot (because covers are removed), from the anodes butts, bath crust bin, bath pans, metal siphoning. These sources of fluoride emissions have got an environmental impact because the dry scrubber canít capture them. Challenge is to find a way to keep the gas inside the tray and the bath bin and maximize the time with full pot enclosed for all pot operations. This presentation shows how improvements on fluoride emissions can be achieved, not only by adding equipment but also by improving working procedures in potroom.

11:10 AM
New Anode Effect Suppression Program: *Dany Vaillancourt*[1]; Pierre Tremblay[1]; [1]Alcoa Aluminerie de Baie-Comeau, 100 Maritime Rd., Baie-Comeau, Quebec G4Z 2L6 Canada

A new anode effect suppression program developed at the Alcoa Reduction plant at Baie-Comeau, Quebec, has helped to decrease dramatically the total anode effect duration in AP-18 prebake pots. In general, this program automatically lowers the anode beam as quickly as possible, until the total voltage of the cell drops below 8 Volts. In addition, the Baie-Comeau crew has developed an anode effect prediction program, which further reduces anode effect durations by permitting a faster response to a pending anode effect. These programs are two successful elements in the overall effort to reduce the quantity of greenhouse gas emitted by the pots during anode effects. The proposed presentation is an overview of these new programs.

11:35 AM
Advances in Alumina Feeding Control: *Pablo Navarro*[1]; [1]Aluar Aluminio Argentino SAIC, R&D, PO Box 52, Puerto Madryn, Chubut U9120OIA Argentina

For modern cell technologies an advanced control of the alumina feeding is mandatory as it has a important effect on the cellís performance. The use of visual patterns, that condense the variables generated by the control system and present information on the alumina content and self-feeding rate of the cells, represent a higher level of abstraction. They proved to be a useful tool to detect tendencies in the first deviation steps and help the potroom operators to focus on abnormal behaviours. A change in the covering practice was carried out to reduce the amount of uncontrolled alumina that enters into the pot. New algorithms were implemented in the Supervisory Level to optimise the parameters of the control system to changes in raw materials quality and different cell conditions. All these changes allowed a significant reduction in the anode effect frequency, reduced process variability, and improved all the alumina feeding variables.

Applications and Processing of Powder Metallurgy Refractory Metals and Alloys: Molybenum Base Alloys and Modeling

Sponsored by: Structural Materials Division, SMD-Refractory Metals Committee, MPMD-Powder Materials Committee
Program Organizer: John J. Stephens, Sandia National Laboratories, Joining & Coating Department, Albuquerque, NM 87185-0889 USA

Monday AM Room: 18
March 3, 2003 Location: San Diego Convention Center

Session Chair: David M. Stepp, US Army Research Laboratory, Research Triangle Park, NC 27709-2211 USA

8:30 AM Opening Remarks

8:40 AM
Designed Experimentation to Study Tantalum and Molybdenum Powder Densification: *John F. Bingert*[1]; Sherri R. Bingert[1]; [1]Los Alamos National Laboratory, ADWP, PO Box 1663, MS A106, Los Alamos, NM 87545 USA

Isopress/sinter processing of refractory metals to high density typically involves significant demands on furnace equipment due to the requirements of extreme temperatures and strict atmospheric control. The ability to determine material response from an economical experimental program is therefore a valuable objective. Toward that goal, this study investigated the effects of four process variables on the densification behavior of tantalum and molybdenum powders. Designed experiments were constructed to efficiently quantify several measures of sinter response as a function of isostatic pressure, pressing temperature, sinter temperature, and sinter time. Response surface analysis was then used to analyze the results and construct empirically-based quadratic models to predict densification-related responses. Sinter simulations were performed using the Ashby HIP micromechanical model, and these results were compared with experimental data and microstructural observations.

9:10 AM
Two-Dimensional Model Simulation of Die-Compaction of Metal Powder by Complex Mold: *Hitoshi Hashimoto*[1]; Zheng-Ming Sun[1]; Yong-Ho Park[1]; Toshihiko Abe[1]; [1]National Institute of Advanced Industrial Science and Technology, Japan, Inst. for Structl. & Eng. Matls., 4-2-1, Nigatake, Miyagino-ku, Sendai 983-8551 Japan

A two-dimensional model simulation of die-compaction of a metal powder by a complex mold was conducted based on discrete element method. In the simulation, force-displacement relations at contacts between two particles and between a particle and mold wall which had been induced from compression test data measured on spherical copper particles were used to calculate forces acting on contacts. A two-dimensional complex mold consisting of three stages (upper stage: 3-mm wide and 1-mm deep, middle stage: 2-mm wide and 1-mm deep, lower stage: 1-mm wide and 1-mm deep) was charged with the copper powder consisting of spherical particles of five sizes (0.050, 0.071, 0.100, 0.141 and 0.200mm) and was pressed under four press conditions. Pressure at each wall of the mold and density distribution in the powder compact were calculated. It was found that local density of the compact well corresponds with the pressure at walls surrounding the compact.

9:40 AM
The Fracture Toughness and Toughening Mechanisms of Wrought LCAC, TZM, and ODS Molybdenum Plate Stock: *Brian V. Cockeram*[1]; [1]Bechtel Bettis Laboratory, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122 USA

The high-temperature strength and creep resistance of Low Carbon Arc Cast (LCAC) pure molybdenum, Oxide Dispersion Strengthened (ODS) molybdenum, and TZM molybdenum make these alloys of interest for various high-temperature structural applications. However, these same alloys have been poorly characterized as to fracture toughness (KIC) and transition temperatures from brittle to ductile behavior. This work reports KIC testing was performed in accordance with ASTM E399 methods over a temperature range of -150C to 1000C using LCAC, ODS, and TZM molybdenum plate stock. The use of bend specimens and compact tension specimens of varying sizes showed that the results obtained with some sub-sized specimens were comparable to values obtained with conventional specimens. Based on the fracture toughness data and failure mode, the transition temperature to brittle behavior, which was defined to occur at a 30 MPa m^1/2, for ODS molybdenum was below room-temperature for both the transverse and longitudinal orientations. The ductile to brittle transition temperature (DBTT) for LCAC and TZM molybdenum in the longitudinal direction was close to 100C, while the transition temperature for the transverse orientation was 150C. In spite of the fact that ODS molybdenum is a powder metallurgy product, it was shown to produce higher toughness values and lower DBTT than LCAC and TZM molybdenum, which are produced by arc melting. It was postulated that the refined microstructure was the reason for this performance difference. Thin sheet toughening is shown to be the dominant toughening mechanism.

10:10 AM Break

10:30 AM
Examination of the Rate and Temperature Sensitivity of a Molybdenum-Rhenium Alloy: *Sean R. Agnew*[1]; Todd Leonhardt[1]; [1]University of Virginia, Matls. Sci. & Eng., 116 Engineerís Way, Charlottesville, VA 22904-4745 USA

This study was conducted to investigate the role of mechanical twinning in the superior low temperature ductility of Mo-47.5 wt% Re, in comparison with pure molybdenum, which undergoes a ductile to brittle transition (DBTT) near room temperature. For Mo-47.5 wt% Re, there is no evidence of a DBTT, even at liquid nitrogen temperature. In fact, the ductility in the cold-rolled condition is actually enhanced by testing at liquid nitrogen temperatures. Another important observation is that at the lowest temperatures, there is essentially no strain rate sensitivity in the flow strength. Mechanical twinning modes typically exhibit less rate and temperature sensitivity than slip modes. Therefore, the current data supports the concept that Mo-Re obtains its ductility enhancement from the activation of mechanical twinning. Microscopic investigations into the level of twinning will also be presented. Moly-rhenium alloys are also prized for their excellent formability, therefore, we also investigated the anisotropy of the sheet material in the cold-rolled and recrystallized conditions.

11:00 AM
Development of Molybdenum Alloy Powders for Ceramic to Metal Seal Applications: *John E. Smugeresky*[1]; Enrique J. Lavernia[2]; Michael S. Ice[3]; John J. Stephens[4]; [1]Sandia National Laboratories, 8724, MS9402, Livermore, CA 94551 USA; [2]University of California-Irvine, Dept. of Matls. Sci. & Eng., Irvine, CA 92697-2575 USA; [3]University of California-Irvine, Irvine, CA 92697 USA; [4]Sandia National Laboratories, Joining & Coating Dept. 1833, MS0889, PO Box 5800, Albuquerque, NM 87185-0889 USA

Molybdenum-Alumina cermets provide both a suitable interface for a leak tight seal and appropriate electrical conductivity for vacuum feedthrough connections. To solve a thermal expansion mismatch between the two materials, Sandia National Laboratories developed Molybdenum-Vanadium alloys with thermal expansion coefficients nearly identical to alumina. A new challenge is to produce alloy powders of sufficient homogeneity and very fine particle size distribution to match that of chemically precipitated Molybdenum and the alumina ceramic. One processing route evaluated cryo-milling as a method to produce the required fine molybdenum alloy powders by mechanically milling elemental metal samples submerged in a slurry of liquid nitrogen. A second processing route evaluated the rotating electrode process for producing powders from bar electrode. This talk will focus on cryo-milling efforts to produce two similar alloys: Mo-27 wt% V and Mo-22 wt% V-3 wt% Fe. These two different alloy systems were each combined from individual powders, blended and then cryo-milled. Each alloy system was milled for 5, 10, 15 and 20 hours respectively. Different milling times allow for comparison of powder size and structure between each time period. The goal is to study how the microstructure and chemical composition change through the milling process. Various analytical techniques were used to determine how the grain size changes as a function of milling time. The effect of how the Fe addition changes the microstructure and grain size of the alloy during the cryo-milling process was noted and will be discussed. Work supported by the US Department of Energy under contract DE-AC04-94AL85000.

11:30 AM
Hydrogen Reduction Route Towards the Production of Nano-Grained Fe-Mo Alloys: *Seshadri Seetharaman*[1]; *Ricardo Morales*[1]; Vijaya Agarwala[2]; Sichen Du[1]; [1]Royal Institute of Technology, Matls. Sci. & Eng., Stockholm SE-100 44 Sweden; [2]Indian Institute of Technology Roorkee, Metall. & Matl. Sci., Roorkee, Uttaranchal 247 667 India

Fe2Mo intermetallic phase with nanograin structure was successfully produced by hydrogen reduction route. Fe2MoO4 was used as the starting material with an average particle size of 80 microns. The activation energy for the chemical reaction was studied using thin powders bed in a thermogravimetric unit in the temperature range 823-1073 K. TEM, SEM and XRD analyses were used for characterization of the intermetallic phase formed. The activation energy for the chemical reaction was found to be 172 kJ/mol. A laboratory scale fluidized bed reactor under isothermal conditions was employed for the bulk processing. The mechanical and other properties of Fe2Mo prepared by this method have been studied using uniaxially cold-pressed pellets and the results are reported in this work.

Automotive Alloys 2003 - I

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA

Monday AM Room: 5B
March 3, 2003 Location: San Diego Convention Center

Session Chair: Subodh K. Das, Secat Inc., Coldstream Rsrch. Campus, Lexington, KY 40511 USA

8:30 AM
Assuring Continued Recyclability of Automotive Aluminum Alloys: Chemical Composition Based Sorting of Wrought and Cast Al Shred: *Adam Jan Gesing*[1]; Ben AuBuchon[1]; Ron Dalton[1]; Richard Wolanski[1]; [1]Huron Valley Steel Corporation, R&D, 41000 Huron River Dr., Belleville, MI 48111-2885 USA

There is continuing development at HVSC of recycling technologies need to assure continued, high-value recyclability of all present and future automotive alloysñparticularly wrought alloys as the use of these increases in the aluminum intensive vehicle. In this paper, we present sort results from a prototype industrial sorter that uses laser induced breakdown spectroscopy to chemically analyze and sort each shredded scrap particle. We demonstrate that the sorter can recognize known alloy particles from each of the major wrought and cast families. We further demonstrate the degree of separation that is feasible within some of the Al alloy families. We also demonstrate that the aluminum alloy mix recovered from the current commercial non-magnetic shredder fraction can be made compatible with secondary wrought alloy compositions by sorting out particles high in selected alloying element concentrations.

8:50 AM
Assuring Recyclability of Automotive Magnesium Alloys: Chemical Composition Based Sorting of Magnesium Shredded Scrap: *Adam Jan Gesing*[1]; Paul Torek[1]; Ron Dalton[1]; Richard Wolanski[1]; [1]Huron Valley Steel Corporation, R&D, 41000 Huron River Dr., Belleville, MI 48111-2885 USA

As the proportion of Mg used in automobile construction increases, there soon will be an economic incentive to separate the Mg scrap recovered from shredded auto hulks into value-added alloy groupings. There are two major Mg alloy families used in automobile construction: aluminum-zinc (AZ) and aluminum-manganese (AM). In this paper, we present magnesium alloy sort results from a prototype industrial sorterñoriginally developed by HVSC for sorting of aluminum-based alloysñthat uses laser induced breakdown spectroscopy to chemically analyze and sort each shredded scrap particle. We demonstrate that the sorter can recognize known alloy particles from AZ and AM families. We further demonstrate the degree of separation that is feasible within some of the Al alloy families based on Al Zn and Mn element concentrations.

9:10 AM
Retrogression Heat Treatment Effects on Hemming in AA6111: *Paul E. Krajewski*[1]; John E. Carsley[2]; [1]General Motors, R&D, MC 480-106-212, 30500 Mound, Warren, MI 48090 USA; [2]General Motors USA

Retrogression heat treatments have been shown to significantly improve the hemmability or tight-radius bending of age-hardenable aluminum alloys. The present paper provides a detailed analysis into the specific effects of retrogression heat treatment on shear band formation and ultimate failure. Hemmed samples are compared both with and without retrogression heat treatments at 300C and 475C. Shear band formation and cracking in hemmed samples are studied after various levels of thermal exposure including the natural aged, partially retrogressed, fully retrogressed, peak aged, and overaged conditions. Ductility in double-notched tensile tests and local plasticity at fracture in standard tensile testing are successfully correlated with bendability.

9:30 AM
Supervised Machine Learning Enhanced Process Diagnosis in Lost Foam Aluminum Casting: *Qi Zhao*[1]; Thomas W. Gustafson[2]; [1]Metal Casting Technology, Inc., 127 Old Wilton Rd., Milford, NH 03055 USA; [2]General Motors Corporation, GM Powertrain at Metal Casting Tech., Inc., 127 Old Wilton Rd., Milford, NH 03055 USA

Machine learning techniques apply human intelligence through various learning algorithms to achieving automatic extraction of hidden predictive information from datasets. The results of proper use of machine learning techniques in high dimensional manufacturing process diagnosis and optimization should be a better understanding of the structure of the data and a clearer perspective of the data relationship than the use of model-dependent statistical analysis, enabling the analyst to reduce hidden variables, build least biased process models, and achieve high prediction performance in process operation. This presentation demonstrates some benefits and limits of applying neural networks and decision trees to the diagnosis and optimization of lost foam aluminum casting of automobile engine parts. It also stresses the necessity of incorporating as much domain knowledge as possible to guide the adventurous knowledge exploration and the interpretation of the resulting patterns/relationships uncovered by the machine learning techniques.

9:45 AM
Stress Corrosion Cracking of Lightweight Automotive Alloys: *Russell H. Jones*[1]; [1]Pacific Northwest National Laboratory, Matls. Sci., PO Box 999, MSIN P8-15, Richland, WA 99352 USA

Stress corrosion cracking can be eliminated or controlled with a knowledge of the material and environmental conditions that cause it. For lightweight automotive materials such as Al-Mg and Mg-Al alloys grain boundary chemistry and microstructure play a key role in SCC. For Al-Mg alloys, the precipitation of the Mg rich b phase (Al3Mg2) and the enrichment of Mg and Cu all play a role. Modifications of the environment with the corrosion inhibitor, potassium chromate, results in an acceleration of the SCC rate even though the general corrosion rate is decreased. This results from the differential effect of the inhibitor on the b and Al phases and the continuing active corrosion of the b phase. For Mg-Al alloys, grain boundary precipitation of the g phase (Mg17Al12) results in the opposite electrochemical conditions, of the particle relative to the matrix, as compared to the Al-Mg alloys. In Mg-Al case, the g phase is the cathode and the matrix the anode while in the Al-Mg case, the b phase is the anode and the matrix is the cathode. The effects of the grain boundary structures on SCC of these alloys will be presented in the context of the role of SCC as a corrosion damage function and how to minimize the impacts of SCC.

10:00 AM
Annealing Study of Al-Mg Alloy AA5754: *Johnson Go*[1]; Taryn Biggs[1]; Warren J. Poole[1]; Matthias Militzer[1]; Mary A. Wells[1]; [1]University of British Columbia, The Ctr. for Metallurgl. Process Eng., Vancouver, BC V6T 1Z4 Canada

Process modelling of the microstructural changes that occur during continuous annealing is an important activity in terms of quality as the final sheet formability can be affected by the annealing parameters after cold working. The present work describes a laboratory investigation of the kinetics of recovery and recrystallization in an automotive alloy AA5754 during annealing. Using this data, a microstructure model based on the internal state variable approach has been developed to predict the softening kinetics and final recrystallized grain size based on the degree of prior cold work and thermal history experienced by the material. Continuous heating tests using the Gleebleô 1500 were performed simulating heating rates of industrial continuous annealing lines to validate the model predictions. Electron backscatter diffraction experiments were also undertaken in order to obtain information on texture evolution during annealing. The samples appeared to become less textured after annealing.

10:15 AM
Studies of Early Stages of Hardening in Al-Mg-Cu Alloys: *Libor Kovarik*[1]; Stephen A. Court[2]; Michael J. Mills[1]; [1]The Ohio State University, 2041 College Rd., Watts Hall #477, Columbus, OH 43210 USA; [2]Alcan International Ltd., Banbury Lab., Southam Rd., Banbury, Oxfordshire OX16 2SP UK

The early stages of aging in ternary Al-Mg-Cu alloys was studied using two alloys with small Cu/Mg ratio (0.05 & 0.127 at.%/at.%). The effect of excess of vacancies, which was varied by solutionizing (quenching) temperature, on the extent of rapid hardening was examined. The experimental studies involved tensile testing and microstructure examination using conventional TEM. The rapid hardening was found to be substantial (about 60 MPa) only for the alloy with higher Cu/Mg ratio, and it was found independent of the amount of as-quenched excess vacancies. Using conventional TEM, it was found that the vacancies do not stay in the solid solution immediately after quenching, but rather form dislocation loops. A much higher density of loops is found for samples quenched from higher temperatures. A greater retention of vacancies in the solid solution for samples quenched from higher temperatures may however be expected due to the enhanced hardening rate at longer times (following the rapid hardening regime). The results suggest that the early stages of hardening are not controlled by long range diffusion of substitutional atoms, nor by the amount of quenched in vacancy loops.

10:30 AM
Effect of Microstructure on Springback: *C. Delfina Joseph*[1]; Judy Schneider[1]; [1]Mississippi State University, Mechl. Eng., 210 Carpenter Eng. Bldg., PO Box ME, Mississippi State, MS 39762 USA

Use of weight-saving materials to produce lightweight components with enhanced dimensional control is important to the automotive industry. This has increased the need to understand the material behavior with respect to the forming process at the microscopic level. A test matrix is developed based on the orthogonal array of Taguchi method. Experiments were conducted for V-bending process using Al 6022-T4 to study the variation of springback due to factors such as bend radius, sheet thickness, grain size, plastic anisotropy, heat treatment, punching speeds, and time. Previous studies have speculated that springback angles for aluminum alloys continued to increase for periods up to several months after forming. The design of experiments was used to evaluate the strongest material and process parameter interactions that contribute to springback in sheet metal forming processes.

10:45 AM
Rheological Characteristics of AZ91 Alloy in the Semi-Solid State: *Faouzi Messaoud*[1]; Lhoucine Azzi[1]; Frank Ajersch[1]; [1]Ecole Polytechnique, Cheml. Eng., Montreal, Quebec H3C 3A7 Canada

Magnesium alloys are gaining more and more interest in automotive applications for structural and non structural components in order to reduce vehicle weight. Semi-solid forming of these alloys has shown to the an effective method of fabricating near net shape components using a thixomolding precess. This study focuses on the rheological and microstructural characteristics of semi-solid AZ91 alloy. Apparent viscosities were measured during continuous cooling and at isothermal conditions using a Couette and a squeezing flow viscometer. The evolution of the viscosity as a function of cooling rate and duration of shear were experimentally determined. The results of the measurements were used to develop analytical models describing the thixotropic nature of these types of alloys in the semi-solid state.

11:00 AM
Comparison of the Recovery and Recrystallization Behaviour of Ingot and Continuous Cast AA5754 During Annealing: *Sujay Sarkar*[1]; Mary A. Wells[1]; Warren J. Poole[1]; Matthias Militzer[1]; [1]University of British Columbia, Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T1Z4 Canada

The influence of processing route on the recovery and recrystallization behaviour during annealing of cold rolled AA5754 produced via ingot and continuous casting was examined. Specifically, a series of annealing tests were conducted in salt and oil baths at temperatures ranging from 200 to 400°C and for various lengths of times. Recovery and recrystallization kinetics were followed by measuring the change in the mechanical properties of the material as a function of time and temperature. Texture measurements using EBSD were also done to quantify differences in texture development during recrystallization for both the ingot and continuous cast AA5754.

11:15 AM
Evaluation of Al-Foam Sandwiches Application to the Bottom Structure of a Concept Car: *Natalia S. Ermolaeva*[1]; Prabhu V. Kandachar[1]; [1]Delft University of Technology, Industl. Design Eng., Landbergstraat 15, Delft 2628 CE The Netherlands

Numerical evaluation via optimal design with respect to the minimum structural weight has been done for the application of Al-foam core sandwiches with Al alloy facings as floor panels of the bottom structure of a concept car. A structural optimization system based on the Multipoint Approximation method with Response Surface fitting (MARS) and MSC. Marc FEA code has been applied. The bending and torsion stiffness of the bottom structure along with the demand of geometrical stability of its components were considered as main constraints. It has been shown that (a) an ultra-low density ($\rho < 70$ kg/m^3) Al foam cannot be used as a core material for considered structural application (no optimal solution has been achieved), (b) low and medium density Al-foams give no mass savings in comparison to all-Al-alloy structure, and (c) a high-density ($\rho = 500$ kg/m^3) Al-foam reduces the weight of the entire bottom structure by 17%.

11:30 AM
Development of Discontinuously Reinforced Al Alloy Composites for Automotive Applications: *Qingjun Zheng*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. Metallurgl. & Matls. Eng., A129 Bevill Bldg., 126th Seventh Ave., Tuscaloosa, AL 35487-0202 USA

Discontinuously reinforced Al alloy composites (DRACs) have superior mechanical properties and light weight and have received growing attentions of automotive engineers. In this work, in-situ processing of AlN particulate reinforced DRACs was investigated. In comparison with SiC particulate reinforced DRACs, AlN particulate reinforced DRACs can also give good mechanical properties, while AlN is stable in the normally used Al alloy matrix in both the service and processing temperature range. Assisted by the thermodynamic modeling, processing of equilibrium AlN reinforcement was achieved in the Al and Al-Si alloy melts by bubbling N2 and NH3 gas in the temperature range of 1323K-1573K. The Products were characterized using X-ray diffraction, optical microscope, scanning electron microscopy, and energy dispersive X-ray analyses. The results showed that AlN particles formed in-situ were small in size (<5 microns) and uniformly dispersed in the Al-alloy matrix.

11:45 AM
Al-Cu-Si-Ge: A New High Strength Aluminum Alloy: *David Mitlin*[1]; Velimir Radmilovic[2]; Ulrich Dahmen[2]; J. W. Morris[3]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, MS 72-150, 1 Cyclotron Rd., Berkeley, CA 94720 USA; [3]University of California, Berkeley & Lawrence Berkeley Natl. Lab., Matls. Sci. Dept., MS 66, 1 Cyclotron Rd., Berkeley, CA 94720 USA

Al-Cu-Si-Ge alloys display a unique combination of ultra-rapid aging response, high peak hardness and extended aging microstructural stability. The purpose of this work is to explain these properties in terms of the role that the Si-Ge additions have on modifying the conventional Al-Cu aging sequence. In both AlCu and AlCuSiGe the room temperature microstructure consists of both GP zones and theta-double-prime precipitates. Upon aging at 190∞C Al-Cu displays the well known precipitation sequence; the slow dissolution of GP zones and theta-double-prime and the gradual formation of theta-prime. In the quaternary alloy, Si-Ge particles quickly nucleate and grow during elevated temperature aging (they are detected after as little as 30 min. at 190∞C). The Si-Ge particles then act as nucleation sites for theta-prime precipitates, resulting in a peak aged microstructure consisting of a dense distribution of theta-prime attached to Si-Ge.

Cast Shop Technology: Cast Shop Safety

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; David H. DeYoung, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday AM — Room: 6C
March 3, 2003 — Location: San Diego Convention Center

Session Chair: Seymour G. Epstein, The Aluminum Association Inc., Washington, DC 20006 USA

8:30 AM Invited
The Aluminum Industryís Efforts to Prevent Molten Metal Explosions: *Seymour G. Epstein*[1]; [1]The Aluminum Association, Inc., 900 19th St. NW, Ste. 300, Washington, DC 20006 USA

For more than 50 years the aluminum industry has strived to fully understand molten aluminum-water explosions and how they can be prevented. Much has been learned through individual company efforts and through programs instituted by The Aluminum Association and funded by aluminum companies in the US and abroad. The Association efforts have included a number of basic and applied research studies, an international incident reporting program, a scrap rejection notification program, guidelines and training aids on handling and remelting scrap and sow, testing of fabrics to protect employees exposed to molten metal and pot bath, and a continuing series of safety workshops. The information gleaned from these efforts is incorporated in the Third Edition of Guidelines for Handling Molten Aluminum published by the Association in July 2002.

8:55 AM Invited
Melting Safety for Aluminum Processing Facilities: *F. R. Hubbard*[1]; D. C. Pierce[2]; [1]IMCO Recycling, Inc., Hwy. 27 S., Roane County Industl. Park, Rockwood, TN 37854 USA; [2]Consultant

Some progress has been made in the last several years in improving aluminum melting safety performance in the USA. Suppliers have begun to realize their role in melting safety and plant personnel are more aware of ways to avoid the causes of melting incidents. Special equipment, such as dryers or shredders, helps reduce risks of accidents. However, in spite of past efforts, injuries, burns and even fatalities still occur. Scrap and aluminum materials to be remelted are still received in melting plants in a condition or containing objects that call for special action or processing. Plants would do well to consider an approach that could make a difference. In addition to the receiving/inspection crew, everyone in the plant must understand the risk issues surrounding the melting of aluminum (internal, purchased or scrap) and everything else going into a furnace (hardeners, flux, etc.) Managers and supervisors should lead the way in getting everybody involved in preventing accidents when melting aluminum.

9:20 AM Invited
Cause and Prevention of Explosions Involving DC Casting of Aluminum Ingot: *J. Martin Ekenes*[1]; Torstein Saether[2]; [1]Hydro Aluminium Hycast AS, PO Box 603, Otis Orchards, WA 99027-0603 USA; [2]Hydro Aluminium Hycast, Sunndalsora Norway

Production of aluminum alloy process ingot for extrusion and forging applications is commonly performed by means of a hot-top casting technology. Explosions involving hot-top casting operations are preventable. This paper identifies the hazards of hot-top casting. The impact of equipment design, process control and quality of workmanship are examined in the context of preventing explosions.

9:45 AM Invited
Evaluating RSI Sows for Safe Charging into Molten Metal: *Jake J. Niedling*[1]; Mike Scherbak[1]; [1]Alcoa Inc., Ingot Tech., 900 S. Gay St., Knoxville, TN 37902 USA

RSI (Remelt Scrap Ingot) material is a common melting furnace feedstock for the production of aluminum alloys. The use of this material has been limited within Alcoa Inc. to dry hearth charging only due to its potentially higher susceptibility for molten metal explosions. Many advancements have been made over the past 15 years in the production of RSI sows. Alcoa Inc. decided to re-evaluate this material for potential direct charging into molten aluminum. The key contributing factors related to safe use have been investigated and the findings will be presented. This will include material characterization,

characterization of saltcake, sow drying tests, shrinkage cavity modeling, simulated RSI explosion tests to determine minimum moisture pick-up and safety incident analysis.

10:10 AM Break

10:20 AM Invited
Investigating Molten Metal Explosions: *John E. Jacoby*[1]; [1]Consultant, 3398 N. Hills Rd., Murrysville, PA 15668 USA

All molten aluminum explosions should be thoroughly investigated to determine their root cause. An explosion is a traumatic incident in an aluminum plant. Every cast house should have a written procedure that describes how an investigation should be conducted because after a serious incident occurs the concerned personnel may be too traumatized to think clearly about the procedures to follow. The procedure should include: care of the injured, investigation team makeup, preservation of plant and accident scene, key plant personnel to be notified, involvement of explosion experts, witness interview procedures, sampling procedures, final report requirements, etc. If a formal procedure is available when an incident occurs it is usually possible to determine the root cause of the explosion. This enables plant personnel to prevent recurrence of similar incidents in the future.

10:45 AM Invited
Industry Research Efforts to Identify Fabrics for Molten Aluminum Environments: *Charles D. Johnson*[1]; [1]The Aluminum Association, Inc., 900 19th St. NW, Ste. 300, Washington, DC 20006 USA

Early research carried out by the aluminum industry identified several shortcomings of FR fabrics used within the general molten metals industries. Further research was called for to determine whether these fabrics could provide adequate protection in molten aluminum settings. This work led to a specific ASTM test method for FR fabrics, and an extensive testing program undertaken by The Aluminum Association. The results of that testing program will be summarized in this presentation.

11:10 AM Panel Discussion

Computational Methods in Materials Education: Insertion of Computational Methods I

Sponsored by: TMS-Education Committee
Program Organizers: Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, State College, PA 16082-5006 USA; Mark D. Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA

Monday AM Room: 13
March 3, 2003 Location: San Diego Convention Center

Session Chair: Long-Qing Chen, Pennsylvania State University, Matls. Sci. & Eng. Dept., University Park, PA 16802-5005 USA

8:30 AM Opening Remarks

8:35 AM Invited
An ìAb-Initioî Approach to the Teaching of Materials Science Curricula: *Nicola Marzari*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Cambridge, MA 02139-4307 USA

While computational materials science is largely established as a scientific discipline in its own right, it remains much less defined as an academic subject in the classroom. In particular, the teaching of computational materials science runs the risk of being identified with an enumeration of appropriate and convenient tools, thus missing the rigor and scientific appeal that is inherent in designing and performing computer experiments. We try to devise an innovative and unifying approach to overcome this difficulty, establishing computational materials science as an accurate and predictive description of the energetics of materials, and as a vehicle to explore the complexity of the phase space deriving from this energetics. In doing this we provide a common language for the development of the ìstructureî and ìthermodynamicsî curricula, where the quantum-mechanical energy governs the ground state properties of materials, together with their excited states that are then averaged to obtain macroscopic properti es. The cornerstone of this approach rely on an insightful and descriptive teaching of quantum mechanics to non-physics majors, and in the large space given to computational statistical mechanics, together with the development of an appropriate suite of computer experiments to continuously support the class material.

9:05 AM Invited
Introducing Computational Approaches in Materials Science & Engineering: *Anthony D. Rollett*[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., 4315 Wean Hall, Pittsburgh, PA 15213 USA

Lacking a course devoted to computational materials science, several experiences of introducing computational approaches into courses in the Materials Science & Engineering (MSE) curriculum are described. In a Junior-level course on Materials Processing, heat flow is discussed in some detail because it controls many different processes from solidification to annealing. Students are required to write their own one dimensional heat flow code and verify its output against a standard analytical model. A wide range of aptitude for programming has been observed. Some students complete the assignment with ease and make their own extensions, whereas other individuals struggle to complete the basic assignment. In a graduate course on texture & anisotropy, computation is essential because of the complexity of describing preferred orientation and grain boundary character in polycrystals. Once the appropriate tools for describing orientation (Euler angles, matrices, Rodrigues vectors, quaternions) have been introduced, students are given assignments to familiarize them with symmetry operators, fundamental zones and the partitioning of orientation space (to calculate volume fractions).

9:35 AM Invited
My Experience in the Use of Computational Thermodynamics in Teaching Thermodynamics and Phase Diagrams: *Y. A. Chang*[1]; [1]University of Wisconsin-Madison, Matls. Sci. & Eng., 1509 Univ. Ave., Madison, WI 53706 USA

I have been using PANDAT (a thermodynamic computational tool) as an aid in teaching thermodynamics and phase diagrams at the senior/graduate level at the University of Wisconsin for the past few years. In this presentation, I will attempt to give examples to demonstrate that such a computational tool can aid students to better understand the relationships between the characteristic features of phase diagrams in terms of the relative thermodynamic stabilities of phases in question. It may even also help the students to better visualize ternary phase diagrams.

10:05 AM Break

10:35 AM Invited
Development of Computational Materials Science Software for Undergraduates: *Suzanne E. Mohney*[1]; Gary L. Gray[2]; Andrew J. Miller[2]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Eng., 109 Steidle Bldg., University Park, PA 16802 USA; [2]Pennsylvania State University, Dept. of Eng. Sci. & Mech., Earth & Eng. Scis. Bldg., University Park, PA 16802 USA

We have developed two software packages and have tested them in an undergraduate materials processing course for juniors in materials science and engineering. The goal of the first software package is to give students experience solving heat transfer and diffusion problems in two dimensions when a convenient analytical solution is not available, to allow students to better visualize the solutions to their problems, and to introduce more sophisticated design problems into the course. The program provides a convenient graphical user interface to a finite difference solver that we programmed in MATLAB. A demonstration of the software will be given, and student feedback and examples of homework problems will be described. The second package we have developed is an atomistic simulation of diffusion from which students can generate data to discover, on their own, the outcome of many random walks.

11:05 AM Invited
Computational Methods in Materials at Lehigh: *Jeffrey M. Rickman*[1]; [1]Lehigh University, Matls. Sci. & Eng., 5 E. Packer Ave., 244 Whitaker, Bethlehem, PA 18015 USA

Various courses stressing computational methods have been introduced into the curriculum in the last few years. In this talk I will highlight the contents of three courses, namely an undergraduate methods courses taken by sophomores majoring in materials science and engineering and two graduate-level courses. The undergraduate course stresses numerical methods and data analysis as applied to problems in materials science. The graduate-level courses stress mathematical methods and some simulational techniques, especially as they apply to the study of the kinetics of phase transformations.

11:35 AM Invited
The Need for Teaching Modules: *Afina Lupulescu*[1]; M. E. Glicksman[1]; [1]Rensselaer Polytechnic Institute, Dept. of Matls. Scis. & Eng., Troy, NY 12180-3590 USA

Two critically-related problems currently face undergraduate and graduate departments offering materials science and engineering: 1) declining enrollments, and 2) attracting and retaining students to the study of materials. To draw and hold the attention of students to any classical field of scientific or engineering, students must become convinced that their choice offers a vibrant, challenging, yet economically sound future. How can faculty help attract and convince students to materials science and engineering? Improved methods of teaching become high priority. Teaching modules to be discussed allow instructor and students easier access to new texts in diffusion, kinetics, crystal growth and solidification processing. Their inherent flexibility encourages modification to suit the taste and biases of all users: students and instructor. The modules were developed to address easing the adoption of new texts and material by reducing the steepness of ìlearning curvesî and simultaneously adding interest and excitement to the learning experience.

Computational Phase Transformations: Atomistic Modeling I - Phase Stability

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Monday AM — Room: 11B
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Murray S. Daw, Clemson University, Physics & Astron., Clemson, SC 29634-1911 USA

8:30 AM Opening Remark

8:35 AM Invited
New Ground State Structures in Old Intermetallic Systems: *Alex Zunger*[1]; [1]National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401-3393 USA

We discover that Au-rich $Cu_{1-x}Au_x$ and Pt-rich $Ni_{1-x}Pt_x$ contain a composition range in which there is a quasi-continuum of stable, ordered ìadaptive structuresî made of (001) repeat units of simple structural motifs. This is found by searching ~$3x10^6$ different fcc configurations whose energies are parameterized via a ìcluster expansionî of LDA-calculated total-energies of just a few structures. This structural adaptivity is explained in terms of an anisotropic, long-range strain energy.

9:05 AM
Ab Initio Investigation of Atomic Displacements in the YBCO Superconductor: *Didier deFontaine*[1]; Vidvuds Ozolins[2]; Nathan Speed[1]; [1]University of California, Dept. of Matls. Sci., 577 Evans Hall, Berkeley, CA 94720-1760 USA; [2]Sandia National Laboratories, Livermore, CA 94551-0969 USA

The recent discovery of a ìpseudogap stateî has led to renewed interest in High-Tc superconductors. Synchrotron X-ray diffraction investigations by Islam and co-workers have shown the existence of well-defined satellite reflections in both under-doped and in optimally-doped detwinned YBCO crystals. Here we show that the observed satellite intensity can be explained, at least in part, by quasi-periodic atomic displacements resulting from oxygen ordering in the Cu-O planes of these superconductors. To that aim, we have performed first-principles fully-relaxed total energy calculations in oxygen deficient YBCO supercells. Calculated displacements of Y, Ba, Cu, and oxygen atoms from their positions in the ideal stoichiometric compound were then inserted in structure factor calculations which agreed semi-quantitatively with experimentally determined diffraction scans along the H direction in reciprocal space. Temperature effects were studied qualitatively by Monte Carlo simulations of oxygen ordering in Cu-O planes.

9:25 AM Invited
A New Approach to Predict the Structure of Alloys: *Gerbrand Ceder*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 13-5056, Cambridge, MA 02139 USA

The ability to predict the crystal structure of a material, given its constituent atoms, is one of the most fundamental problems in materials research. Knowledge of the crystal structure is essential to predict or rationalize properties of the material, from mechanical behavior, to optical and electronic properties. Despite its importance, the structure problem remains unsolved and most crystal structure determinations are performed after synthesis, by experimental means. While first principles computations can be used to predict with high accuracy a structural energy, ground state searches are usually limited to calculating the energy of a small number of pre-defined structures. Hence it is difficult to make predictions for completely novel and unknown systems. In order to drastically improve the capability of predicting the ground states of intermetallic alloys, we present an algorithm that can rank a relatively large number of trial structures in terms of the probability that they are ground states. First principles predictions can then be performed on the most likely candidates. With each first principles calculation, the candidate list is improved. This technique makes it possible to predict intermetallic ground states with ~90% accuracy using only ~20 first principles calculations. Unlike previous methods, this approach is not limited to super structures of a given lattice type and extends relatively easily to multi-component systems.

9:55 AM
Using First-Principles Calculations to Improve Thermodynamic Databases: *Axel van de Walle*[1]; Gautam Ghosh[1]; Mark D. Asta[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

We illustrate how thermodynamic properties of alloys can be calculated from first principles using the Alloy Theoretic Automated Toolkit. Recent additions to this toolkit are presented, including the construction of special quasirandom structures, the calculation of point defect energies, structure generation tools, and the calculation of non-configurational entropy. Special attention is devoted to utilities that enable the calculation of vibrational entropy using transferable bond-length-dependent force constants. We also describe how the resulting thermodynamic data can be combined with thermodynamic databases for use in calphad-type calculations using an easy-to-use interface with Thermocalc. Examples of applications will be given for Ti-based and Ni-based alloys.

10:15 AM Break

10:30 AM Invited
Ordering Trends in FCC-Based Alloys: *Patrice E.A. Turchi*[1]; Vaclav Drchal[2]; Josef Kudrnovsky[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci. (L-353), PO Box 808, 7000 East Ave., Livermore, CA 94551 USA; [2]Institute of Physics, Acad. of Scis. of the Czech Republic, Na Slovance 2, Prague-8 CZ-182-21 Czech Republic

The six phase diagrams made of Rh, Ir, Pd, and Pt indicate in the solid phase the existence of a miscibility gap located at low temperature when compared with the melting points of these transition metals. Using a first-principles electronic-structure approach departures from phase separation tendencies are predicted. The methodology is based on the Generalized Perturbation Method applied to the fully relativistic Tight-Binding Linear Muffin-Tin Orbital description of the electronic structure of the chemically random configuration of the alloy. Finite temperature effects are accounted for with the generalized mean-field Cluster Variation Method. Ordering trends and stability properties are rationalized as functions of simple electronic parameters. Work performed under the auspices of the US Department of Energy by the University of California Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

11:00 AM
First-Principles Investigation of the Ordered Si_4C Phase: *Wolfgang Windl*[1]; Otto F. Sankey[2]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210-1178 USA; [2]Arizona State University, Physics, PO Box 851504, Tempe, AZ 85287-1504 USA

We have performed intensive theoretical studies - including ab-initio Monte Carlo simulations - on the ordered Si_4C phase which has been proposed first by R¸cker et al. theoretically [Phys. Rev. Lett. 72, 3578 (1994)] and which has been reportedly synthesized by Kouvetakis et al. [Appl. Phys. Lett. 72, 930 (1998)]. We calculate structural parameters which are significantly different from experimental values, and an IR spectrum with a peak position very similar to their experiment but a much narrower width. We suggest that the experimental findings might be consistent with the assumption of a system of Si_4C sections/crystallites and other Si-C structures embedded in a Si matrix.

11:20 AM
Chemical Disorder-Induced Glass Transition in Silicon Carbide: *Linn W. Hobbs*[1]; Xianglong Yuan[2]; [1]Massachusetts Institute of Technology, Depts. of Matls. Sci. & Eng. & Nucl. Eng., Rm. 13-4054, 77 Massachusetts Ave., Cambridge, MA 01239-4307 USA; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Rm. 13-4050, 77 Massachusetts Ave., Cambridge, MA 02139-4307 USA

Glass transitions arise from a variety of structural and entropic circumstances. We have discovered one driven by chemical disorder during a molecular dynamics investigation of the radiation-induced amorphization of silicon carbide, a structural phase transformation that was difficult to understand on the basis of the substantial topological overconstraint present in SiC crystal structures. Simulations of displacement cascades (as well as some experimental measurements) had shown significant induced chemical disorder (especially C on S sites) and interstitial defect configurations necessarily involving homonuclear bonds. We simulated chemical disorder over a large range of homonuclear bond fractions by random atom switching in crystalline SiC followed by equilbration using molecular dynamics methods (and Tersoffís 1994 empirical potential for SiC) over a large temperature range. Crystallinity was marginally metastable at 300 K for random site occupation (homonuclear:heteronuclear bond ratio = 1), but the configurations structurally amorphized (lost topological order) for ratios above 0.3 when given sufficient thermal energy (at 2000 K) and exhibited a reversible glass transition at 3000 K.

11:40 AM
Phase Stability Modeling of the Al-Hf System: *Gautam Ghosh*[1]; Axel van de Walle[1]; Mark D. Asta[1]; Greg B. Olson[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

As a part of our integrated approach for computational materials design, we will present the recent results of phase stability modeling of the Al-Hf system. Due to very limited experimental thermodynamic data in this system, we have carried out a comprehensive first-principles calculations to describe the phase stability of solid solutions and the intermetallic phases. The results of first-principles calculations are then integrated within CALPHAD formalism to model the Al-Hf phase diagram.

Defects and Deformation of Crystalline Solids - in Honor of Dr. Man H. Yoo - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
Program Organizers: Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Department, Houghton, MI 49931-1200 USA; Sean R. Agnew, University of Virginia, Materials Science and Engineering Department, Charlottesville, VA 22904-4745 USA; K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA

Monday AM — Room: 17A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: John Cahn, NIST, Gaithersburg, MD 20899-8555 USA; Tetsuo Mohri, Hokkaido University, Div. of Matls. Sci. & Eng., Sapporo 060-8623 Japan

8:30 AM Introduction by K. N. Subramanian of Michigan State University

8:40 AM Invited
First-Principles Studies of Phase Partitioning and Interfacial Segregation: *Mark D. Asta*[1]; Vidvuds Ozolins[2]; C. Woodward[3]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208-3108 USA; [2]Sandia National Laboratories, Livermore, CA USA; [3]UES, Inc., Air Force Rsrch. Lab., WPAFB, Dayton, OH 45432 USA

The application of first-principles methods to the study of bulk phase stability and coherent-interface properties in metallic alloys is described. The computational approach combines first-principles calculations of intermetallic-compound and point-defect formation energies, with statistical models for the free energies of ordered and disordered substitutional alloy phases. We focus on applications to precipitation-strengthened Al-Sc alloys where first-principles methods are used to calculate partitioning and interfacial segregation energies for ternary additions in two-phase Al/Al_3Sc microstructures. Results are compared to recently measured compositional profiles derived from three-dimensional atom-probe microscopy that show pronounced segregation of Mg to coherent Al/Al_3Sc interfaces. First-principles calculations reveal a substantial electronic contribution to the driving force for interfacial segregation in this system.

9:05 AM Invited
First Principles Simulations of Point, Line, and Planar Defects in Structural Metals: *Christopher F. Woodward*[1]; [1]UES, Inc., Air Force Rsrch. Lab., WPAFB, 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

We review how first principles methods have been used to study point, line, and planar defects in structural metals. Such methods have been used extensively to study point and planar defects in intermetallic alloys. Using reference energies derived from first principles calculations several groups have estimated site selection and phase partitioning of solutes using simple models for the free energy. Also, planar faults such as stacking faults and interfacial boundaries have been modeled using standard methods for stoichiometric alloys. The effects of variations in chemistry have been estimated using free energy models (the cluster expansion) and novel first principles methods (the coherent potential approximation). Most recently we have applied a flexible boundary method to calculate from first principles the equilibrium structure and lattice friction stress of isolated line defects (dislocations) in the BCC transition metals. In the time permitted we will compare the results of some of these studies and explore the strengths and limitations of these methods.

9:30 AM
Theoretical Investigation of Dislocations Cell Structure Formation Within Reaction-Diffusion Equation: T. Shoji[1]; *T. Mohri*[1]; [1]Hokkaido University, Div. of Matl. Sci. & Eng., Grad. Sch. of Eng., Kita13 Nishi 8, Kita-ku, Sapporo, Hokkaido 060-8628 Japan

Theoretical approach to collective behavior of dislocations based on Reaction-Diffusion equation (R-D equation) was initiated by the pioneering work of D. Walgraef et al. Most works, however, have been focused on the reproduction of the formation process of dislocation microstructures during the fatigue test. In the present study, we attempt to extend it to the study of microstructural evolution process of dislocations during a conventional uniaxial tensile test for a FCC single crystal with two operative slip systems. The present calculation reproduced a typical stress-strain curve of a FCC single crystal with three stages of work hardening. Along the stress-strain curve, the microstructural evolution process is visualized. It is observed that the dipolar walls of dislocations are evolved on the primary slip system with the strain in the first stage of work hardening and that the dislocation cell structure by the activation of the secondary slip system is developed from the second to the third. It is confirm ed that the formation of dislocation cell structure is due to the inter-slip system reactions, e.g. Lomer-Cottrell locking and tangling of moving and forest dislocations. The underlying principle of collective dislocations behavior is also explored.

9:50 AM Invited
The Role of Lattice Vacancies in Intermetallic Compounds: W. Sprengel[1]; X. Y. Zhang[1]; *H.-E. Schaefer*[1]; [1]Stuttgart University, Inst. of Theoretl. & Appl. Physics, Pfaffenwaldring 57, Stuttgart 70569 Germany

The concentrations of vacancies, their mobilities and their locations in intermetallic compounds have been studied recently with fundamental contributions of Man Yoo. The role of vacancies for atomic processes, diffusion, creep, plasticity etc. was elucidated in a variety of binary intermetallic compounds by employing specific techniques as positron annihilation spectroscopy[1,2] and dilatometry.[3] Novel information on atomic processes could be obtained for very high temperature intermetallics as MoSi2[4] and for binary compound semiconductors.[2] [1]H.-E. Schaefer et al., Intermetallics 7, 277 (1999). [2]A. A. Rempel et al., to be published. [3]H.-E. Schaefer et al., Phys. Rev. Letters 82, 948 (1999). [4]X. Y. Zhang et al., to be published.

10:15 AM Break

10:35 AM Invited
Surface Nanostructure Controlling of B2 FeAl Single Crystal by Vacancy Clustering: *Shuji Hanada*[1]; Kyosuke Yoshimi[1]; Tomohide Haraguchi[1]; [1]Tohoku University, Inst. for Matls. Rsrch., 2-1-1, Katahira, Aoba-ku, Sendai 980-8577 Japan

Nanopores which have several tens nm in diameter can be formed near surfaces of B2 FeAl single crystal by supersaturated vacancy clustering. To produce the nanoporous surfaces, surface treatment, quenching conditions and aging conditions are important; i.e., strain-free and clean surfaces, extremely high vacancy concentration, moderate temperature enough for vacancy migration, reduction or non-oxidation atmosphere, and so on. Surface nanostructure was observed before and after vacancy clustering by SEM and AFM, and during in-situ heating experiments by TEM. The pores have specific morphology and crystallography with pore surfaces faceting toward (100) planes, so that it is possible to control pore shape by changing surface orientation of the single crystal. The pore size and density are also controllable by quenching and aging temperatures. This nanoporous surface structure will be applied to the hybridization with functional biomolecules.

11:00 AM
Grain Boundary Constraint Effects on the Constitutive Response of Tantalum Bicrystals: Experiments and Finite Element Analysis: *Alexander Ziegler*[1]; Geoffrey H. Campbell[1]; Mukul Kumar[1]; James S. St^lken[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., 7000 East Ave., L-371, Livermore, CA 94550 USA; [2]Lawrence Livermore National Laboratory, New Techs. Eng. Div., 7000 East Ave., L-356, Livermore, CA 94550 USA

A series of uniaxial compression tests were performed on tantalum bicrystals in order to quantify the role of grain boundary constraint in strain localization, slip system activation, slip transmission, and the concomitant constitutive response. Tantalum single crystals were diffusion bonded to form a (110) twist boundary and compressed along the [110] direction. Interrupted testing facilitated monitoring the resulting three-dimensional deformation, and using a combination of volume reconstruction and surface grid evaluation both the effective states of stress and strain over the cross-sectional area could be measured as a function of distance from the twist boundary. Local strains and strain gradients were measured as a function of position on the surface of the test specimen. Post-test metallurgical characterization was performed using Electron Back-Scattered-Diffraction (EBSD). The results, a spatial distribution of slip patterning and mapping of crystal rotation near the twist-boundary was analyzed and co mpared to the known behavior of the individual single crystals. The detailed shape change and lattice rotation measurements were compared with finite element simulations. The influence of the constraint induced multi-axial stress-state on the constitutive assumptions of crystal plasticity shall be critically examined.

11:20 AM Invited
Bulk and Defect Properties of Ordered Intermetallics: *C. L. Fu*[1]; M. H. Yoo[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA

We review the interdisciplinary effort by first-principles quantum mechanical calculations and applied continuum mechanics in understanding the fundamental factors that govern the deformation and facture behavior of ordered intermetallics. This includes Ti-Al, Fe-Al, Ni-Al, and Ni-Si based compounds. More specifically, elastic constants, point defect self-energies, various shear fault energies, and cleavage energies have been determined by first-principles calculations. By using these calculated results, continuum modeling based on dislocation theory and fracture mechanics correctly explained the intrinsic properties of flow strength in Ni3Al and TiAl and contrasting fracture behavior in NiAl and FeAl alloys. Physical constants that are relevant to mechanical behavior of single-phase TiAl and Ti_3Al and two-phase $TiAl/Ti_3Al$ alloys will also be summarized and discussed. Work sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences. ORNL is operated by UT-Battelle, LLC, for the USDOE under contract DE-AC05-00OR22725.

11:45 AM
A Model for Lattice Strain Effects on Spin/Orbital Ordering in Manganese Oxides: Hyunjung Lee[1]; Jaejun Yu[1]; Tae-Won Noh[1]; *Jong K. Lee*[2]; [1]Seoul National University, Sch. of Physics, Seoul 151-742 Korea; [2]Michigan Technological University, Dept. of Matls. Sci. & Eng., Houghton, MI 49931 USA

Role of lattice strain in colossal magnetoresistance (CMR) manganites is of particular interest as such materials can be harnessed as the next generation magnetoresistance materials. Specifically, the strain dependence of magnetic and orbital ordering is an interesting subject as controlled application of CMR manganites is feasible through a thin film processing where elastic strain is present due to lattice mismatch. To simulate strain effects on the magnetic, orbital, and charge ordering, an effective Hamiltonian is introduced in which inter-site spin/orbital interactions are mapped into an Ising-like model. In the model, electronic contributions are described through the exchange couplings for the spin and orbital degrees of freedom. The elastic strain contribution is formulated in terms of the Discrete Atom Method in order to express an appropriate description for both the Jahn-Teller interactions and the inter-site elastic couplings. The model results at different doping levels and lattice strains show that both charge and orbital orderings are sensitive to elastic strain and, in particular, orbital ordering is essential in the process of strain relaxation.

Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong: High Strain Rate Deformation

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Marc Andre Meyers, University of California-San Diego, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; George T. Gray III, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA; Kenneth S. Vecchio, UC San Diego, Dept of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA

Monday AM Room: 16B
March 3, 2003 Location: San Diego Convention Center

Session Chair: Marc Andre Meyers, University of California-San Diego, Dept. of Mechl. & Aeros. Eng., La Jolla, CA 92093-0411 USA

8:30 AM
Dislocation Mechanics Based Constitutive Equations: *Frank J. Zerilli*[1]; [1]Naval Surface Warfare Center, Indian Head Div., 101 Strauss Ave., Indian Head, MD 20640 USA

A review of constitutive models based on the mechanics of dislocation motion will be presented with focus on the models of Zerilli and Armstrong and the critical influence of Armstrong on their development. The models were intended to be as simple as possible while still reproducing the behavior of real metals. The key feature of these models is their basis in the thermal activation theory propounded by Eyring in the 30ís. The motion of dislocations is governed by thermal activation over potential barriers produced by obstacles, which may be the crystal lattice itself (Peierls stress) or other dislocations or defects. Typically, in bcc metals, the lattice interaction is predominant, while in fcc metals the dislocation-dislocation interaction is the most significant. When the dislocation-lattice interaction is predominant, the yield stress is temperature and strain-rate sensitive, with essentially athermal strain hardening. When the dislocation-dislocation interaction is predominant, the yield stress is athermal, with a large temperature and rate sensitive strain hardening. In both cases, a significant part of the athermal stress is accounted for by grain size effects, and, in some materials, by the effects of deformation twinning. In addition, some simple strain hardening models will be described starting from a differential equation describing creation and annihilation of mobile dislocations. Finally, an application of thermal activation theory to polymeric materials will be described.

9:00 AM
Constititive Behavior of HCP Metals: *E. Cerreta*[1]; G. (Rusty) T. Gray[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

The anisotropic response of low symmetry materials is particularly difficult to model. To characterize the deformation behavior of HCP metals for such models, zirconium and hafnium were studied.

Data was collected through different tests to establish a constitutive model, which predicts the anisotropic mechanical response. Here, the mechanical behavior as a function of strain rate, temperature, and texture in hafnium is discussed and compared with zirconium. Specimens were tested in compression quasi-statically and on the Hopkinson Bar in two different specimen orientations; through thickness and in plane specimens have the compression axis aligned parallel and perpendicular to the c-axis, respectively. This creates substantial differences in the resulting deformation. Additionally, the substructural evolution of quasi-statically tested hafnium specimens is discussed in terms of work hardening rates. These observations are compared with substructures in specimens tested to 5% strain on the Hopkinson Bar and to zirconium.

9:15 AM

Mechanical Response and Constitutive Modeling of Plate Steels: *Carl M. Cady*[1]; Shuh-Rong Chen[1]; George T. (Rusty) Gray[1]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA

The strain rate and temperature sensitivity of the flow stress and the insensitivity of the strain hardening rate indicate that thermal activation over a Peierls barrier is the rate controlling mechanism for the three different steels (HY-65, manganese steel, and the DH-36) studied. The stress/strain response for the steels is shown to be strongly influenced by strain rate and temperature which is consistent with this controlling mechanism. Constitutive modeling efforts are underway to predict the mechanical behavior of materials outside the regions where data for strain-rate and temperature already exists. The Zerelli Armstrong Model can accurately capture the constitutive response of these steels. The Taylor impact test was used to validate the model and show how well the model fit works even outside the region where experimental data exists.

9:30 AM

High Strain Rate Characterization of Be and Be-Al Alloys: *Kathryn A. Dannemann*[1]; Charles E. Anderson[1]; Gordon R. Johnson[2]; [1]Southwest Research Institute, Eng. Dynamics Dept., PO Drawer 28510, San Antonio, TX 78228-0510 USA; [2]Network Computing Services, 1200 Washington Ave. S., Minneapolis, MN 55415 USA

Characterization testing was performed on three commercially available, extruded Be alloys: S200F (~98.5 wt% Be), and AlBeMet 162 and Beralcast 310 (Be-Al alloys containing ~62 wt% Be). The S200F and AlBeMet 162 extrusions were produced from vacuum hot pressed billets; the Beralcast 310 extrusions were made from castings. The extruded Be and Be alloys were of research interest owing to reports of improved ductility in the extrusion direction relative to other Be product forms. Dynamic tension and compression tests were performed using a split Hopkinson pressure bar (SHPB) system at strain rates up to 1400 s^{-1}. For the Be-Al alloys, torsion tests were also conducted at varying strain rates (up to 40 s^{-1}) using a hydraulic test system. Due to anisotropy concerns, mechanical tests were conducted in both longitudinal and transverse orientations when possible. Strength constants for the Johnson-Cook constitutive model were determined for the two Be-Al alloys.

9:45 AM

3-D Parametric Dislocation Dynamics Applied to the Formation and Release of Dislocation Pile-Ups and Localized Heating: *William R. Grise*[1]; [1]Morehead State University, Dept. of IET, Rm. LC-105C, Morehead, KY 40351 USA

The formation of dislocation pile-ups and their release, resulting in localized heating, is studied by means of 3-dimensional ìparametric dislocation dynamicsî, as developed by Prof. Ghoniem at UCLA. We have adapted the parametric dislocation dynamics technique to do the following: (1) calculate the plastic work due to the pile-up release and the resulting, localized, temperature rise. It is believed that this temperature rise, concentrated in a small space, is an important factor in explosive initiation of energetic crystals. The results of the 3-D dislocation dynamics will be compared to previous work on pile-up release, using simpler models. In particular, the research conducted herein is intended to evaluate the need for, and the possible benefits to be gained from, such complex calculations of dislocation behavior as they move and interact in three-dimensional space, when set side-by-side with earlier work in the field.

10:00 AM

The Effect of Microstructure on Strain Rate Sensitivity of Nickel: *Fereshteh Ebrahimi*[1]; Luis E. Forero-Gomez[1]; Eboni F. Westbrooke[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, Gainesville, FL 32611 USA

There have been suggestions that nanocrystalline materials may deform by grain boundary sliding at relatively low homologous temperatures. Therefore, it is expected that the flow stress of these materials to be more strain rate sensitive in comparison to those with conventional grain sizes. In this study the strain rate sensitivity of nickel with various microstructures was evaluated at room temperature. Within the micrometer range, cold working and annealing were used to vary the grain size of a commercial Ni200. Pre-straining of the annealed nickel samples was performed to create different dislocation cell sizes. Low temperature annealing was employed to increase the grain size of the electrodeposited nanocrystalline nickel into sub-micrometer range, comparable to the dislocation cell size of the pre-strained nickel samples. In this paper the results of this study will be discussed in terms of different deformation mechanisms applicable to various microstructures.

10:15 AM Break

10:30 AM

Results from Taylor Experiments with an Internal Grid: *Joel Wayne House*[1]; [1]Air Force Research Laboratory, AFRL/MNMW, 101 W. Eglin Blvd., Ste. 135, Eglin AFB, FL 32542-6810 USA

A series of Taylor Impact Experiments[1] were conducted using specimens with an internal grid. The grid was a series of interconnected lines created by drilling small diameter holes parallel and perpendicular to the specimen axis, in a single plane. The grid was filled with a solder selected for its low melting temperature, good wetting properties, and color contrast with the OFE Copper material of the specimen. Flash X-rays of the specimens identified how well the solder filled the grid and established the initial grid geometry. Recovered specimens were flashed X-rayed to determine the final grid geometry. The final grid geometry was also examined optically by physically sectioning the recovered specimens to reveal the plane of the grid. The Taylor Impact Experiment produces dynamic loading to generate an integrated experiment.[2-4] In the experiment the material state, defined by (P, e, T,), varies with both time and position. The integrate nature of the experiment makes it less useful for determining specific parameters of constitutive theories, but allows those model that have been developed, or characterized, with quasi-static and high rate data to be validated under different experimental conditions. Using a numerical representation of the continuum, and assuming numerical accuracy with an adequate description of the boundary condition, validation of the constitutive theory is implied through comparison with the changing specimen geometry. In its simplest form the final geometry from the experiment is compared with that predicted from the analysis. (See for example Reference 5) With additional diagnostics of the experiment the transient wave behavior, relative to the constitutive theory, can be studied. The internal grid provides an additional diagnostics capability to study the high strain rate behavior of the material and to further validate constitutive theories within continuum software. In this study, the comparison will be based upon Johnson-Cook[6], Zerrilli-Armstrong[7], and the Mechanical Threshold Stress[8] relationships. [1]Taylor, G.I., Roy. Soc. of London, Series A, vol. 194, 1948, pp. 289-299. [2]Wilson, L., House, J., and Nixon, M., ìTime Resolvable Deformation from the Cylinder Impact Test,î AFATL-TR-89-76, Air Force Armament Laboratory, Eglin Air Force Base Florida, November 1989. [3]House, J., Aref, B., Foster, J., and Gillis, P., J. of Strain Analysis, vol. 34, no. 5, 1999, pp. 337-345. [4]Maudlin, P., Gray, G., Cady, C., and Kaschner, G., Roy Soc. of London, Series A, vol. 357, 1999, pp. 1707-1729. [5]Johnson, G. and Holmquist, T., J. Appl. Phy. vol. 64, 1988, pp. 3901-3910. [6]Johnson, G. and Cook, W., Proc. 7th Int. Symp. Ballistics, The Hague, The Netherlands, 1983. [7]Zerilli F, and Armstrong, R., J. Appl. Phy. vol. 61, 1987, pp. 1816-1825. [8]Follansbee, P., and Kocks U., Acta Metallurgica, vol. 36, 1988, pp. 81-93.

10:45 AM

Compressive Deformation Behavior of a Metallic Foam with Regularized Cellular Structure Under Dynamic Loading: *Hidetaka Kanahashi*[1]; Toshiji Mukai[2]; Tatsuhiko Aizawa[3]; Kenji Higashi[4]; [1]The University of Tokyo, Dept. of Metall., Grad. Sch. of Eng., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8904 Japan; [2]Osaka Municipal Technical Research Institute, 1-6-50 Morinomiya, Joto-ku, Osaka 536-8553 Japan; [3]RCAST, The University of Tokyo, High Perf. Matl., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8904 Japan; [4]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Col. of Eng., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

The metallic foam is the lightweight material to be used as an impact energy absorber because of its unique deformation behavior. In general, however, those foams suffer from their weaker and more non-uniform mechanical property than solid materials. The PVC foam reported by Matuana et al. (cell size; 2 μm) had about 4 times as high impact strength as solid PVC. This is the first finding that the designed foam material should be superior to dense solids. The present paper concerns the mechanism of the strain rate dependence on the dynamic response of metallic foam materials and the effect on the microstructuralization of a cell size under dynamic loading by the split Hopkinson pressure bar method.

11:00 AM
Grain Boundary Effects on the Dynamic Deformation of <110> Isoaxial NiAl Bicrystals: *P. D. Peralta*[1]; D. C. Swift[2]; K. J. McClellan[3]; E. Loomis[1]; [1]Arizona State University, Dept. of Mechl. Eng., Main Campus, MC 6106, Tempe, AZ 85287-6106 USA; [2]Los Alamos National Laboratory, P Div., P-24, MS E256, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, MST Div., MST-8, MS G755, Los Alamos, NM 87545 USA

Grain boundary (GB) effects due to elastic and plastic anisotropy on dynamic deformation of materials are studied using isoaxial NiAl bicrystals with <110> tilt boundaries. These bicrystals, along with NiAl single crystals with the same crystallographic load axis, were shock loaded using laser-driven flyers. The shock velocities and surface displacements produced during testing of these samples were quantified using laser interferometry techniques. The recovered samples were studied to characterize the deformation produced during impact loading. The changes on lattice orientation due to plastic deformation in the single crystal and in each grain of the bicrystal were mapped using Orientation Imaging Microscopy (OIM), with emphasis on the region surrounding the GB. Furthermore, dislocation structures were characterized using Transmission Electron Microscopy (TEM). All these measurements are correlated to the measured displacement and velocity distributions to quantify the deformation mechanisms and to identify GB effects. Research supported by Los Alamos National Laboratory.

11:15 AM
Constitutive Behavior of Metallic Materials at High Temperatures and Strain Rates: *Yellapregada V.R. K. Prasad*[1]; [1]Indian Institute of Science, Dept. of Metall., Bangalore, Karnataka 560012 India

The constitutive behavior of materials in the regime of high temperatures and strain rates is important in designing and optimizing metal working processes like drop forging, high speed extrusion and continuous rolling. In this regime, many of the metals and alloys exhibit flow instabilities like flow localization or intercrystalline cracking. However, low stacking fault energy materials like copper, nickel, some nickel base superalloys and Titanium alloys near their transus offer domains in which restoration mechanisms like dynamic recovery and recrystallization occur. Such domains may be exploited for high speed metal processing and for synthesizing new microstructures with some special properties. The thermally activation strain rate equations obtained in the high temperature and strain rate regimes are described for polycrystalline copper, nickel, IN718 superalloy, Ti-6Al-4V and Zinc alloys, along with the interpretation of the mechanisms involved. The impact of this search in the design and optimization of industrial metal working processes will be discussed.

11:30 AM
Analysis of Intergranular Impurities on the Ductility of Copper Shaped Charge Jets: *Adam J. Schwartz*[1]; Mukul Kumar[1]; Roger W. Minich[1]; [1]Lawrence Livermore National Laboratory, CMS, L-355, 7000 East Ave., Livermore, CA 94550 USA

A geometrical analysis based on the space filling tetrakaidecahedral grain shape is applied to determine the dependence of breakup time in S-doped of e-Cu shaped charge liners on grain size and bulk impurity content. The calculations determine the number of impurity atoms as a function of grain size, number of available sites at intercrystalline defects, and intercrystalline impurity concentration. Experiments have shown that some larger grain size liners with low impurity contents exhibit better dynamic ductility than smaller grain size liners with higher impurity concentrations. This suggests that the ductility of the liner jets is a complex function of microstructure and impurity concentration. Within the range of grain sizes and bulk impurity contents in this study, the analysis suggests that the quadruple nodes and triple lines are saturated with impurities, while only a partial filling of a monolayer of impurities is expected at the grain boundaries. However, if the impurity atoms are assumed to partition only to random grain boundaries, then it is observed that the coverage of such interfaces reaches one complete monolayer, which could perhaps explain the critical transition-like behavior in liner jet ductility. The approach suggests that breakup time is fundamentally related to grain boundary impurity segregation characteristics. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

11:45 AM
Twinning and Constitutive Response of HCP Aggregates: *Carlos N. Tome*[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-8, MS G755, Los Alamos, NM 87545 USA

Twinning is an important deformation mode in HCP materials, which strongly influences texture and hardening evolution during plastic forming. As a consequence, twinning contribution to texture and hardening has to be accounted for in constitutive descriptions of HCP aggregates. Since texture and twinning are strongly related to the crystallography, it is a necessary condition to use polycrystal models as a platform for describing twinning-related constitutive response in a general manner. Here we present a novel twinning model that accounts for directional barriers to dislocation motion posed by the twin lamellae in the grains, and also for the evolution of twin fraction with deformation in the grains. As an application, we simulate in-plane and through-thickness compression in rolled Zr, Be and Mg, and compare with available experimental data. Texture and stress-strain response are discussed in terms of twin fraction evolution. Constitutive response associated with strain path changes is also discussed.

Hot Deformation of Aluminum Alloys: Deformation, Recrystallization and Texture - I

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA; Armand J. Beaudoin, University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; Balasubramaniam Radhakrishnan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6359 USA

Monday AM — Room: 6E
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Hugh J. McQueen, Concordia University, Dept. of Mechl. & Industl. Eng., Montreal, Quebec H3G 1M8 Canada; Farghalli A. Mohamed, University of California, Dept. of Cheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA; Dorte Juul Jensen, Riso National Laboratory, Ctr. for Fundaml. Rsrch. Metal Struct. in Four Dimensions, Roskilde DK 4000 Denmark

8:30 AM Invited
Analysis of Recrystallization Kinectis from Microstructural Evolution and Micro-Hardness Determination: *Mohammed Haroon Alvi*[1]; Bassem Samy El-Dasher[1]; Anthony D. Rollett[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Recrystallization kinetics of hot rolled aluminum alloy is analyzed from the indirect method of micro-hardness test and direct method of microstructural evolution using Electron Back Scattered Diffraction (EBSD) in an SEM. Isothermally annealed samples of hot rolled aluminum alloy were studied using JMAK type analysis to see if there exists any correlation between the two methods on analysis, differing in the scale of observation. A good agreement between the two methods is clearly indicated by similar numerical values of kinetics parameters from the two methods.

8:55 AM Invited
Recrystallization Growth Rates in Hot Deformed Aluminum: *Dorte Juul Jensen*[1]; Roy A. Vandermeer[1]; [1]Riso National Laboratory, Ctr. for Fundaml. Rsrch., Metal Struct. in Four Dimensions, Matls. Rsrch. Dept., Roskilde DK 4000 Denmark

Growth rates of grains with different crystallographic orientations during recrystallization are determined using the extended Cahn Hagel metod. The material is alumimun deformed by plane strain deformation at 400C at a strain rate of 2.5s-1 to a strain of 2. It is found that grains of all orientations have similar growth rates and that the growth rates are almost constant during the entire recrystallization. The results are compared to cold deformation observations and are used as input for simulations of the microstructural development during recrystallization.

9:20 AM Invited
Fundamental Processes Responsible for Continuous Dynamic Recrystallization: An In Situ TEM Study: *Ian M. Robertson*[1]; Lisa

Dougherty[1]; John S. Vetrano[1]; [1]University of Illinois, Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA

Material from interrupted superplastic deformation tests of an Al-4Mg-0.3Sc alloy have been restrained in situ in the transmission electron microscope at nominally the superplastic forming temperature. In material predeformed to 0.2 true strain, the migration of subgrain boundaries, their interaction and trapping at Al3Sc particles, and their disintegration were observed dynamically. The dislocations released during the disintegration of the subgrain boundaries moved rapidly through the matrix and were incorporated into the bounding grain boundaries. The resulting increase in grain boundary energy caused rupture and annihilation of a grain boundary triple point. This process which was accompanied by a large volume rotation, resulting in a common orientation. The interaction of subgrain boundaries with Al3Sc particles and the bypass mechanism have also been observed. These observations will be discussed in relation to the macroscopic response of the material.

9:45 AM

Effect of Hot Deformation on Recrystallization Behavior of Al-4.5Mg Alloy: *Baolute Ren*[1]; [1]Alcoa Technical Center, Alloy Tech., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

In has been observed in commercial production of Al-4.5Mg alloy sheet product that hot rolling parameters, i.e., rolling strain, strain rate, and temperature, have important effects on the recrystallization behavior of the alloy, which affects the microstructure and texture of the hot band and mechanical properties and formability of the final products. In the present work, studies of recrystallization behavior of Al-4.5Mg alloy during hot rolling on the multi stands tandem mill were performed by using computer simulation. The well-known Johnson-Mehl-Avrami model was used, and the recrystallization kinetics at various rolling parameters was calculated. It was found that the rolling parameters directly influence the recrystallization during hot rolling of Al-4.5Mg alloy, which significantly affect the recrystallization texture development. The cube texture can be well developed for the hot line gauge material by properly controlled hot rolling, which significantly reduced mechanical property anisotropy and improved buckle strength of the final product.

10:05 AM

The Texture and Structure of Commercial Purity Aluminium After Hot Rolling: *Andrzej Klyszewski*[1]; Marzena Lech-Grega[1]; Wojciech Szymanski[1]; Janusz Zelechowski[1]; [1]Institute of Non-Ferrous Metals, Light Metals Div. in Skawina, ul. Pilsudskiego 19, Skawina 32-050 Poland

The influence of rolling temperatures on the texture and microstructure of 1050A aluminium alloy has been determined. The results confirm the relation between the hot rolling temperatures and structure parameters. Low temperature at the end of hot rolling process gave ìRî texture and fine grain size. Higher temperature at the end of hot rolling process leads to cube texture domination and coarse grain size.

10:25 AM

Evolution of Recrystallisation Texture and Microstructure in Hot Deformed AA3104: *Jean Savoie*[1]; Hang Yiu[2]; E. M. Lauridson[3]; L. Margulies[3]; S. F. Nielson[3]; S. Schmidt[3]; M. Ashton[4]; R. Sebald[5]; [1]Alcan International Ltd., Kingston R&D Ctr., 945 Princess St., PO 8400, Kingston, Ontario K7L 5L9 Canada; [2]Alcan International Ltd., Banbury Lab., Southam Rd., Banbury, Oxfordshire OX16 7SP UK; [3]Ris National Laboratory, Roskilde DK-4000 Denmark; [4]Manchester Materials Science Centre, Grosvenor St., Manchester M60 1QD UK; [5]Institut f¸r Metallkunde und Metallphysik, Kopernikus Strasse 14, Aachen D-5100 Germany

AA3104 samples were deformed under plane strain compression conditions. They were then annealed to achieve complete recrystallisation. Crystallographic textures, obtained from standard X-ray and EBSD methods, as well as microstructures, were characterised at various intermediate annealing times to follow their evolution, in particular for the cube-oriented grains. Two extreme sets of deformation conditions are considered, namely high-Z (low temperature and high strain rate) and low-Z (high temperature and low strain rate), which influence the cube development during both deformation and annealing. At high-Z, little cube was present in the as-deformed microstructure, but this then developed during recrystallisation. On the other hand, at low-Z, most of the cube present in the initial material survived the deformation, but then decreased its intensity during the subsequent annealing. In addition, 3D X-ray diffraction microscopy was utilised to characterise the growth of individual grains into the bulk of the samples deformed at the high-Z condition. The method of indexing individual grains allowed the determination of the average growth rate of cube grains for various annealing times.

10:45 AM

The Effect of Fe/Si Ratio on the Recrystallization Behavior After Thermomecanical Processing of AA1xxx-Alloys: *Stian Tangen*[1]; Trond Furu[2]; Erik Nes[1]; [1]Norwegian University of Science and Technology, Matls. Tech., Alfred Getz vei 2b, Trondheim 7491 Norway; [2]Hydro Aluminium, R&D Matls. Tech., Sunndalsra 6600 Norway

The deformation and annealing behavior in three AA1xxx-alloys after thermomecanical processing has been studied. The effect of different Fe/Si-ratio on the recrystallization behavior after both cold rolling and extrusion + cold rolling have been investigated. Following deformation the materials were isothermally annealed in order to recrystallize at different temperatures. The softening reactions during annealing were followed by means of hardness and electrical conductivity measurements. Recrystallized grain sizes have been measured both by the use of the Optical Light Microscope and the EBSD orientation mapping technique in SEM (OIM). The EBSD technique has also been used to study the texture development during the deformation and annealing of the materials. The particle size distributions after deformation were investigated with a FEGSEM equipped with an image analysis system.

11:05 AM

Effect of Alloy Chemistry on Microstructural Evolution During Deformation of Al Alloys: *Pankaj B. Trivedi*[1]; David P. Field[1]; Hasso Weiland[2]; [1]Washington State University, Sch. of Mech. & Matls. Eng., Pullman, WA 99164-2920 USA; [2]Alcoa Technical Center, Alcoa Ctr., PA 15069 USA

Various microstructural parameters that control the constitutive behavior of Al alloys include dislocation substructure evolution, cell morphology and misorientation angle between neighboring cells. A comparative study on microstructure evolution during tensile deformation of specific 3XXX, 5XXX and 6XXX series Al alloys has been made at relatively small strain levels (up to 10%). The microstructural characterization of deformed specimens was done using transmission electron microscopy and orientation imaging techniques. In general the density of dislocation substructure, grain orientation spread and grain average misorientation increased with deformation. No significant change in the subcell size was observed with deformation in any of the alloys studied. The effect of alloy chemistry and hence precipitate morphology, crystallite lattice orientation and character of neighboring grains on dislocation substructure evolution has been studied.

11:25 AM

Microstructural Evolution During Elevated Temperature Deformation of Strip Cast AA5754 Aluminum Alloy: Hamid Najjar-Azari[1]; Stephane Girard[1]; *David S. Wilkinson*[1]; [1]McMaster University, Matls. Sci. & Eng., Hamilton, Ontario L8S 4L7 Canada

The effect of rolling temperature and processing schedule on the microstructural development of twin-belt strip cast AA 5754 aluminum alloy was studied using microstructural and global texture analysis. The results indicate that rolling the as cast-material at about 350°C does not substantially affect the rolling or the subsequent recrystallization texture of the material when compared to the cold rolled material at equivalent rolling reductions. Under these conditions no dominant orientation emerges along the fl-fiber for reductions up to 90%, while the retained rolling and the rotated cube, {001}<310>, components dominate the recrystallization texture. Furthermore, the material was homogenized and hot (at 560°C)/cold rolled to study the effect on the recrystallized grain size, texture development and the mechanical properties. The results are discussed in the light of the starting as cast structure, the effect of Fe-rich constituent particles on the deformation and recrystallization textures as well as the effect of homogenization and elevated temperature deformation on the distribution of such particles.

11:45 AM

Grain Boundary Sliding and Grain Refinement in As-Cast 7475 Al Alloy Under Hot Deformation: *Rustam Kaibyshev*[1]; Oleg Sitdikov[2]; Alexandre Goloborodko[2]; Taku Sakai[2]; [1]Institute for Metals Superplasticity Problems, Khalturina 39, Ufa 450001 Russia; [2]The University of Electro-Communications, Dept. of Mechl. Eng. & Intelligent Sys., Chofu, Tokyo 182-8585 Japan

Hot deformation and microstructural changes in an as-cast 7475 Al alloy were studied in the temperature range of 400-520°C and at strain rates from 10^{-5} to 10^{-2} s^{-1}. The two deformation domains can be categorized due to different mechanical and microstructural behaviors. At high temperatures and low strain rates, when flow stresses lower than around 50 MPa, the σ vs ε behavior shows a significant strain softening just after yielding and a steady-state flow at strains of above 0.3. The structural changes are mainly characterized by development of deformation bands at an early stage of deformation, followed by new grain evolution during steady state flow due to operation of grain boundary sliding (GBS). Grain refinement occurs by a deformation-induced continuous reaction; that is continuous dynamic recrystallization. In the region of flow stresses higher than 50 MPa, in contrast, no GBS was found and a steady-state flow takes place in a relatively low strain. No grain refinement in layered original grains occurs even in large strain at such deformation conditions. GBS and grain refinement during high temperature deformation of the present 7475 Al alloy will be discussed in detail.

International Symposium on Gamma Titanium Aluminides: Application

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee

Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

Monday AM — Room: 6F
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Young-Won Kim, UES Inc., Matls. & Processes Div., Dayton, OH 45432 USA; Michael V. Nathal, John H. Glenn Research Center, Matls. Div., Cleveland, OH 44087 USA

8:30 AM Introduction

8:40 AM Invited

Establishing an Industrial Base for Wrought Processed Gamma (TiAl): *Heinrich Ewald Kestler*[1]; Nico Eberhardt[1]; Alexander Lorich[1]; Helmut Clemens[2]; Wolfram Knabl[1]; [1]PLANSEE AG, Tech. Ctr., Reutte, Tyrol 6600 Austria; [2]GKSS Research Centre, Inst. of Matls. Rsrch., Geesthacht D-21502 Germany

For more than a decade PLANSEE AG has been actively developing wrought processing techniques for gamma(TiAl). Starting from the first successful demonstration of sheet rolling of gamma(TiAl) from PM-Compacts in the late 90ís an industrial base for the production of a palette of semi-finished and finished components has been established. In this paper we will give a brief review of the activities in automotive and aerospace applications and will also describe the particular challenges which had to and still have to be overcome in certain steps of the processing chain, e.g. quality of precursor material, availability of adequate hot-forming equipment and machining. Additionally advances in joining will be addressed as well as the status in processing high Nb-content, high strength gamma(TiAl)-base alloys. Special emphasis will be put on the successful implementation of an industrial pilot plant for the production of high-performance gamma(TiAl) valves for racing engines. Using this example we will demonstrate that most of the challenges formerly foreseen in terms of material properties, conventional hot-forming, machining, quality and quality assurance in the industrialization were met. It will be shown that amongst these challenges the quality of gamma(TiAl) precursor material remains to be the most important issue and is regarded as having the main impact on furtherindustrial exploitation of gamma(TiAl) base alloys in other applications.

9:10 AM Invited

Gamma Titanium Aluminide and the Automotive Race Engine: *A. W. Sommer*[1]; [1]Del West Engineering, Valencia, CA USA

Gamma Ti-Al offers a very interesting solution when one is trying to enhance the design efficiency of reciprocating parts in an internal combustion engine. The attributes of highly elevated temperature strength/oxidation resistance combined with low density and highly elastic stiffness makes this material a good candidate for the poppet valves. Automotive intake and exhaust valve usage in auto racing presents quite a challenge for the materials specialist. The criteria that a material must meet in order to be selected for such applications will be reviewed in light of the mechanical and physical properties of the gamma aluminides as well as the materials with which they compete. The choices one has available for manufacturing methods to create the basic valve and properly protect it against sliding wear will be discussed in terms of both the structural integrity of the final product in this application and the difficulties associated with each method.

9:40 AM Invited

First Production Results from a Prototype Plant for Mass Production of Gamma TiAl-Valves: *Matthias Blum*[1]; Peter Busse[2]; Georg Jarczyk[1]; Henrik Franz[1]; Hans J. Laudenberg[3]; Klaus Segtrop[3]; [1]ALD Vacuum Technologies AG, R&D Vacuum Metall. & Process Eng., Wilhelm-Rohn-Strasse 35, Hanau 63450 Germany; [2]ACCESS e.V., Inzestrasse 5, Aachen 52072 Germany; [3]TRW Deutschland GmbH, Hannoversche Strasse 39, Barsinghausen 30881 Germany

In a joint research project - supported by the German federal ministry for education and research - a prototype production process for TiAl valves was developed by several automotive companies, an equipment supplier, a valve manufacturing company and research institutions. The project was initiated by ALD Vacuum Technologies AG. The first prototype plant was put into operation at ACCESS at Aachen in January 2002. Up to now about 200 melting and casting trials with 50 valves planks each were carried out under fully automatic process control. The quality assessment datas will be presented, such as LCF testing (at different temperature levels), x-ray inspection results, fracture toughness, metallographical inspection, deviations in chemical composition and engine testing work. The present manufacturing process for the as cast valves will be partly explained. Al work is now focused to demonstrate the manufacturing and casting capabilities to reach the price targets of the automotive industry. Therefore the quality inspection includes a wide variety of valves under different process parameters to identify the cheapest solution to fulfill all the requirements for automotive engine necessities.

10:10 AM

Cast Gamma Ti-Aluminide Shroud Support for ESPR: *Min Lu*[1]; James Barrett[1]; Tom Kelly[2]; [1]PCC Structurals, Inc., Matls. & Tech., 4600 SE Harney Dr., Portland, OR 97026 USA; [2]GE Aircraft Engines, 1 Nuemann Way, Cincinnati, OH 45215 USA

In early 2000, PCC and GEAE were teamed together to participate in an ESPR program funded by NEDO. ESPR stands for Environmentally Compatible Propulsion System for Next-Generation Supersonic Transport. This program aims at the research and development of technologies required for high speed transport propulsion system up to Mach 2.5. The targets of research include: reduce carbon dioxide emission by 25% as compared to the current jet engines; reduce nitrogen oxides emission to a level that is 1/7 of the current jet engines; and in the meantime, achieve significant noise reduction. Cast gamma titanium-aluminide is designated to produce a shroud support for weight reduction purpose. Engine-test quality pieces were successfully cast at PCC with a fixed production process. Subsequent machining, carried out by GEAE, yielded final products that have been delivered to the program. Engine test is scheduled to be completed by the end of 2002. This talk will mainly cover shroud supportís casting and machining processes . Engine test results will be shared pending on availability. Some other ESPR cast gamma activities will also be introduced.

10:30 AM Invited

Accelerated Insertion of Materials: The Challenges of Gamma Alloys, are Really not Unique: *Dennis M. Dimiduk*[1]; Patrick L. Martin[1]; Rollie Dutton[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Bldg. 655, 2230 Tenth St., WPAFB, OH 45433-7817 USA

The proceedings from the first International Symposium on Gamma Titanium Aluminides, held in 1995, begin with an article entitled ìGamma Titanium Aluminides: An Emerging Materials Technology.î Eight years have passed since that conference, and while much technical progress has been achieved with these alloys, it is clear that gamma alloys remain an ëemerging materials technology.í For this presentation we hypothesize that two principal reasons account for the protracted emergence of these alloys: I) a lack of engineering practices for low ductility materials in structural applications, and II) the inertia inherent in the current empirical approach to materials development. This has led to a profound disconnect between the systems design community having a three year product development cycle juxtaposed to the materials and processes development cycle that exceeds ten years. This discontinuity results from our inability to transform an understanding of limiting materials and processes attributes into decision making tools for design teams. While the first of these suggests the need for growth in several technical capabilities as well as processing technologies, the second is pervasive and influences even evolutionary advances within more common non-intermetallic alloys. This presentation examines historical practices for alloy development over the last ~40 years, discusses the current climate related to their continued use, and introduces a vision of the ëaccelerated insertion of materialsí within the context of gamma-alloy technologies. The presentation also addresses driving forces or ëproduct pullí for gamma alloys, especially as they currently exist within the US Air Force, and highlights selected critical needs for low-ductility materials.

11:00 AM Invited
Gamma Titanium Aluminides for Gas Turbine Engine ComponentsñA Review of Activities and Recent Accomplishments: *Kathleen A. Sargent*[1]; [1]Air Force Research Laboratory, Propulsion Direct., AFRL/PRTC, 1950 Fifth St., WPAFB, OH 45433-7251 USA

A major emphasis in the gas turbine engine industry historically has been to improve engine performance through advanced component designs that have higher temperature capability and reduced weight. This initiated a more extensive use of advanced materials throughout the engine, and an application of advanced materials in complex, innovative component designs. The US Air Force has conducted several research projects looking into the applicability of gamma titanium aluminides to a variety of components including compressor blades, shrouds, diffusers and disks. This paper will provide an overview of the work that has been completed and is ongoing for gamma titanium aluminide engine components.

11:30 AM Invited
A ìHow Toî Design Highlights with Gamma Titanium Aluminides: *Ron Isaac Prihar*[1]; [1]Pratt and Whitney, Compressor Sys. Module Ctr., Aircraft Rd., Middletown, CT 06457 USA

Pratt and Whitney has recently been involved in the development and testing of Titanium - Aluminide (TiAl) materials on its High Pressure Compressor rotors and airfoils. Titanium -Aluminide materials are being considered due to their higher specific strength and stiffness at temperatures beyond conventional nickel based superalloys. As with all new materials the TiAl cannot be designed as if it were a typical titanium or nickel part. The following must be taken into consideration when designing and planning for production incorporation with structural intermetallics: ∑Material processing ∑Casting ∑Forging ∑Material structural properties ∑Ease of Manufacturing ∑Creating Components out of Castings ∑Creating Components with Forgings Material machining sensitivity as regards to geometry ∑Material machining requirements: ∑Tooling allowables ∑Machining Speeds. Each component has its issues related to the specific application which must also be addressed: ∑Rotors ìNotch sensitivityî or ìhow do you handle rotor attac hmentî ∑Airfoils ìTip Rubsî or ìhow will the material respond to hard rubs on abradable materialsî. The following paper will discuss these issues and others based on Pratt & Whitney experiences. The paper will also provide recommendations and ìsanity checksî when dealing with new structural intermetallic materials such as TiAl.

12:00 PM
Mechanical Surface Treatments for Enhancing Fatigue Performance of Gamma Titanium Aluminides at Ambient and Elevated Temperatures: *Lothar Wagner*[1]; Janny Lindemann[1]; Dan Roth-Fagaraseanu[2]; [1]BTU Cottbus, Physl. Metall. & Matls. Tech., PO Box 101344, Cottbus 03013 Germany; [2]Rolls-Royce Deutschland, Ltd. & Company KG, Eschenweg 1, Dahlewitz 15827 Germany

Gamma titanium aluminides are attractive candidates for applications where high specific strength and stiffness at elevated temperatures are required. Previous work has shown that surface strengthening by mechanical surface treatments can be used to improve the fatigue performance. In the present investigation, rotating beam as well as axial fatigue tests on smooth (k_t = 1.0) and notched (k_t = 1.7 and 3.3) specimens were performed in fully reversed loading (R = -1) at ambient and elevated (T = 650∞C) temperatures. Shot peening, roller-burnishing and deep rolling using a wide variation in process parameters such as Almen intensity and rolling force were utilized to optimise fatigue performance. Fatigue results were compared with electrolytically polished conditions serving as reference. The improvement of the fatigue performance caused by mechanical surface treatments will be correlated to process-induced roughnesses, dislocation density and residual stress-depth profiles and their effects on fatigue crack nucleation and microcrack growth.

12:20 PM
Processing, Microstructure and Tensile Properties of Gamma TiAl PM Alloy 395MM: *Ulrike Habel*[1]; Gopal Das[2]; C. Frederick Yolton[1]; Young-Won Kim[3]; [1]Crucible Research, 6002 Campbells Run Rd., Pittsburgh, PA 15201 USA; [2]Pratt & Whitney, 400 Main St., MS 114-40, PO Box 109600, E. Hartford, CT 06108 USA; [3]UES, Inc., 4401 Dayton Xenia Rd., Dayton, OH 45432-1894 USA

The microstructure and mechanical properties of argon gas atomized gamma TiAl Alloy 395MM, Ti-46Al-3.7 (Nb, Cr, Mo)-0.4 (B, C) (in at%), consolidated by hot isostatic pressing (HIP) are being evaluated for potential applications in gas turbine engines. Powders with sizes <150 μm were HIPíed to a fully dense billet with a fine near-gamma microstructure. Part of the billet was upset forged in the alpha+gamma field to a fine-grained pancake by 85% reduction. Both HIPíed and forged materials were heat treated in the alpha field to produce fully lamellar (FL) microstructures. The effects of process parameters on the evolution of microstructures were determined using various microscopy, x-ray diffraction, DTA analysis. Preliminary tensile tests measured RT strengths of 735 MPa for the HIP condition and 650 MPa for the HIP + forged condition, and low ductility (< 0.2%). The FL heat treatment was shown to alter their tensile properties significantly: Strength levels were lowered to 585 MPa, but the ductility levels i ncreased to above 1% in both materials. Possible interrelationships among powder morphology, chemical homogeneity, microstructures and mechanical properties will be discussed.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C.T. Liu: Intermetallics I–Titanium Aluminide

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Seetharama C. Deevi, Philip Morris USA, Research Center, Richmond, VA 23234 USA; Fritz Appel, GKSS Research Centre, Geesthacht D-21502 Germany; Robert W. Cahn, University of Cambridge, Materials Science and Metallurgy, Cambridge CB2 3QZ UK; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Guo Liang Chen, University of Science and Technology-Beijing, State Key Laboratory for Advanced Metals and Materials, Beijing 100083 China; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208-3108 USA; Shuji Hanada, Tohoku University, Sendai 980-8577 Japan; Linda Horton, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6132 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Dong-Liang Lin, Shangai Jiao-Tong University, Shangai 200030 China; T. G. Nieh, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; Masaharu Yamaguchi, Kyoto University, Department of Materials Science & Engineering, Kyoto 606-8501 Japan

Monday AM — Room: 8
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Seetharama C. Deevi, Philip Morris USA, Rsrch. Ctr., Richmond, VA 23234 USA; Linda L. Horton, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6132 USA

8:30 AM Introduction by Dr. S. C. Deevi

8:40 AM Invited
A Long-Lasting Collaboration: *Robert W. Cahn*[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ England

The author and Chain Liu have been closely acquainted for almost twenty years, and have collaborated in a range of ways. We began by sharing an interest in order-disorder transformations and went on to share in the organization of a range of conferences in various parts of the world. I have spent some time for research at Oak Ridge, benefiting from Dr. Liuís wide-ranging expertise. Then, in 1992, we joined forces, together with two other colleagues, to create a new journal devoted (like this Conference) to intermetallics; this has been an exhilarating adventure. I look forward to personally paying tribute to Dr. Liu in San Diego.

9:05 AM Invited
Dislocation/Twin/Interface Interactions During Deformation of PST TiAl Single Crystals: *David P. Pope*[1]; [1]University of Pennsylvania, Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

PST TiAl crystals have been deformed in compression such that the deformation axis lies in the (111) interfacial planes, producing so-called ìchanneled flowî. The sample simply shortens axially and spreads laterally in the channels defined by the (111) interfacial planes with zero strain perpendicular to the lamellae. We have observed how the deformation processes interact with the boundaries using AFM and find that both the macroscopic displacement vector and the total shear vector in each layer lies in the lamellar boundaries. However the deformation bands are of very different character. They consist of either just super dislocations or just ordinary dislocations. But others consist of a special combination of twinning and ordinary dislocations in fixed ratio, such that the net shear vector lies in the boundary, even though the individual twinning and dislocation shear directions are inclined to it. This complex interaction results in completely ëchanneledí flow. We have also shown that the cooperative twi nning and slip is homogeneous on the nano-scale, i.e., the twinning and slip occur in the same volume of material.

9:25 AM Invited

Physical Aspects of Hot-Working of Gamma-Based Titanium Aluminides: *Fritz Appel*[1]; Michael Oehring[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str., Geesthacht D-21502 Germany

Castings of gamma-based titaninium aluminide alloys suffer from shrinkage porosity, segregation of alloying elements, texture, and coarse microstructure. Attaining chemical homogeneity and refinement of the microstructure are therefore the most important prerequisites for engineering applications. To this end large effort has been expanded to establish wrought processing for these materials. The applied techniques bear a number of similarities to the processing of conventional materials, however, many elements of processing have to be adjusted to the constraint set by the ordered structure of intermetallic phases. These involve (i) a significant plastic anisotropy in the deformed state, (ii) low dislocation mobility, (iii) low diffusivity and grain boundary mobility, and (iv) high brittleness. These factors will be addressed by analysing mechanical data of forging operations and the obtained microstructures. Particular emphasis is placed on hot working of novel TiAl alloys with enhanced high-temperature capability.

9:45 AM Invited

Stabilization of Lamellar Structure of TiAl PST Crystals During Creep: *Kouichi Maruyama*[1]; Hee Y. Kim[2]; [1]Tohoku University, Dept. of Matls. Sci., Aoba-yama 02, Aoba-ku, Sendai 980-8579 Japan; [2]University of Tsukuba, Inst. of Matls. Sci., 1-1-1, Ten-nodai, Tsukuba 305-8573 Japan

Degradation of lamellar structure in PST crystals of a TiAl alloy was studied to discuss how to stabilize their lamellar structures during creep. Stability of the lamellar structure is crucial for creep resistance at high temperatures, but degradation of the lamellar structure is unavoidable in as-grown PST crystals. Coarsening of lamellar spacing and spheroidization of lamellae are the major degradation events, and both of them occur as a consequence to lamellar boundary migration. The lamellar structures of TiAl alloy contain four types of lamellar boundaries. Among the four types of boundaries, gamma/alpha2 boundary has the highest stability, and sustains the original lamellar structure even after a large amount of creep deformation. On the basis of the TEM analysis, a lamellar structural design concept will be proposed to make a stable lamellar structure with high creep deformation resistance.

10:05 AM Break

10:25 AM Invited

Grain Refinement in Gamma TiAl Wrought Alloys: *Young-Won Kim*[1]; [1]UES, Inc., Matls. & Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Gamma alloys consist of g-TiAl dispersed with a2 and B2 phases. Depending on their distribution, there form numerous types of microstructures, which are grouped into duplex, nearly lamellar and fully-lamellar (FL) types. Due to their greater fracture-toughness and resistance to elevated-temperature deformation/fracture, FL material is favored for engineering applications. Since annealing of wrought alloys for FL microstructures is conducted in the single phase alpha field, the resulting lamellar grain sizes are large (often greater than 300 µm) and widely distributed. This has been a major concern in gamma alloys in terms of tensile properties and damage tolerance, and naturally a great deal of investigation has been made in refining lamellar grains. Over the last eight years, we have established two types of grain refining methods for wrought gamma alloys. One is to add small amounts of boron which result in refined lamellar grains in hot-worked material upon alpha annealing and subsequent cooling. The secon d methods are to process the material in the alpha field, that can produce in-situ lamellar structures having grain sizes as fine as 30 µm. This presentation discusses our understanding of the grain-refining mechanism and the potential and limitations for each method.

10:45 AM

In-Situ TEM Observation of Interface Sliding in a Refined Lamellar TiAl Alloy: *Luke L. Hsiung*[1]; Adam J. Schwartz[1]; T. G. Nieh[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., 7000 East Ave., L-352, PO Box 808, Livermore, CA 94551-9900 USA

We have previously reported that interface sliding with a cooperative motion of interfacial dislocations plays a crucial role in the creep deformation behavior of refined lamellar TiAl. Since the multiplication and motion of lattice dislocations within alpha-two and gamma lamellae become largely restricted as a result of the refined lamellar microstructure, the deformation strain is mainly accommodated by the motion of pre-existing interfacial dislocations. It is also possible that the interfaces in lamellar TiAl could migrate directly through the cooperative motion of interfacial dislocations and lead to the coarsening of lamellar spacing. Therefore, shear-induced interface sliding could result in a weakening effect when lamellar TiAl with a refined microstructure is employed for engineering applications. Although it is anticipated that the interface-sliding phenomenon is more prevalent at elevated temperatures, the current investigation provides the direct evidence of the occurrence of interface sliding and migration within refined lamellar TiAl even strained at room temperature. This work was performed under the auspices of the US Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

11:00 AM Invited

Superplasticity in a Large-Grained TiAl Alloy: *Dongliang Lin*[1]; Feng Sun[1]; Jing Hu[1]; [1]Shanghai Jiao Tong University, Sch. of Matls. Sci. & Eng., & Open Lab. of Edu. Ministry of China for High Temp. Matls. & Tests, 1954 Hua San Rd., Shanghai 200030 China

The superplastic behavior was systematically investigated in a large-grained Ti-47Al-2Mn-2Nb-B alloy having nearly equiaxed •„ phase with grain size of 95•Ïm, in which a small amount of fine particles •·2 distribute uniformly. Superplastic deformation was examined at a temperature range of 1025 to 1100°… and a strain rate range of 4°ø10-5 to 1.28°ø10-3 s-1. The large-grained TiAl alloy exhibits all deformation characteristics of conventionally fine-grained superplastic alloys without the prerequisites of fine grain size and grain boundary sliding. All the values of strain rate sensitivity, m are larger than 0.3. In most cases, an elongation over 200% was gained. A maximum elongation of 287.5% with an m value of 0.39 was obtained at 1100°… and an initial strain rate of 4°ø10-5 s-1.Mirostructure evolution during superplastic deformation was characterized by optical microscopy (OM), orientation imaging microscopy (OIM) and transmission electron microscopy (TEM). Metallographic examination has shown that the average grain size of large-grained TiAl alloy decreased during superplastic deformation, after that a much finer grain size of 10 to 3~5 •Ïm could be obtained. Electron back-scattered diffraction analysis revealed that significant grain refinement was obtained at different deformation levels with an increase in the density of low angle and high angel grain boundaries. A direct evidence of dynamic formation of grain boundaries with misorientation of 15-30®¨ was found, which was evolved from subboundaries. The formation of subboundaries and the evidence of dislocation glide in the interior of grains were revealed by TEM observation. A continuous recovery and recrystallization process similar to that in FeAl and Fe3Al was proposed as superplastic deformation mechanism in the large-grained TiAl alloy.

11:20 AM

Grain Boundary Strain Accommodation and Microcrack Initiation in Gamma-TiAl: Benjamin A. Simkin[1]; Boon-Chi Ng[1]; *Thomas R. Bieler*[1]; Martin A. Crimp[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

The inability to consistently produce tensile ductilities greater than 2% continues to limit the applications of TiAl intermetallic alloys. In the present study, we have examined the role grain boundaries play in this limited ductility by examining the nature of deformation strain transfer and microcrack initiation in Ti-47.9Al-2Cr-2Nb, which is a near gamma equiaxed alloy. Electron Channeling Contrast Imaging (ECCI) has been used in conjunction with electron back scattered diffraction (EBSD) and selected area channeling patterns (SACPs) for imaging and quantifying defects in bulk crystals. Two experimental configurations have been used to carry out mechanical loading. In the first, ex-situ 4-point bending has been carried out to various strains, allowing the tensile surface to be examined for microcrack initiation sites. In the second configuration, in-situ notched 4-point bend specimens have been deformed until significant crack propagation has occurred to allow the nature of deformation at crack tips to be examined. In both cases twins and dislocations associated with cracking and grain boundary strain transfer have been identified using trace analysis based on measured crystal orientations. In all cases, it has been found that crack initiation occurs as a result of an inability to accommodate deformation twinning strain at grain boundaries. Analysis shows that a simple geometric assessment of the possible deformation systems on both sides of the grain boundaries, based on previous work by Luster and Morris, predicts active deformation systems well. However, good geometric compatibility has not been found to be a sufficient predictor of the ability to transfer strain and suppress microcracking at grain boundaries. This work has been supported by the Air Force Office of Scientific Research under grant F49620-01-0116 and the Michigan State University Composite Materials and Structures Center.

11:35 AM Invited

Infrared Brazing of TiAl Intermetallic Using Ag-Based Braze Alloys: R. K. Shiue[2]; *S. K. Wu*[1]; S. Y. Chen[1]; [1]National Taiwan University, Dept. of Matls. Sci. & Eng., Taibei 106 Taiwan; [2]National Dong Hwa University, Dept. of Matls. Sci. & Eng., Hualien 974 Taiwan

Titanium aluminides have been extensively studied due to their high specific strength, and fairly good corrosion as well as oxidation resistance. The development of joining process always plays a crucial role in application of these alloys. Infrared vacuum brazing is a novel technique featured with a rapid thermal cycle. The present work reports the successful infrared brazing of TiAl using pure Ag as brazing filler metal. The transient microstructural evolution in the joint as well as its bonding strength is extensively evaluated. The infrared brazed joint is primarily comprised of Ag-rich phase solid solution with Ti and Al. There are three phases in the reaction layer, including Ti(Al,Ag), Ti3(Al,Ag) and Ag-rich. The formation of Ti3Al in the reaction layer can be attributed to the dissolution of TiAl substrate into the Ag-rich molten braze. It is found that the Ag-rich phase dissolves much more Al than Ti. The consumption of Al can result in locally enrichment of Ti atoms, so Ti3(Al,Ag) phase is formed. T he use of pure Ag filler metal demonstrates excellent bonding strength of the joint. All specimens brazed at 1050∞C were fractured at TiAl substrate. Specimens brazed at 1100∞C above 60 seconds demonstrate shear strength up to 385 Mpa, however, the fracture location changes from TiAl substrate into the brazement. The fracture location is either at the Ag-rich matrix or at the interface between the reaction layer and Ag-rich matrix.

11:55 AM

Cathodic Arc Deposited Thin Film of Coatings Based on TiAl and Their Applications: *Sohini PalDey*[1]; Seetharama C. Deevi[1]; [1]Philip Morris USA, R&D, 4201 Commerce Rd., Gate C, Door 17, Richmond, VA 23234 USA

Intermetallics based on TiAl are being considered for a variety of structural applications due to their unique properties such as low density and high strength. Cathodes of TiAl are used to obtain thin film coatings of (Ti,Al)N to protect the tool materials from wear and abrasion. (Ti,Al)N coatings are essential for machining of hard metals and superalloys due to their high hardness and good thermal stability. In addition, they can be used for dry machining applications. In this paper, we summarize the influence of composition and processing variables of TiAl cathodes on the cathodic are deposition of (Ti,Al)N coatings. The hardness and adhesion properties of the coatings deposited on M2 steel were evaluated and correlated with the composition (Al/Ti ratio) of the coating. In addition, gradient (Ti,Al)N coatings were developed by varying bias voltage during the deposition of coatings. Our results indicate that the gradient coatings of (Ti,Al)N provide much better adhesion as compared to a single layer (Ti,Al) N coating containing a fixed amount of Al and Ti. We compare and contrast the status of structural and functional applications of TiAl, and show that the functional applications of intermetallics are at an advanced stage of industrial acceptance.

12:10 PM Invited

Microstructural Characterization and Creep Behavior of an As-Cast Ti-48Al-2V Alloy: M. Sujata[1]; *D. H. Sastry*[1]; C. Ramachandra[2]; [1]Indian Institute of Science, Dept. of Metall., Bangalore 560 012 India; [2]Hindustan Aeronautics, Limited, Central Matls. & Processes Lab., Foundry & Forge Div., Bangalore 560 017 India

Solidification paths were established in a two phase (α_2+γ) alloy of nominal composition Ti-48Al-2V (at.%). The room temperature microstructure of the alloy consisted of colonies of (α_2+γ) lath dendrites and interdendritic segregate. The (α_2+γ) lath colony size was determined to be about 76mm. Based on the dendritic morphologies and the orientation of the (α_2 /γ) lath striations observed in the shrinkage cavities, it was established that primary solidification occurs in the alloy. In the temperature range of 993-1098K over a stress range of 187-420 MPa, the alloy exhibited a power law creep behavior, with a stress exponent value of 4.0-4.6. The activation energy for creep was found to be in the range of 320-340kJ/mol. This is slightly higher than that for diffusion of Ti in single phase γ (TiAl). The results however suggest the operation of dislocation climb controlled creep mechanism in the alloy.

International Symposium on Structures and Properties of Nanocrystalline Materials: Theory and Simulations

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee

Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Monday AM	Room: 14B
March 3, 2003	Location: San Diego Convention Center

Session Chairs: Michael Baskes, Los Alamos National Laboratory, MST-8 MS G755, Los Alamos, NM 87545 USA; Pawel Keblinski, Rensselaer Polytechnic Institute, Matls. Sci. & Eng., Troy, NY 12180-3590 USA

8:30 AM Opening Remark

8:35 AM

Grain Boundary Diffusion Creep in Nanocrystalline Materials: *Pawel Keblinski*[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng., 110 8th St., MRC115, Troy, NY 12148 USA

Molecular-dynamics (MD) simulations are used to study grain-boundary diffusion creep of model nanocrystalline microstructures. Fully dense model microstructures, were grown by MD simulations of a melt into which small, randomly oriented crystalline seeds were inserted. In order to prevent grain growth and thus to enable steady-state diffusion creep to be observed on a time scale accessible to MD simulations (of typically 10-9 s), our input microstructures were tailored to (i) have a uniform grain shape and a uniform grain size of nm dimensions and (ii) contain only high-energy grain boundaries which are known to exhibit rather fast, liquid-like self-diffusion. Our simulations reveal that under relatively high tensile stresses these microstructures, indeed, exhibit steady-state diffusion creep with a strain rate that agrees quantitatively with that given by the Coble-creep formula.

9:05 AM

Hybrid Simulations with Finite Element Method and Molecular Dynamics for Nanocrystalline Ni: *Xiaodong Wang*[1]; Sung H. Whang[1]; [1]Polytechnic University, Dept. of Mechl. Eng., Six MetroTech Ctr., Brooklyn, NY 11201 USA

In nanostructured metals and alloys, viscous deformation at / above room temperature is mainly attributed to the grain boundary sliding. As the grain size approaches the critical size where dislocation activity is absent under practical loading conditions, the grain itself may be regarded as a purely elastic entity while the grain boundary may be considered active atomic aggregates, which carry out elastic and plastic deformation as a function of time. We present a computational modeling study on the creep behavior of nanocrystalline Ni doped by various interstitials such as C, S, and B. The key aspect of this modeling is to employ the molecular dynamics simulations for the grain boundaries, and the finite element calculations for the orthotropic grains. The matching between molecular and continuum descriptions is chosen to minimize the reflection of phonons at the atomistic and continuum boundaries. The major advantage of such a hybrid approach is the decoupling of both lengthscale and timescale. Thus, transient problems at the continuum timescale can be solved without expensive time integration at the molecular timescale.

9:35 AM

Deformation Mechanism in Nanocrystalline Ni:GB Sliding, Dislocations and Collective Processes: *Helena Van Swygenhoven*[1]; Peter M. Derlet[1]; A. Hasnaoui[1]; M. Samaras[1]; [1]Paul Scherrer Institute, NUM/ASQ, Villigen-PSI CH-5232 Switzerland

Molecular dynamics computer simulations of fully 3D-nc metals with mean grain sizes up to 20nm have shown that their exists a critical grain size below which there is no dislocation activity. Above this limit, partial dislocations start being emitted from the GBs. It is shown that GB sliding is triggered by atomic shuffling and stress assisted diffusion and that the nucleation and emission and the subsequent absorption of the partials in the opposite GBs is strongly related to the GB dislocations. It is also shown that collective grain motions occur forming shear planes that extend over a number of grains. (PRB 64 2001; PRB 66, 2002; Science April 4, 2002; Acta Mater. 2002 in press; Scripta Mater 2002 in press.)

9:55 AM Break

10:15 AM
Atomic Structure and Properties of Computer Quenched Nickel: *Frank J. Cherne*[1]; Michael I. Baskes[1]; Ricardo B. Schwarz[1]; Srivilliputher G. Srinivasan[1]; [1]Los Alamos National Laboratory, MST-8, Struct. & Prop. Rel., PO Box 1663, MS G755, Los Alamos, NM 87545 USA

Current interest in nanostructured devices have opened a number of questions regarding the structure-property relations of nanomaterials. Typical bulk material properties and processing techniques are require special consideration for the nanoscale. In this study, we explore the nucleation and growht of crystalline materials at the nanoscale. We perform molecular dynamics calculations using both embedded atom method and modified embedded atom method potentials to describe the properties of nickel. We start with a liquid and rapidly quench to a low temperature where the material microstructure is stable. We examine the effects of varying the quench rates (2.5 x 10^{11} to 10^{13} K/s) upon the microstructure and the stress-strain relationships of the material formed after quenching. At quench rates lower than 5 x 10^{12} K/s we observe homogeneous nucleation and growth of crystalline material whose microstructure depends upon the size of our system. At higher quench rates an amorphous solid is formed.

10:35 AM
Yield Stress of Nanocrystalline Materials: Role of Coble Creep: *Chandra Shekhar Pande*[1]; Robert A. Masumura[1]; [1]Naval Research Laboratory, Code 6325, Washington, DC 20375 USA

It is shown that modeling of strengthening by nanocrystalline materials need consideration of a mechanism involving both dislocation interactions and sliding due to Coble creep. Such a mechanism is considered in this paper. A model based on using Coble creep (with a threshold stress) for finer grains and conventional Hall-Petch strengthening for larger grains, appears to be most successful in explaining experimental results provided a grain size distribution is incorporated into the analysis to account for a distribution of grain sizes occurring in most specimens. The nature of Coble Creep in nanocrystalline materials is also discussed.

10:55 AM
Interfacial Strain Accomodation Mechanism at the Nano-Scale: *Moneesh Upmanyu*[1]; Balasubramaniam Radhakrishnan[1]; Gorti Sarma[1]; [1]Oak Ridge National Laboratory, Compu. Sci. & Math., PO Box 2008, MS 6359, Oak Ridge, TN 37831-6359 USA

The objective of this study is to develop a fundamental understanding of micro-mechanisms that control deformation of nano-scale grain boundary microstructures. The first objective is to understand the effect of size constraints on grain boundary structure. To this end, we have performed energy minimization studies of flat tilt/twist grain boundaries in pure Al as a function of simulation cell size. Detailed structural analysis is carried out to ascertain the sensitivity of the grain boundary structure in the sub-nanometer regime. The studies are performed for high symmetry grain boundaries with well established equilibrium boundary structures. The second objective of this study is to study deformation behavior of such flat bi-crystals well past the elastic regime, and to determine interfacial micro-mechanisms that compete with conventional dislocation based-strain accomodation. Besides simulation size, the deformation behavior is also studied as a function of degrees of freedom associated with the grain boundary, such as misorientation axis (tilt/twist boundary), degree of misorientation as well as inclination of the grain boundary. Structural analyses are performed at the boundary after deformation and used to explain the trends observed in the stress-strain curve associated with bi-crystal deformation. The deformation study is also extended to deformation of tri-crystals with specific grain boundaries.

11:15 AM
Atomistic Studies of the Fracture of Nanocrystalline Materials: *Diana Farkas*[1]; Antoine Latapie[1]; [1]Virginia Tech, Matls. Sci. & Eng., Blacksburg, VA 24060 USA

We describe atomistic crack propagation studies in nanocrystalline bcc iron samples. A combination of intragranular and intergranular fracture is observed. Mechanisms such as grain boundary accommodation, grain boundary triple junction activity, grain nucleation and grain rotation are observed to dictate the plastic deformation energy release. Intergranular fracture is shown to proceed by the coalescence of nanovoids formed at the grain boundaries ahead of the crack. The simulations also show that at an atomistic scale the fracture resistance and plastic deformation energy release mechanisms increase with increasing temperature.

11:35 AM
Study of the Mechanical and Electronic Properties of Semiconductor Heterostructures by Path Probability and Tight-Binding Molecular Dynamics Method: *Kinichi Masuda Jindo*[1]; Ryoichi Kikuchi[2]; [1]Tokyo Institute of Technology, Matls. Sci. & Eng., Nagatsuta, Midori-ku, Yokohama 226-8503 Japan; [2]University of California, Matls. Sci. & Minl. Eng., Berkeley, CA 94720-1760 USA

The atomic and electronic structures of semiconductor heterostructures including steps, misfit dislocations and interface disorder are studied by using the tight-binding molecular dynamics (TBMD) simulations. Atomic structures of misfit dislocations both edge type 1/2 <110> (001) and 60∞dislocations in the semiconductor heterostructures, like Si-Ge superlattices and GaAs/Si, InP/GaAs(001) systems are studied by using order of N [O(N)] calculational method. It is shown that the deep gap states associated with the misfit dislocations depend strongly on the atomistic configurations of the interface. The path probability method in the statistical physics is used to study the influence of the interface disorder on the electronic properties of the semiconductor heterostructures. It is shown that the junction relaxation influences quite significantly on the electronic and thermodynamic properties (e.g., critical layer thickness for generation of misfit dislocations) of semiconductor heterostructures.

Magnesium Technology 2003: Magnesium Melt Protection, Environmental and Recycling

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, LMD-Magnesium Committee, International Magnesium Association, MPMD-Solidification Committee
Program Organizers: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

Monday AM Room: 2
March 3, 2003 Location: San Diego Convention Center

Session Chairs: Nigel Ricketts, CSIRO, Mfg. Sci. & Tech. Queensland Ctr. for Adv. Tech., Kenmore, Queensland 4069 Australia; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

8:30 AM Special DedicationñByron Britt Clow, 1922-2002
R. Brown

8:45 AM
Various Techniques to Study the Surface of Magnesium Protected by SF_6: *Kari Aarstad*[1]; Gabriella Tranell[2]; G. Pettersen[2]; Thorvald Abel Engh[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., Alfred Getz vei 2B, Trondheim 7491 Norway; [2]SINTEF Materials Technology, Trondheim Norway

When magnesium is protected from oxidation and burning with SF_6 in air, a protective film is formed on the surface. The morphology, composition and kinetic growth of this film has been studied by heating magnesium under controlled atmospheres in a hot stage under an optical microscope. Pictures of the forming film were recorded continuously through a CCD camera connected to the microscope. In these experiments, magnesium film formation was studied at temperatures both below and above the melting point. The film formed on the sample was studied with microprobe, Focused Ion Beam Milling and Transmission Electron Microscope. The thickness of the film varied between 0.3 μm and 1 μm in these experiments, increasing with increasing temperature and holding time. Large spots or flakes containing magnesium fluoride may form on the film. These spots grew until they covered 25-50% of the total surface.

9:10 AM
Magnesium Melt Protection at Magnesium Elektron Using HFC-134a: *Paul Lyon*[1]; Philip D. Rogers[1]; John F. King[1]; S. P. Cashion[2]; Nigel Ricketts[3]; [1]Magnesium Elektron, Techl. Dept., PO Box 23, Swinton, Manchester M27 8DD England; [2]University of Queensland, PO Box 883, Kenmore, QLD 4069 Australia; [3]CSIRO, Mfg. Sci. & Tech., Queensland Ctr. for Adv. Tech., PO Box 883, Kenmore, Queensland 4069 Australia

Several workers are actively seeking alternatives to SF6. CAST/AMC have patented the use of a HFC gas HFC-134a. This gas has a GWP 95% lower than SF6. Magnesium Elektron (MEL) and CAST have collaborated on the use of HFC-134a to achieve successful production plant trials for ingot manufacture. This paper provides details and results of those plant trials at MEL. MEL ingot production is currently based on either SF6 or SO2 as active gas. Comparative evaluation of HFC-134a included: - development of gas mixing equipment - Distribution optimisation - Optimisation of concentration and flow rates - Assessment of breakdown products and potential implications. Results of over 150 production scale Mg-Al-Zn melts demonstrated that HFC-134a could offer equal protection to SF6 or SO2. Use of HFC-134a by MEL for commercial production is proposed.

9:35 AM
Melt Protection for the AJ52 Magnesium Strontium Alloy: *Donald L. Argo*[1]; Michel Lefebvre[1]; [1]Noranda, Inc. Technology Centre, Matls. Eng., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada

Melt protection of the AJ52 high temperature alloy developed by Noranda for use in applications requiring excellent creep and bolt load retention properties was investigated. While the alloy has been readily held at casting temperatures using standard cover gas protection protocols, it was decided look at the degree of protection provided by a wider array of cover gases. The investigation was performed using a laboratory scale enclosed furnace containing approximately 3 kg of molten AJ52 alloy. A typical protective gas distribution ring over the melt was used to introduce various cover gas combinations in terms of flowrate and concentration. The cover gases investigated extensively included SF6/air, SF6/air/CO2, SF6/CO2, SO2/nitrogen, SO2/air. As well, some of the newer cover gases being proposed as alternatives to SF6, such as HFC134a and Novec 612, were evaluated on a more limited scale. The paper presents the results of the investigation and the most appropriate methods of providing cover gas protection for the AJ52 high temperature alloy.

9:50 AM
Measured SF6 Emissions from Magnesium Die Casting Operations: *Scott Charles Bartos*[1]; Jerry Marks[2]; Ravi Kantamaneni[3]; Curtis Laush[4]; [1]US Environmental Protection Agency, Climate Protection Partnerships Div., 1200 Pennsylvania Ave. NW (6202J), Washington, DC 20460 USA; [2]J. Marks and Associates, 312 NE Brockton Dr., Lees Summit, MO 64064 USA; [3]ICF Consulting, 1850 K St. NW, Ste. 1000, Washington, DC 20006 USA; [4]URS Corporation, 9400 Amberglen Blvd., Austin, TX 78720 USA

The US Environmental Protection Agency (EPA) is collaborating with magnesium producers, casters, and recyclers to reduce emissions of sulfur hexafluoride (SF6), a potent heat trapping pollutant. SF6 is commonly used to protect molten magnesium from oxidation. Through EPAís voluntary partnership, US partner companies have diligently measured and reported their SF6 use for three years. The current method used to estimate SF6 emissions from magnesium operations assumes that all SF6 used in a given year is emitted to the atmosphere. The study presented in this paper seeks to determine how much SF6 reacts with the molten metal (i.e., is ìconsumedî) in a typical magnesium die casting operation, and therefore is not actually emitted to the atmosphere.

10:20 AM Break

10:35 AM
Technologies for Efficient Mg-Scrap Recycling: *Gerhard Hanko*[1]; Gernot Macher[2]; [1]University of Leoben, Non-Ferrous Metall., Franz-Josef Strasse 18, Leoben, Styria 8700 Austria; [2]Ecka Granules-Non Ferrum, Mg-Recycling, Buermooser Landesstrasse 19, St. Georgen, Salzburg 5113 Austria

Currently, only high grade clean Mg-scrap without impurities can be recycled easily into high purity alloys. More complex handling is required for old magnesium-base or postconsumer scrap as automotive parts and electronic devices. The additional process steps determine the economical attractiveness of Mg-recycling. This article will provide a detailed overview of the current research activities of Ecka Granules-Non Ferrum in cooperation with the department of non-ferrous metallurgy at the University of Leoben concerning different Mg-Recycling technologies. Aim of the investigations is the optimization and implementation of refining methods in consideration of metal loss reduction.

11:00 AM
Chemical Composition and Cleanliness During Recycling of the AJ52 Magnesium Strontium Alloy: *Donald L. Argo*[1]; Peter Forakis[1]; Michel Lefebvre[1]; [1]Noranda, Inc. Technology Centre, Matls. Eng., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada

Chemistry and cleanliness control are of paramount importance during recycling of alloys. While it is known that the Sr level of the AJ52 melt is stable, this paper looks at the stability of the various alloying constituents in the AJ52 alloy in regard to repeated re-melting and melting scrap with prime alloy. The control of iron levels in relation to Mn concentrations and temperature is discussed and methods of adding ìmakeupî levels of Al, Mn, Sr outlined with the types of recoveries that can be expected. As well, whenever scrap is recycled cleanliness becomes an issue and methods of refining recycled AJ52 material ranging from the use of fluxes (normally to be avoided with AJ52 alloys) to argon sparging are discussed and the results of cleanliness measurements are presented.

11:25 AM
The Oxidation of Magnesium Alloys in Solid and Semisolid States: *F. Czerwinski*[1]; [1]Husky Injection Molding Systems Ltd., Dvlp. Eng., Bolton, Ontario L7E 5S5 Canada

A combination of thermogravimetric measurements and microscopic analysis was used to assess the oxide growth on a solid and semisolid Mg-9%Al-1%Zn alloy. It was revealed that the alloy exhibits protective or nonprotective oxidation depending on the temperature and exposure time. The growth mechanism and morphologies formed at various stages are analyzed.

Martensitic Transformations in Low Symmetry Materials - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Robert D. Field, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Richard W. Fonda, Naval Research Laboratory, Washington, DC 20375 USA

Monday AM — Room: 7B
March 3, 2003 — Location: San Diego Convention Center

Session Chair: Robert D. Field, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

8:30 AM Invited
Theories of the Crystallography of Twinning and Martensitic Transformations and Their Application to Low Symmetry Materials: *Alan Crocker*[1]; [1]University of Surrey, Dept. of Physics, Guildford, Surrey, GU1 2SB UK

The year 2003 marks the fiftieth anniversary of the publication by Wechsler Lieberman and Read of their classic paper on the crystallography of martensitic transformations. This was soon followed by the publication of other versions of the theory all of which incorporated a lattice invariant shear, particularly twinning. The 1950ís also saw the development of several theories of the crystallography of twinning. However, these theories were mainly aimed at obtaining a better understanding of twinning and martensitic transformations in relatively simple crystal structures and could not readily be applied to complex materials, and in particular to the actinides. New versions of the theories based on a more powerful mathematical notation were therefore developed. In this presentation the basic assumptions and formalisations of the theories will be summarised, examples given of their application to several materials of low crystal symmetry and some suggestions made about further developments which are desirable.

9:05 AM
Dislocation Model of Martensite Transformation: Yaw-Wang Chai[1]; J. P. Hirth[2]; *Robert C. Pond*[1]; [1]University of Liverpool, Matls. Sci. & Eng., Fac. of Eng., Brownlow St., Liverpool, Merseyside L69 3BX UK; [2]Retired, 114 Ramsey Canyon Rd., Hereford, AZ 85615 USA

Current understanding of martensitic transformations is based on the phenomenological theory of martensitic crystallography (PTMC). However, experimental observations suggest that martensite habit planes are comprised of terrace-plane islands separated by an array of transformation dislocations, or disconnections, and a second array of lattice-invariant defects, mainly slip dislocations or twins. In this paper, we use dislocation theory to evaluate the properties of the two types of arrays. In particular, we explore the equivalence of the PTMC and defect model for identical transformations. It will be demonstrated that the dislocation model is consistent with diffusionless transformation accompanying interface motion, and that no long-range stress field arises. Experimental observations obtained using TEM which corroborate the defect model of martensitic interfaces will be reviewed.

9:25 AM
Investigation of Differences in Morphology and Transformation Mechanisms of Alphaí Formed from Delta Plutonium at Low Temperatures: *Jeffery J. Haslam*[1]; Mark A. Wall[1]; Kerri J.M.

Blobaum[1]; Adam J. Schwartz[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., PO Box 808, Livermore, CA 94551 USA

The existence of ìdouble C-curvesî in the TTT diagram of the delta to alphaí transformation of delta-stabilized plutonium has been reported previously. We are undertaking a systematic study aimed at elucidating the underlying crystallographic, morphological, and mechanistic differences between the upper and lower C-curves. Applying both isothermal and continuous cooling treatments, we observe morphological differences in alphaí particles in Pu - Ga alloys. For transformations in the upper C-curve, the morphology of the alphaí particles is lenticular and suggests a preference for maximum separation between particles. At lower temperatures, the transformation is confined to the grain boundary regions, which suggests grain boundary nucleation. In this regime, alphaí appears to form as narrowly spaced packets of thinner plates with parallel alignment. We will present optical and transmission electron microscopy observations and discuss possible mechanisms that could produce the observed morphologies. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

9:45 AM
Lattice Stability of FCC δ-Phase Plutonium Alloys: *Luis A. Morales*[2]; Andrew C. Lawson[1]; Siegfried S. Hecker[3]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS H805, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Nucl. Matls. Tech. Div., MS G721, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G754, Los Alamos, NM 87545 USA

The addition of a few atomic percent of Al or Ga retains the high-temperature FCC δ-phase of plutonium to room temperature and below. However, it is now agreed that the FCC δ-structure is not the equilibrium phase at room temperature. Under certain conditions of cooling or under modest isostatic pressures, it transforms martensitically to a monoclinic αí- phase that is closely related to the low-temperature phase of the unalloyed element. We report on recent diffraction experiments that show small crystallographic distortions of the FCC δ-phase to tetragonal or orthorhombic structures. These distortions occur when the δ-phase is cooled, but the αí-phase is somehow suppressed, and also at room temperature when there is a combination of microstress induced by spatial inhomogeneity of the solute element and/or the effects of long-term self-irradiation damage.

10:05 AM Break

10:20 AM
3D Phase Field Model of Low-Symmetry Martensitic Transformation in Polycrystal: Simulation of $\zeta í_2$ Martensite in AuCd Alloys: *Yongmei M. Jin*[1]; Andrei Artemev[2]; Armen G. Khachaturyan[1]; [1]Rutgers University, Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA; [2]Carleton University, Mechl. & Aeros. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada

A 3D Phase Field model of the martensitic transformation that produces a low symmetry phase in polycrystals is developed. The transformation-induced strain mostly responsible for the specific features of the martensitic transformation is explicitly taken into account. The high computational efficiency of the model turns out to be almost independent on the complexity of the polycrystal geometry. An example of the cubic->trigonal transformation in AuCd alloys producing $\zeta í_2$ martensite is considered. The development of the transformation through nucleation, growth and coarsening of orientation variants is simulated for both single crystal and polycrystalline materials. The effect of an external load on the martensitic microstructure in the polycrystalline material is studied. It is shown that the elastic coupling between different transformed grains of the polycrystal drastically affects the microstructure and its response to the applied stress. The obtained self-accommodating morphologies of the multivariant martensitic structure are in agreement with those observed in the experiments.

10:40 AM
Martensitic Transformations in B2 Cubic Alloys: *Jason C. Lashley*[1]; Timothy W. Darling[2]; Jason C. Cooley[3]; Dan J. Thoma[3]; [1]Los Alamos National Laboratory, MST-8, MS G770, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-10, MS K764, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, MST-6, MS G770, Los Alamos, NM 87545 USA

Many modern shape-memory materials are based not on FCC ìausteniteî as in steel, but on body-centred cubic structures, in particular the B2 structure alloys like NiTi. Body-centred structures may be subjected to a shearing motion to which the material has very little resistance. Plausibly, this ìZener instabilityî can provide the means for these structures to undergo a martensitic phase transition, where the actual distances the atoms move are small enough that exact reversibility (shape-memory effect) is possible. There are many B2 structures, but only some of them transform. Some are equi-atomic, highly ordered alloys and some are off-stoichiometry that must be quenched to retain a non-equilibrium B2 structure. We have made thermal, transport, mechanical and elastic measurements on several B2 martensitic alloys and present our findings in terms of the differences between various B2 alloys and between B2 and other structures.

11:00 AM
Determining Martensitic Transformation Mechanisms by Coupling Symmetry Principles: *S. G. Srinivasan*[1]; D. M. Hatch[2]; H. T. Stokes[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Brigham Young University, Physics & Astron., Provo, UT 84602 USA

Determining mechanisms of martensitic transformations (MT) is a challenging proposition even for high symmetry materials. We have developed a systematic, general, and robust methodology to determine mechanisms in reconstructive MT by coupling crystallographic group-theoretical arguments to atomistic energy calculations. Our symmetry-based (criteria imposed) algorithm identifies a small number of subgroup candidate mechanisms. Our approach is to then use the interatomic forces and energies obtained from atomistics, the so-called ìphysicsî insight, to interrogate the various symmetry pathways obtained from the symmetry algorithm. Since both steps of the procedure are amenable to computer automation, we can rapidly identify the minimum energy path(s) between any of the 230-crystallography space-groups. This method successfully obtained a generalization of the classical Burgers mechanism for a pressure driven BCC to HCP transformation in titanium. We outline the use of our approach to the study of MT in low symmetry materials.

11:20 AM
A New Mechanism for HCP-Omega Transformation in Pure Titanium: *D. R. Trinkle*[1]; D. M. Hatch[2]; S. G. Srinivasan[3]; R. G. Hennig[1]; H. T. Stokes[3]; R. C. Albers[4]; J. W. Wilkins[1]; [1]Ohio State University, Physics Dept., Columbus, OH 43210 USA; [2]Brigham Young University, Physics & Astron., Provo, UT 84602 USA; [3]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [4]Los Alamos National Laboratory, T-11, MS B262, Los Alamos, NM 87545 USA

The pressure driven HCP-Omega transition in pure titanium is a reconstructive martensitic transformation (MT). It has often been suggested that this occurs via the classical Silcock mechanism. Using a systematic, general, and robust methodology, obtained by coupling crystallographic group-theoretical arguments to atomistic (both tight-binding and ab-initio) calculations, we interrogated the various pathways determined by our symmetry algorithm. We find, for the first time, direct evidence for a mechanism that can be visualized as a combination of hcp first transforming to bcc via a generalized Burgers mechanism followed by the collapse of two out of three bcc (111) planes to yield the omega phase. This ìcompositeî mechanism, however, still appears as a single transformation. This is consistent with, (a) the mechanism postulated for pure titanium using just the orientation-relationships, and (b) the intermediate bcc phase observed experimentally during the hcp-omega transformation in Ti-V alloys.

Materials and Processes for Submicron Technologies - III: Advances in Microelectronic and Photonic Materials and Processes

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee, TMS
Program Organizers: Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ USA; Mahesh Sanganeria, Novellus Systems, Inc., San Jose, CA 95134 USA

Monday AM — Room: 15A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: N. M. (Ravi) Ravindra, New Jersey Institute of Technology, Dept. of Physics, Newark, NJ 07102 USA; Jun-Ho Choy, Simon Fraser University, Dept. of Physics, Burnaby, British Columbia V5A 1S6 Canada

8:30 AM Invited

Light Emission in SiliconñSome Perspectives: *Anthony T. Fiory*[1]; Aravind Balakrishnan[1]; N. M. Ravindra[1]; [1]New Jersey Institute of Technology, Dept. of Physics, Newark, NJ 07102 USA

Abstract not available.

9:00 AM Invited

Effect of Amorphous-to-Crystalline Transformation of Si on the Redistribution of NiSi2: *Chel-Jong Choi*[1]; Tae-Yeon Seong[1]; Hua Gan[2]; Grant Pan[2]; K. N. Tu[2]; [1]Kwangju Institute of Science & Technology, Dept. of Matls. Sci. & Eng., Kwangju 500-712 Korea; [2]University of California-Los Angeles, Dept. of Matls. Sci. & Eng., Los Angeles, CA 90095-1595 USA

We implanted a high dose of Si and a low dose of Ni consecutively into (001) Si wafers to form a near surface layer of amorphous Si containing the implanted Ni on the Si. A post-implantation annealing at 450 to 550°C leads to the formation of randomly oriented NiSi2 precipitates in the amorphous Si. Then an additional annealing at 650 to 950°C transforms the amorphous Si into crystalline Si. In this two-step annealing, we do not find embedded NiSi2 in the crystalline Si, rather that the NiSi2 moves to the surface of the Si. Images of plan-view and cross-sectional view of TEM will be presented to describe the silicidation behaviors.

9:30 AM

Condensation of Carbon Vapor in the Absence of a MW Heating: Oxana Vasilievna Kharissova[1]; *Juan Antonio Aguilar*[2]; Eder Zavala[1]; Ubaldo Ortiz[2]; [1]University of Nuevo Leon, Facultad Fisico Mamematicas, Ciudad Universitaria, F-101, San Nicol·s de los Garza, Nuevo LeÛn 66450 MÈxico; [2]University of Nuevo Leon, FIME, Cuidad Universitaria, Pedro de Alba s/n, San Nicolas de los Garza, Nuevo LeÛn 66450 MÈxico

Processing of carbon nanotubes by graphite vaporization with microwaves as an energy source (800W and 2.45GHz) under two conditions, air and high vacuum (10-4Torr), for 20-90 min was studied. The oven temperature was approximately 1200°C. The condensed material was collected on a target of quartz. The samples were then characterized and studied by Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), and X-ray diffraction. Thin films were studied by AFM in contact mode at room temperature, varying both scanning frequency and force. The formation of multiwalled carbon nanotubes (MWCNs) in the samples was detected. Layer angle, height and distance of MWCNs were measured. An important amount of nanotubes, nanoparticles and fibers, which appeared to be highly graphitized and yet structurally perfect, was also found in the samples.

9:50 AM

Photoelectrochemical (PEC) Characterization of Screen-Printed Composite Electrodes in Conjunction with Aqueous Methylene Blue/Fe(II) Electrolyte: *I. S. Bayer*[1]; I. Eroglu[2]; L. Turker[2]; [1]University of Illinois at Chicago, Dept. of Mechl. Eng., Chicago, IL 60607 USA; [2]Middle East Technical University, Dept. of Cheml. Eng., Ankara 06531 Turkey

Semiconductor photo-electrochemistry has wide range of applications like etching, photo-etching, anodic oxidation, surface passivation and semiconductor characterization. Development of such materials whose electrical and optical properties could be controlled will be useful in many ways. In energy conversion, semiconductor/electrolyte interface may be used for photo-electrolysis, photo-catalysis and charge storage in addition to photo-electrochemical power generation. There are numerous physical and chemical ways of growing composite layers on various substrates. In this study, the electrodes were fabricated by screen-printing method. Screen-printing method is a process to manufacture ceramic films. The cell design composed of various (150 to 250 microns) porous ceramic films (TiO2, ZnO, WO3, PbO and Sb2S3) hydraulically pressed and printed on nickel (Ni) mesh substrates with liquid polytetrafluoroethylene as a polymeric binder matrix. The methylene blue/Fe(II) electrolyte is contained in the pores of the electrode. The counter electrode was an activated carbon composite fabricated in a similar manner. Photo-electrochemical (PEC) properties of these cells were studied in solutions at different pH values. Transient photo-voltage data were used to calculate chemical reaction kinetic rate constants. The current-voltage characteristics in the dark and under illumination were also studied. Figure 1 (a) shows the transient open-circuit photo-voltage characteristics (rise and decay curves) for five different cell configurations. The inset to figure 1(a) displays log(Voc) versus time during decay of photo-voltage. Photo-voltage excitation by light (700 W m-2) was very quick (2.3 min) whereas voltage decay stabilized in 40 minutes. The following mathematical relation was used to represent the decay kinetics of photo-voltage: Vdecay=Vsd exp(-t/Ù+C) where Vsd, t, Ù, and C are steady-state decay voltage, time, relaxation time constant and arbitrary constant respectively. The slopes obtainable from the inset represent relaxation time constants corresponding to each cell configuration. It was observed that curves are piecewise linear during the overall decay process indicating that the decay process is controlled by two competing chemical reactions. Table 1 shows calculated reaction rate constants derived from the transient decay curves. Current-voltage characteristics of each cell configuration were studied at room temperature and results from the data are displayed in Table 1. The nature of the I-V curves suggested formation of a rectifying junction in each cell configuration. The diode equation was used to obtain junction quality information for each cell configuration. I=I0exp(eV/nkT)-1 The junction ideality factor (n) for each cell configuration is presented in Table 1. As a result of this investigation, it was found that PbO composite electrode yields a high photo-voltage as well as displays less rectifying I-V characteristics. The slow decay in photo-voltage in each cell configuration can be ascribed to the surface states as well as two competing second order solution reactions. All cell configurations were also tested against long-term light soaking. Transient open-circuit voltage and short circuit current data indicated that TiO2 and WO3 composites were the most stable electrodes studied so far.

10:10 AM Break

10:30 AM Invited

Silicon Nitride Processing for Control of Optical and Electronic Properties of Silicon Solar Cells: *Bhushan L. Sopori*[1]; [1]National Renewable Energy Laboratory, Golden, CO 80401 USA

Recently, almost all Si solar cell manufacturers have adopted the use of thin films of silicon nitride (SiN) as antireflection coatings for solar cells. This widespread acceptance of SiN coatings is prompted by the fact that they also produce very effective surface passivation and serve as barrier layers for control of metallization. Furthermore, deposition and processing of these coatings is accompanied by diffusion of H deep into the bulk of the Si, which, in turn, leads to passivation of impurities and defects and a concomitant increase in the solar cell performance. A typical nitridation process uses a plasma-enhanced CVD method to deposit about 750 ≈ of a SiN film on the front side of the solar cell. Next, metallization patterns are screen printed on the front and the backsides of the cell using Ag and Al pastes, respectively. Then, the cell is rapidly annealed in an IR furnace, whereby metallization contact is fired through the nitride and back contact is alloyed. It is clear that the role of nitride is quite complex, and a control of optical and electronic properties of the device requires a detailed knowledge of various mechanisms that influence optical parameters, interface charge, and transport of H. This paper will review current understanding of these mechanisms and present a systematic process sequence for optimization of the solar cell performance.

11:00 AM

Interfacial Sliding and Plasticity in Back-End Interconnect Structures in Microelectronic Devices: *Chanman Park*[1]; *Indranath Dutta*[1]; Keith A. Peterson[1]; Joseph Vella[2]; [1]Naval Postgraduate School, Ctr. for Matls. Sci. & Eng., Dept. of Mechl. Eng., Monterey, CA 93943 USA; [2]Motorola, Process & Matls. Characterization Lab., 2200 W. Broadway Rd., MD-360, Mesa, AZ 85202 USA

Back-end interconnect structures (BEIS) consisting of Cu lines embedded in low-K dielectrics (LKD) can be subjected to substantial imposed strains when the device or package is thermally cycled. This results from the large differences in thermal expansion coefficients (CTE) between Si, LKD and Cu. Such deformation may induce local plasticity, creep or interfacial sliding within the interconnect structure. These effects are expected to become more prominent with decreasing line dimensions, and increasing ILD compliance, necessitating fundamental studies of the involved deformation mechanisms. Here we report the results of atomic force microscopy (AFM) studies of plastic deformation and interfacial sliding in single and bi-layer Cu-low K ILD structures on Si. The AFM measurements demonstrated that plasticity of interconnect lines, accommodated by diffusionally-controlled interfacial sliding occurs in the interconnect structure, resulting in dimensional distortion due to both local (device-level) and far-field (package-level) stresses. The mechanics and mechanisms of these distortions, and their impact on device reliability are also discussed. This research was supported by NSF grant # DMR 0075281.

11:20 AM

TaN-TiN Binary-Component Thin Films as Diffusion Barriers for Copper Interconnects: *Haiyan Wang*[1]; Abhishek Gupta[1]; Ashutosh Tiwari[1]; A. Kvit[1]; Xinghang Zhang[1]; Jagdish Narayan[1]; [1]North Carolina State University, Matls. Sci. & Eng., 2141 Burlington Lab., CB 7916, Raleigh, NC 27695-7916 USA

TiN, used as a diffusion barrier for Cu interconnects, offers numerous advantages such as low resistivity, well controlled microstructure and well established process technology in IC fabrication. Compared with TiN, TaN thin film has higher thermal stability and fulfills the thickness limitation for next generation ULSI devices. Considering the advantages of these two materials, we have investigated the binary components of TaN-TiN thin films as prospective diffusion barrier materials. By pulsed laser deposition, TiN and TaN targets were arranged in a special configuration that they can be ablated in a sequential manner to obtain TiN-TaN alloy or TiN/TaN superlattice structure. The various concentrations of binary components were obtained by engineering the target configuration. 60% TaN resulted in superlattice of TaN(3nm) /TiN(2nm), while 30% and 75% TaN generated uniform TaXTi1-X N alloys. TiN buffer layers were deposited first to achieve those epitaxial binary components. X-ray diffraction and transmission electron microscopy (TEM) analysis showed the epitaxial nature of these films. Microstructure and uniformity of the superlattice and alloy structures were studied by TEM and Scanning transmission electron microscopy with Z-contrast (STEM). Nanoindentation results suggested high hardness for these new structures and four point probe electrical resistivity measurements showed interesting metallic behavior with very small value of temperature coefficient of resistivity. Cu diffusion characteristics in these binary components were studied and compared after post-deposition annealing at 700°C for 30mins. Studies of Cu diffusion profile were performed using STEM and Electron Energy Loss Spectroscopy (EELS).

11:40 AM
Mechanism of Electron Scattering in Nanoscale Cu Interconnects: *Choong-Un Kim*[1]; J. Y. Park[1]; N. L. Michael[1]; P. Gillespie[2]; R. Augur[2]; [1]The University of Texas at Arlington, Matls. Sci. & Eng., Arlington, TX 76019 USA; [2]International SEMATECH, Austin, TX 78741 USA

With a continuing effort for miniaturization of microelectronic devices, the size of the interconnect used in those devices is expected to reach nanoscale not in a distant future. Since the surface fraction is large in nano-sized interconnect, an increase in the electrical resistivity due to electron surface scattering can be resulted. This, increase in the resistivity by surface scattering (known as a size effect), is becoming a critical matter because it has a potential of limiting electrical performance of the devices. This paper presents the mechanism of the size effect found in dual damascene processed Cu interconnects with a size varying from 50nm to 750nm. With various characterization and analysis conducted, our study finds that the size effect in real interconnects may be a result of impurity scattering. While the influence of the surface scattering and grain boundary scattering is found to exist in nanoscale interconnects, the majority of the increased resistivity is determined to be a result of increased content of impurities or crystal defects.

Materials Processing Under the Influence of Electrical and Magnetic Fields - I

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee
Program Organizers: Joanna R. Groza, University of California-Davis, Chemical Engineering Material Science Department, Davis, CA 95616 USA; George S. Dulikravich, The University of Texas at Arlington, Multidisciplinary Analysis, Inverse Design, and Optimization (MAIDO) Program, Department of Mechanical and Aerospace Engineering, Arlington, TX 76019 USA; Nagy H. El-Kaddah, University of Alabama, Department of Metallurgical & Materials Engineering, Tuscaloosa, AL 35487-0202 USA; James W. Evans, University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA; Zuhair Munir, University of California, College of Engineering, Davis, CA 95616-5294 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Monday AM — Room: 14A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Hans Conrad, North Carolina State University, Raleigh, NC 27695-7907 USA; Robert J. Dowding, US Army Research Laboratory, Aberdeen Proving Ground, MD 21005-5069 USA

8:30 AM Opening Remarks

8:35 AM Keynote
Effects of an Electric Field and Current on Phase Transformations in Metals and Ceramics: *Hans Conrad*[1]; [1]North Carolina State University, Matls. Sci. & Eng. Dept., Raleigh, NC 27695-7907 USA

Effects of an electric field and current on the equilibrium and kinetics pertaining to solid-state transformations in metals and ceramics are reviewed. Examples of the influence of an electric field on equilibrium include: (a) phase separation in glasses and (b) solubility of alloying elements in Al. Examples of the influence of an electric field on kinetics include: (a) precipitation in Al and Fe alloys, (b) quench hardening of steel and (c) phase coarsening in Sn-Pb alloys. The influence of an electric current on kinetics include: (a) precipitation in Al and Fe alloys and (b) intermetallic compound growth. The mechanisms responsible for the various effects are considered.

9:05 AM Invited
Remarkable Phase Changes: *Rustum Roy*[1]; Jiping Cheng[1]; Peelamedu Ramesh[1]; Larry Hurtt[1]; Yi Fang[1]; Dinesh Agrawal[1]; [1]The Pennsylvania State University, Matls. Rsrch. Inst., University Park, PA 16802 USA

By physical separation of the E and H fields in a single mode Te103 cavity, we have observed for the first time the profound differences between the interaction with all materials by magnetic and electric fields. For example, all ferro and ferromagnetic oxides become noncrystalline ìnano-glassesî in 5-10 seconds in a 2.45 GHz magnetic field. In the same chamber at the same time beautiful euhedral crystals are formed at the E field maximum. These crystalnano-glass and crystalcrystal phase changes also occur in many non-ferromagnetic oxides with 3d and 4f electrons. The wealth of new data provides an immediate major opportunity for new theories of material-electromagnetic interactions.

9:35 AM Invited
Nanocrystalline Materials from Powder Sintering: *Alla V. Sergueeva*[1]; Guodong Zhan[1]; Nathan A. Mara[1]; Amiya K. Mukherjee[1]; [1]University of California, Cheml. Eng. & Matl. Sci., Davis, CA 95616 USA

Nanocrystalline materials can be sintered in bulk form from the corresponding Fe-based amorphous powders. High viscosity of the initial powders in the supercooled liquid region allows production of materials with densities close to theoretical value at relatively low temperatures using electric field assisted sintering under high pressure. Appropriate heat treatment of the consolidated amorphous specimens leads to the formation of a nanocrystalline structure with subsequent densification. Characteristics of the nanostructure can be controlled by the variation of the annealing conditions. In situ crystallization of the initially amorphous powders during sintering has been also applied.

10:05 AM
Effect of Magnetic Field Applied During Secondary Annealing on Texture and Grain Size of Silicon Steel: *Cristiane B. Bacaltchuk*[1]; Hamid Garmestani[1]; Gilberto Castello-Branco[1]; Anthony D. Rollett[2]; [1]Florida A&M Univeristy-Florida State University College of Engineering and Center for Materials Research and Technology, Mechl. Eng. Dept., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310-3706 USA; [2]Carnegie Mellon University, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Temper cold rolled silicon steel samples were secondary annealed for 1 hour at 737, 787 and 837C with and without an applied magnetic field oriented perpendicular to the plane of the sheets. The final grain size was the same for both annealing conditions. Although magnetic annealing did not affect grain growth, annealing in the presence of magnetic field did influence texture development. The results show that the gamma fiber decreased after magnetic annealing for both 737 and 787∞C and increased after ordinary annealing, especially for annealing at 737∞C. Magnetic annealing was also effective in developing the Goss texture component. According to the results the intensity of the Goss component started increasing at the lowest annealing temperature (737∞C), and continued to increase as the annealing temperature increased. For the annealing without a magnetic field, this texture component first appeared only at the highest annealing temperature.

10:25 AM
Surface Tension Measurements of Casting Irons by Electromagnetic Levitation Melting Technique: *Deming Wang*[1]; Tony Overfelt[1]; [1]Auburn University, Mechl. Eng., 201 Ross Hall, Auburn, AL 36849 USA

The paper introduces an electromagnetic levitation facility, Vulcan-I, which was developed to measure the surface tension of molten metals under microgravity environment in the parabolic flights of a

NASA KC-135 research aircraft. The facility was recently modified to measure the surface tensions of molten casting irons on the earth-base. In the contactless measurement method, surface tension is obtained by measuring the frequency of surface oscillation of a levitated melted metal droplet. Theoretical background, numerical simulation and description of the experimental facility are presented in the paper. An electromagnetic analysis model was developed to calculate the levitating force and absorption power on the measured sample, which are very important to design the induction coil for an electromagnetic levitator on the earth-base. The simulation results were verified by experimental measurements on Vulcan-I. As an application of the technique, the surface tensions of pure iron, compact graphitic and ductile irons were measured by the modified electromagnetic levitator on the earth-base.

10:45 AM Break

11:00 AM
Magnetic Annealing of Fe-Ni Alloys: Effects on Local Atomic Arrangements and Magnetic Properties: *Cullie J. Sparks*[1]; Gene E. Ice[1]; J. L. Robertson[1]; Jianming Bai[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., MS 6118, Oak Ridge, TN 37831-6118 USA

We report on diffuse x-ray scattering measurements of the local atomic structure in Fe25Ni75 permalloy and other Fe-Ni solid-solution alloys before and after annealing in a magnetic field. These measurements characterize the near-neighbor atomic-pair distributions and reveal an anisotropic alignment of the Fe-Ni, Fe-Fe and Ni-Ni near-neighbor pairs after annealing in a magnetic field. Single-crystal measurements show that the magnitude of the response to magnetic annealing depends on the direction of the applied magnetic field relative to the crystallographic orientation. In addition to changes in the magnetic anisotropy, other properties are also affected. These measurements directly confirm predictions that magnetic annealing rearranges the local atomic order and leads to an anisotropic alignment of chemical pairs. We discuss the application of magnetic annealing to alter materials properties.

11:20 AM
Consolidation of Boron Carbide (B_4C) by the Plasma Pressure Compaction (P^2C) Method: B. R. Klotz[1]; K. C. Cho[1]; *R. J. Dowding*[1]; [1]US Army Research Laboratory, Aberdeen Proving Ground, MD 21005-5069 USA

Boron carbide (B_4C) powder has been densified by a novel method of powder consolidation known as Plasma Pressure Compaction (P^2C). The P^2C technique allows for rapid consolidation of powder by Joule heating of the powder bed. Powder is placed in graphite dies, and uniaxial pressure and high current D.C. voltage are applied to achieve densification. Pure boron carbide powder was able to be consolidated by the P^2C method at lower temperature and hold time to densities equal to those achieved by conventional hot pressing. With the addition of a small amount of alumina (Al_2O_3) as a sintering aid, densities as high as 97% theoretical were attained.

11:40 AM
Decomposition of MgH2 by SPS: *J. Schmidt*[1]; R. Niewa[1]; M. Schmidt[1]; Yu. Grin[1]; [1]Max-Planck-Institut f¸r Chemische Physik fester Stoffe, N˜thnitzer Strafle 40, Dresden 01187 Germany

Spark plasma sintering is one of the advanced synthesis and consolidation techniques developed in the last decades. A special characteristic of the process is the application of a pulse electric current to a graphite pressing tool and/or the powder compact to be sintered. Up to now, the mechanism of this process is not completley clarified. We studied the decomposition of MgH2 while the SPS process. The set-up chosen includes the temperature measurement inside the sample. Results are compared with data obtained by thermal analysis and indicate clear evidence for an activation in the SPS process.

Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction: Prognosis Opportunities & Applications

Sponsored by: Structural Materials Division,
Program Organizers: James M. Larsen, US Air Force, Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA; Leo Christodoulou, Defense Advanced Research Agency, Arlington, VA 22203-1714 USA; William J. Hardman, Naval Air Systems Command, Propulsion and Power Directorate (AIR 4.4.2), Patuxent River, MD 20670-1534 USA; Andrew J. Hess, Naval Air Systems Command, Propulsion and Power Directorate, Patuxent River, MD 20670-1534 USA; J. Wayne Jones, University of Michigan, College of Engineering, Department of Materials, Science and Engineering, Ann Arbor, MI 48109 USA; Stephan M. Russ, Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA

Monday AM — Room: 16A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: James M. Larsen, US Air Force, AF Rsrch. Lab. Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA; Leo Christodoulou, Defense Advanced Research Projects Agency, DARPA/DSO, Arlington, VA 22203-1714 USA

8:30 AM Invited
Materials Prognosis as a Tool to Assure Readiness and Maximize the Utilization Potential of Complex Mechanical Systems: *Leo Christodoulou*[2]; James M. Larsen[1]; [1]US Air Force, AFRL/MLLMN, 2230 Tenth St., Ste. 1, WPAFB, OH 45433-7817 USA; [2]Defense Advanced Research Projects Agency, DARPA/DSO, 3701 N. Fairfax Dr., Arlington, VA 22203-1714 USA

Performance and life limits for structural materials in complex mechanical systems are often established based largely on a ìfear of failureî. That is, the potential impact of component failure, in terms of human, performance, and/or financial costs, is deemed so extreme that life-management procedures must go to almost any lengths to prevent failure. Conventional approaches for avoiding such failure often involve lengthy inspections, leading to highly conservative ìgo, no-goî operational decisions, or requiring maintenance processes may significantly impair system readiness. This presentation will highlight novel methods, both local and global, for interrogating materials to determine their intrinsic behavior and then linking this signature to physics-based multi-scale models that predict damage accumulation in materials and the cascading effect on future performance. The problem of prediction is made tractable by constraining the predictions to the short term.

9:00 AM Invited
A Prognostic Development Strategy and the Way Forward: *Andrew J. Hess*[1]; [1]NAVAIR, AIR 4.4.2, B106, Unit 4, 22195 Elmer Rd., Patuxent River, MD 20670 USA

Prognostics is one of the more challenging aspects of the JSF Prognostic and Health Management (PHM) or any vehicle advanced diagnostic monitoring and health management system. It also has the potential to be the most beneficial, both in terms of reduced Operational and Support (O&S) and Life Cycle costs and improved safety of the air vehicle. The evolution of aircraft diagnostic monitoring systems have lead to the recognition that predictive prognostic is both desired and technically possible. The new JSF supportability concept, Autonomic Logistics, includes a comprehensive and robust air vehicle PHM system as one of its key enabling elements. This presentation will attempt to discuss the evolution of diagnostics to prognostics from individual platform program histories; the fleet users needs and capabilities; and the diagnostic, prognostic and health management system designer and developers needs and capabilities perspectives. Some of the advance diagnostics and PHM capabilities envisioned for the JSF will be discussed in relation to the Autonomic Logistics concept. Some of the needs to fully realize this predictive prognostic capability will be discussed through attempting to identify holes and barriers in the technology base and exploring proposed ways forward.

9:30 AM Invited
Turbine Engine Health Managing Future Direction: Theodore George Fecke[1]; [1]Air Force Research Laboratory, AFRL-PRTC, 1950 Fifth St., WPAFB, OH 45433 USA

The USAF owns many engines, about 25,000 in 1999, and including spares and consumables is in excess of $32 billion. This massive inventory could double in cost with the recent commitment to acquire the F-22 RAPTOR aircraft by 2005, a year that will see the completion of the IHPTET (Integrated High Performance Turbine Engine Technology) program and the move to the Versatile Advanced Affordable Turbo Engine (VAATE). VAATE will begin a new series of research thrusts for the Air Force Research Laboratory to further the development of the gas turbine engine. The achievements of the IHPTET program, a 2 X increase in thrust based on the YF119 engine, will be pushed again to realize a 2.5 X increase in thrust, but the main focus will change from performance to affordability. Affordability is the Number One concern for the DoD and is defined as: Affordability has become the number one priority to the Department of Defense. The Joint Strike Fighter (JSF) and DARPA Unmanned Combat Air Vehicle (UCAV) programs are prime e xamples of this shift in emphasis towards aircraft system affordability. With the development cost of a military fighter engine hovering around $1.5-2.0B, production costs in the $5M to $10M range for F119 and JSF engines, and maintenance costs averaging $450/engine flight hour (EFH) or more for various fielded combat engines, the turbine engine community is seriously constraining the number of opportunities for new products. Add to this the fact that the number of military engines purchased is continuously going down (projections for the next 30 years show almost a 10/1 ratio between commercial and military engines to be produced), and a cost-of-ownership crisis to the military may be at hand. Clearly, the turbine engine research community must make propulsion affordability the primary focus in the VAATE programs. The combination of high engine life-cycle costs, concerns for greater safety and changing operational demands create the need for improved engine diagnostics and prognostics. Engine maintenance rep resents perhaps the largest part of overall aircraft maintenance costs and, due to safety considerations tends to be performed conservatively. Better information on the actual engine condition, usage and life monitoring can significantly reduce maintenance costs. There is a need to develop these capabilities further and combine data from an array of sensors to enable engine health management using more advanced diagnostic and prognostic techniques, and the latest sensor technologies.

10:00 AM Break

10:30 AM Invited
The Role of State Awareness Sensors for Advanced Prognostic Systems: *Jerrol W. Littles*[1]; Bradford A. Cowles[1]; Richard A. Holmes[1]; Robert B. Berkley[1]; [1]Pratt & Whitney, E. Hartford, CT 06108 USA

Comprehensive prognostic systems have significant potential to provide customers with even more safe, reliable, and cost-effective gas turbine engines. One concept for advanced prognostics utilizes the incorporation of data from a suite of state awareness sensors into advanced lifing algorithms. Such a system will implement the most comprehensive life algorithms and use the data from advanced sensors to ground the life assessments. The data provided by the sensor suites is anticipated to have at least three fundamental purposes: 1) provide a real-time assessment of engine operating conditions for incorporation into engine usage algorithms, 2) provide real-time damage accumulation feedback to the advanced damage evolution algorithms, and 3) provide real-time feedback to the pilot for immediate catastrophic failure avoidance. A current program, sponsored by DARPA and AFRL, is focused on using advanced life algorithms to predict the effects of dwell cycles on crack propagation in engine components under realistic operating conditions. A suite of sensor are implemented to perform an early assessment of their technology readiness levels for potential incorporation into a real-time on-board comprehensive prognostics system for advanced aircraft engines.

11:00 AM Invited
Structural Health Monitoring of Aerospace Vehicles Using Advanced Signal Analysis Techniques: *Mark M. Derriso*[1]; [1]Air Force Research Laboratory, Air Vehicles Direct., 2790 D. St., WPAFB, OH 45433 USA

The Air Force Research Laboratory (AFRL), Air Vehicle Directorate has been involved with maturing the technology of Structural Health Monitoring (SHM) for several years. Past SHM efforts have focused on developing better sensor technology. Today sensor technology is growing at an exponential rate with the development of fiber optical, MEMS, piezoelectric, and nano-sensors while data fusion methods have been largely ignored. The current research program at AFRL/VA takes a more balanced approach to SHM. Although smaller, wireless and more reliable sensors are needed, more research is focused on data interpretation, data fusion, advanced digital signal processing techniques and physics-based models. This presentation will highlight the current research effort of AFRL/VA in the area of SHM for current and future aerospace vehicles.

11:30 AM Invited
A Coupled Approach to Damage Prognosis: *Chuck Farrar*[1]; [1]Los Alamos National Laboratory, Eng. Sci. & Applic., MS T-006, Los Alamos, NM 87545 USA

Los Alamos National Laboratory (LANL) along with research partners at Stanford University and Virginia Tech has launched a large-scale, multi-disciplinary program that to develop and integrate advanced microelectronic sensing technology, tera-scale predictive modeling capabilities for damage evolution, and techniques for correlation-based data compression to address the problem of damage prognosis in aerospace, civil, and mechanical engineering systems. These Damage Prognosis solutions realize information fusion between a robust, densely populated micro-electronic sensing array and a system-specific predictive modeling capability derived from benchmarked high-fidelity tera-scale models. The tera-scale predictive models will be compressed to allow the surrogate predictive models to be deployed on the monitored system using advanced micro-electronic processing hardware that is directly coupled with the sensing array. This paper will begin by discussing the hardware and software components of this system and the current proof of principal experiments being conducted at Los Alamos.

Measurement and Interpretation of Internal/Residual Stresses: Polycrystalline Deformation Modes: Expt and Model

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Shaping and Forming Committee
Program Organizers: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Bimal K. Kad, University of California, Ames Laboratory, La Jolla, CA 92093-0085 USA

Monday AM — Room: 17B
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Sci. Ctr., Los Alamos, NM 87545 USA; Raj Vaidyanathan, University of Central Florida, AMPAC/MMAE, Orlando, FL 32816-2455 USA

8:30 AM Invited
Crystal-by-Crystal Finite Element Simulations of Internal Stresses in Polycrystalline Metals: *Paul R. Dawson*[1]; Donald E. Boyce[2]; [1]Cornell University, Sibley Sch. of Mechl. & Aeros. Eng., 196 Rhodes Hall, Ithaca, NY 14853 USA; [2]Cornell University, Sibley Sch. of Mechl. & Aeros. Eng., 191 Rhodes Hall, Ithaca, NY 14853 USA

Finite element simulations of the elastoplastic deformations of polycrystalline solids provide the means to compute detailed pictures of their mechanical behaviors. The stress distributions within and among crystals can be obtained as well as histories of the evolution of variables used to quantify the mechanical state. In simulations in which individual crystals are discretized by one or more finite elements, the list of variables includes the orientation of the atomic lattice and the strength(s) of potentially active slip systems. Neutron diffraction experiments supply peak distribution data from which lattice strain (peak shift), texture (peak intensity), and dislocation density (peak width) can be extracted for sets of crystals defined by particular combinations of scattering vector and reflecting crystallographic plane. We will compare the simulation results to experiments by creating direct analogues of the experimental distributions wherever possible. We will then draw conclusions regarding modeling demands placed on the grain interaction assumptions.

9:00 AM Invited
Grain Interaction Stresses Associated with Deformation Twinning in the Hexagonal Materials Beryllium, Magnesium, and Zirconium: *Donald W. Brown*[1]; Sean R. Agnew[2]; Mark A.M. Bourke[1]; Thomas M. Holden[1]; William R. Blumenthal[1]; Carlos Tome[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [2]University of Virginia, Matls. Sci. & Eng., Charlottesville, VA 22904-4745 USA

The development of internal stress and texture of hexagonal close packed metals, Be, Mg, and Zr in particular, have been investigated with neutron diffraction. The relative activity of deformation mechanisms was interrogated through comparison of the observed textures with those calculated within a visco-plastic self-consistent model. The conditions of deformation, i.e. sample temperature, strain rate, and initial texture, were varied to study the complicated interplay between slip and twin deformation mechanisms in these low symmetry metals. In beryllium, for example, it was possible to force the activity of deformation twinning through manipulation of the initial texture of the sample.

9:30 AM
In-Situ Neutron Diffraction and Polycrystal Modeling of Deformation of a Textured Magnesium Alloy: *Sean R. Agnew*[1]; Donald W. Brown[2]; Thomas M. Holden[2]; Carlos N. TomÈ[2]; Sven C. Vogel[2]; [1]University of Virginia, Matls. Sci. & Eng., 116 Engineerís Way, Charlottesville, VA 22904-4745 USA; [2]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

The mechanical behavior of noncubic metals, is controlled by the relative strengths and hardening responses of a variety of possible slip and deformation twinning modes. The initial texture and straining direction play critical roles in determining which deformation mechanisms will be active. Polycrystal models of elasto-plasticity provide a means to simulate such behavior and, in fact, probe the activity of the various deformation modes. Experimental validation of these models can be performed using measured flow curves, texture developments, and internal strains. The internal strains necessary for this purpose can be measured by in-situ straining using neutron diffraction. Magnesium alloy AZ31B plate with a basal texture was tested in through-thickness compression, in-plane tension, and in-plane compression. The responses of the former two were dominated by dislocation slip and characterized by only subtle texture evolution. The latter sample, however, underwent extensive deformation twinning resulting in a complete reorientation of the texture.

9:50 AM
Evidence on the Development of Large Grain-Orientation-Dependent Residual Stresses in a Cyclically-Deformed Alloy: *Y. D. Wang*[1]; H. Tian[2]; A. D. Stoica[1]; P. K. Liaw[2]; X.-L. Wang[1]; J. W. Richardson[3]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, 701 Scarboro Rd., Oak Ridge, TN 37830 USA; [2]The University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [3]Argonne National Laboratory, Intense Pulsed Neutron Source, Oak Ridge, IL 60439 USA

The micro-mechanical behaviors, particularly the distributions of stresses, on the scale of grain size during or after cyclic deformation of metals and alloys are crucial to a full understanding of damage mechanisms of fatigued materials. Whether the micro-stress is produced and how it develops during cyclic loading with a small amount of monotonic strains but a large amount of accumulated strains remain open questions. Here we report a recent experimental investigation on the development of grain-orientation-dependent (or intergranular) residual stresses in 316 stainless steel during high-cycle fatigue. The lattice strain distributions for different {hkl}-planes in the fatigued samples were measured in the Intense Pulsed Neutron Source (IPNS), at the Argonne National Laboratory, using the neutron time-of-flight technique. The intergranular stresses were determined by constructing the stress-orientation-distribution function (SODF) directly from the measured strain distributions. An important finding is that a large intergranular residual stress is developed in the sample where micro-cracks start to appear at the surface. With an increasing number of fatigue cycles, the intergranular residual stress decreases, and finally disappears when the sample reaches failure.

10:10 AM Break

10:25 AM Invited
Influence of Deformation Modes and Twin Boundaries on Intergranular Stress Development: *Mark R. Daymond*[1]; Edward C. Oliver[1]; [1]Rutherford Appleton Laboratory, ISIS Fac., Chilton, Didcot, Oxon. OX11 0QX UK

Plastic deformation in engineering alloys with hexagonal crystal structures tends to occur by a combination of (multiple) slip and twinning deformation modes, due to their restricted symmetry. Twinning is a fundamentally different deformation mechanism to slip, occurring by a sudden reorientation of the crystal lattice, causing rapid stress relief. This presents a particular challenge for a realistic representation within models of the deformation of polycrystalline aggregates. The work hardening of the aggregate is strongly linked to the relative activity of the slip and twinning modes. Twin boundaries pose barriers to the passage of dislocations, and therefore twinning simultaneously causes hardening of slip systems, which will be evidenced by the build up of intergranular stress. We can obtain insight into these mechanisms by comparing results from diffraction experiments which directly probe the strain state of various grain orientations with predictions from elasto-plastic self-consistent models.

10:55 AM
In-Situ Deformation Induced Interal Strains in TiAl-Based Alloys: *Bimal Kad*[1]; Bjorn Clausen[2]; Mark Bourke[2]; [1]University of California-San Diego, Structl. Eng., 409 University Ctr., La Jolla, CA 92093-0085 USA; [2]Los Alamos National Laboratory, LANSCE-12, Los Alamos, NM 84505 USA

Neutron Diffraction measurements are performed on in-situ compression loaded TiAl based alloys. Diffraction data is recorded across the relevant $\alpha_2+\gamma$ two-phase field, from which most promising material compositions are derived. Single phase α_2-Ti_3Al and γ-TiAl behavior is extracted using sample compositions to the left and right, respectively, of the $\alpha_2+\gamma$ phase field, and composite behaviour is extracted using a numebr of samples with varied compositions and processing conditions within this two-phase field. Results show the apparent anisotropy of deformation of either phase, particularly in the <a> and <c> directions, as observed in the loaded and unloaded state following load excursions into the plastic regime. Such response is modified in the two-phase composites, in what could provide subtle evidence of the specific contibutions of the α_2 phase towards improving the bulk γ-phase response in commercially viable alloys.

11:15 AM
Phase Strain Evolution During Creep Deformation of a Precipitation Strengthened Titanium Aluminide: *Hahn Choo*[1]; Dongyi Seo[2]; Jonathan Beddoes[3]; Mark A.M. Bourke[4]; Sven Vogel[4]; [1]The University of Tennessee, Matls. Sci. & Eng., 319 Dougherty Hall, Knoxville, TN 37996 USA; [2]National Research Council, Inst. for Aeros. Rsrch., Struct., Matls. & Propulsion Lab., Montreal Rd., Bldg. M-13, Ottawa, Ontario K1A 0R6 Canada; [3]Carleton University, Mechl. & Aeros. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada; [4]Los Alamos National Laboratory, Matls. Sci. & Tech., MS H805, Los Alamos, NM 87545 USA

We investigated the micromechanics of creep deformation in a fully lamellar TiAl. In this study, Ti-48Al-2W samples with or without precipitates were mechanically loaded under 276MPa at 1033K, and time-resolved in-situ neutron diffraction measurements were made. The elastic phase strain evolution in the constituent phases and the changes in the load-partitioning behavior were monitored as a function of time during the primary and secondary creep deformation. Previous macroscopic creep studies of the fully-lamellar TiAl-W had indicated that the dominant rate-controlling mechanism was the emission of dislocations from lamellar interfaces. Furthermore, precipitates along interfaces substantially reduced the primary creep transient strain by hindering interface dislocation motion along lamellar interfaces. The micromechanical understanding of the creep behavior obtained using neutron diffraction will be discussed in correlation with macroscopic creep results and deformed microstructures.

11:35 AM Cancelled
In-Situ Investigation and Simulation of the Strain and Texture State Within a Solid AlMg3 Torsion Sample Using Synchrotron Radiation: *RenÈ V. Martins*[1]; Lawrence Margulies[1]; Ulrich Lienert[2]; Anke Pyzalla[3]; [1]Risoe National Laboratory, AFM, PO Box 49, Roskilde DK-4000 Denmark; [2]Argonne National Laboratory, Adv. Photon Source, 9700 S. Cass Ave., Argonne, IL 60439 USA; [3]TU-Berlin, Inst. for Matls. Sci. & Tech., Berlin D-10587 Germany

Microstructural Processes in Irradiated Materials: Advanced Experimental Techniques I

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

Monday AM — Room: 11A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Gary Was, University of Michigan, Dept. of Nucl. Eng. & Radiologl. Scis., Ann Arbor, MI 48109-2104 USA; Brian Wirth, Lawrence Livermore National Laboratory, Matls. Sci. & Tech. Div., Livermore, CA 94551 USA

8:30 AM
Irradiation Induced Vacancy and Cu Aggregations in Fe-Cu Model Alloys of Reactor Pressure Vessel Steels: State of the Art Positron Annihilation Spectroscopy: *Masayuki Hasegawa*[1]; Y. Nagai[1]; Z. Tang[1]; T. Chiba[2]; E. Kuramoto[3]; M. Takenaka[3]; [1]Tohoku University, Inst. for Matls. Rsrch., Katahira 2-1-1, Aoba-ku, Sendai, Miyagi 980-8577 Japan; [2]National Institute for Materials Science, Adv. Matls. Lab. Japan; [3]Kyushu University, Rsrch. Inst. for Appl. Mech. Japan

We report a new phenomenon that positrons are sensitively trapped at the Cu ultrafine precipitates (nanoparticles) in Fe-Cu model alloys, which is further powered by the CDB (coincincidence Doppler broadening) and 2D-ACAR (two dimensional angular correlation of annihilation radiation) techniques to clarify the microscopic structures of the nanoparticles. It is demonstrated that vacancy-Cu complexes are formed by neutron irradiation and aggregate into nanovoids. Their inner surfaces are covered by Cu atoms. The dissociation of vacancies from the nanovoids by annealing around 400∞C leads to the formation of the Cu nanoparticles which consist of only Cu atoms and anneal out around 650∞C. The Fermi surface (FS) of the Cu nanoparticles obtained by the 2D-ACAR has the characteristic shape of ìBCCî Cu FS given by a full-potential linearized augmented plane wave (FLAPW) band structure calculation. This evidences the BCC Cu nanoparticles in Fe.

9:15 AM
Field Ion Microscopy and 3D Atom Probe Techniques Applied to Radiation Damages in Metallic Materials: *Philippe Jean Pareige*[1]; [1]Rouen University, Groupe de Physique des MatÈriaux, CNRS-6634, ERTn∞1000, Rsrch. Inst. on Matls., Saint Etienne du Rouvray 76801 France

The evolution of mechanical properties of irradiated materials may be explained looking at the microstructures at the atomic scale. This basic consideration: tracking matter at the atomic scale to predict the integrity of large structure, leads to the development of specific sophisticated analytical techniques. The field ion microscope (FIM) and 3D atom probe (3DAP) techniques are now extensively used in that domain of irradiation effects in metallic materials. From model alloys to complex industrial materials, an accurate nanoscale insight of the structures under irradiation is achieved. In the field of irradiation damages, computational simulation has also shown its ability to bring new information. The one to one atomic scale comparison between 3DAP experiments and numerical simulations that can be made is of prime interest and need to be improved. After an introduction on the FIM-3DAP techniques, a review of their application on irradiated matter will be presented.

10:00 AM Break

10:30 AM
Recent Results of Small Angle Neutron Scattering Studies on the Effects of Flux, Fluence, Temperature and Composition on Nanostructural Features in RPV Steels: *Brian D. Wirth*[1]; G. Robert Odette[2]; R. Doug Klingensmith[3]; Gene Lucas[3]; [1]Lawrence Livermore National Laboratory, MSTD/CMS, PO Box 808, L-353, Livermore, CA 94551 USA; [2]University of California-Santa Barbara, Mechl. Eng., Santa Barbara, CA 93106 USA; [3]University of California-Santa Barbara, Cheml. Eng., Santa Barbara, CA 93106 USA

The continued safe operation of nuclear reactors and their potential for lifetime extension depends on ensuring reactor pressure vessel (RPV) integrity, which embrittles as a result of the formation of a high number density of nanometer-sized copper rich precipitates (CRPs) and sub-nanometer defect-solute clusters during neutron exposure. Small angle neutron scattering (SANS) has been a key tool in the development of physically-based embrittlement models by characterizing the nanostructural features as a function of key embrittlement variables. SANS is particularly effective since the nanoscale features are coherent and extremely small (<= 1-2 nm), producing scattering at high angles that is largely absent in unirradiated controls. Further, neutrons interact by both magnetic and nuclear contrast, and the corresponding magnetic to nuclear scattering ratio contains information on both the mix and composition of the features. In this talk, we provide a summary of recent SANS results on the evolution of CRPs.

10:50 AM
Positron Annihilation Spectroscopy and Small Angle Neutron Scattering Characterization of Nanostructural Features in Irradiated Fe-Cu-Mn Alloys: *Stephen C. Glade*[1]; Brian D. Wirth[1]; G. Robert Odette[2]; Palakkal Asoka-Kumar[1]; Phil A. Sterne[1]; [1]Lawrence Livermore National Laboratory, MSTD/CMS, PO Box 808, L-280, Livermore, CA 94551 USA; [2]University of California-Santa Barbara, Mechl. Eng., Santa Barbara, CA 93106 USA

Irradiation embrittlement in nuclear reactor pressure vessel steels results from the formation of a high number density of nanometer-sized copper rich precipitates and sub-nanometer defect-solute clusters. We present recent results of a complementary positron annihilation spectroscopy (PAS) and small angle neutron scattering (SANS) characterization study of the size, number density and chemical composition of the nanometer-sized features formed in model reactor pressure vessel alloys following neutron irradiation. PAS is unique in identifying individual vacancies and vacancy clusters through lifetime measurements along with the local chemical environment through coincidence Doppler-broadening measurements of the orbital electron momentum spectrum. As well, positrons preferentially localize in copper precipitates and as such, PAS provides a complementary microstructure probe to SANS and atom probe tomagraphy.

11:10 AM
Atom Probe Tomography of Radiation-Sensitive KS-01 Weld: *Michael K. Miller*[1]; Kaye F. Russell[1]; Mikhail A. Sokolov[1]; Randy K. Nanstad[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6136 USA

The high copper (0.37 %), high manganese (1.64%), high nickel (1.23%) and high chromium (0.47%) KS-01 test weld exhibited a high sensitivity to neutron irradiation. After neutron irradiation to a fluence of only 0.8 x 10^{19} n. cm^{-2} (E > 1 MeV) at a temperature of 288∞C, this atypical weld exhibited a Charpy T_{41J} shift of 169K, a shift of the fracture toughness transition temperature of 180K, a decrease in upper shelf energy (USE) from 124 J to ~78 J, and an increase in the yield strength from 600 to 826 MPa. Atom probe tomography revealed a high number density of Cu-Mn-Ni-enriched precipitates. The number density (3 x 10^{24} m^{-3}), size (l_g = 3.3 ± 0.8 nm) and average composition (Fe- 13.3 at. % Cu, 26.6% Mn and 9.7% Ni) of these features will be compared to other pressure vessel steels. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, US Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the US Department of Energy.

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Alloy Phases Committee

Program Organizers: Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Michael R. Notis, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18015 USA; Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA

Monday AM — Room: 12
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: L. J. Chen, National Tsing Hua University, Dept. of Matls. Sci. & Eng., Hsinchu 300 Taiwan; Douglas J. Swenson, Michigan Technological University, Dept. of Matls. Sci. & Eng., Houghton, MI 49931 USA

8:30 AM Invited
Thermally Stable, Oxidation Resistant Capping Technology for Ti/Al Ohmic Contacts to n-GaN: Chris M. Pelto[1]; *Austin Y. Chang*[1]; [1]University of Wisconsin-Madison, Matls. Scis. & Eng., 1509 University Ave., Madison, WI 53706-1595 USA

In this presentation, we will report a new capping technology utilizing the intermetallic TiAl3 for Ti/Al ohmic contacts to n-GaN. The electrical performance of this TiAl3-capped contact is similar to that of a standard Ti/Al/Ni/Au contact but processed at a much lower temperature! Moreover, this novel contact structure is insensitive to oxygen in the annealing ambient. A value of □c = 1.1◊10-5 □ cm2 following 5-20 min at 600∫C in air has been routinely achieved, similar to that attained when oxygen-gettered Ar was used. The performance of the TiAl3-capped bilayer was found to be stable following thermal aging for more than 100 h at 350∫C in air, comparable to an optimally annealed Ti/Al/Ni/Au contact aged at the same time. These results demonstrate that this contact technology could be used in actual practice for process annealing of laser structures alongside a Ni/Au contact to p-GaN in air.

8:50 AM Invited
Ni, Pd, or Pt as Contact Materials for GaSb and InSb Semiconductors: Phase Diagrams: *Herbert Ipser*[1]; Klaus W. Richter[1]; [1]University of Vienna, Inst. f. Anorgnische Chemie, Waehringerstr. 42, Wien A-1090 Austria

The development of well defined and thermally stable ohmic contacts for III-V semiconductors like InSb and GaSb is still a challenging problem in semiconductor device technology. As device processing usually includes the exposure to elevated temperatures, interface reactions often occur during metallization and further heat treatment. It is thus important to understand the respective phase equilibria of the involved elements. From the thermodynamic point of view, binary and ternary compounds in equilibrium with the respective compound semiconductor would be the best choice for contact materials as these contacts will be stable even after long exposure to elevated temperatures. These possible candidates for contact materials may be directly obtained from the phase diagrams. During the last years we investigated several phase diagrams of transition metals with GaSb and InSb. Our experimental results in the systems Ga-M-Sb and In-M-Sb (M = Ni, Pd, Pt) are discussed in the context of contact chemistry.

9:10 AM
Contacts to the Antimonide Based Compound Semiconductors: *Joshua Robinson*[1]; Wayne Liu[1]; Sammy Wang[1]; Suzanne Mohney[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA

The antimonide based compound semiconductors are of growing interest for both optoelectronics and low power electronic devices. For optimal performance of these devices, shallow, thermally stable contacts with desirable electrical characteristics are needed. As the first stage of a study of contacts to these semiconductors, we have conducted a survey of the phase equilibria in the metal-Ga-Sb and metal-In-Sb systems and have begun studies of the reaction kinetics of key metals in contact with GaSb. We recommend candidates for non-reactive contacts (including the elements W and Re), and we present the results of an investigation of phase formation in Pd/GaSb contacts. The reaction between Pd and GaSb occurs at low temperature and provides desirable features for use in certain types of ohmic contacts. Finally, we describe the successful application of the information we have gathered to the design of a shallow, thermally stable, low-resistance ohmic contact to p-InGaSb.

9:25 AM
Effects of Phase Stability and Doping on the Thermoelectric Properties of IrSb3 Skutterudite Compound: *Sung Wng Kim*[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Interdisciplinary Grad. Sch. of Sci. & Eng., Dept. of Matls. Sci. & Eng., 4259 Nagatsuta, Midori-Ku, Yokohma, Kanagawa 226-8502 Japan

$IrSb_3$ base skutterudite compounds has a potential for thermoelectric applications because of the high carrier mobility, high Seebeck coefficient, and relatively low thermal conductivity. Polycrystalline $IrSb_3$ compounds were prepared by powder metallurgy techniques. The relationships between phase stability and thermoelectric properties have been investigated in binary $IrSb_3$ alloys. Also, effects of doping on the phase stability and transport properties have been investigated using Ge, Ru, Pd, and Pt as a dopant. The thermoelectric properties were measured on hot-pressed polycrystalline samples from 300K to 900K. High Hall mobility (up to 1000cm^2/Vs) and good Seebeck coefficient (up to 240μV/K) were obtained in binary $IrSb_3$ alloys. It is shown that the electrical resistivity, Seebeck coefficient, and Hall mobility depend strongly not only on the phase stability but also on the doped impurities. Our theoretical analysis suggests that the effective mass is significantly affected by both the doping levels and the doped impurities.

9:40 AM
Structural, Optical Characteristic of ZnO Thin Films Prepared: *Sun-Hong Park*[1]; Koung-Bo Kim[2]; Seon-Hyo Kim[1]; Chul Hwan Choi[3]; [1]Pohang University of Science and Technology, Environ. Sci. & Tech., San 31 Hyoja-dong Nam-gu, Pohang, Kyungbuk 790-784 S. Korea; [2]Ninex, Myung-ji University, Youngin, Kyunggi 449-728 S. Korea; [3]Pohang University of Science and Technology, Dept. of Matls. Sci. & Eng., San 31, Hyja-dong, Pohang 790-784 S. Korea

The high quality ZnO thin films were prepared by a metal-organic chemical vapor deposition on a sapphire (a-Al2O3) substrate. The synthesis of ZnO films were performed over the substrate temperatures of 400-700° and at chamber pressures of 0.1-10torrs. The structural and optical properties of ZnO films were investigated in terms of the deposition conditions such as substrate temperature, working pressure, and the ratio of Zn precursor (Diethylzinc) to oxygen. The ZnO films which are preferentially oriented to 34.42° with respect to (002) plane, were obtained under the process conditions of 650-700° and 3-5torrs. Those films show a narrow full-width at half maximum (FWHM) of 0.4-0.6??. Also, the results of photoluminescence show a strong near band edge emission at 3.36eV at 10K as well as a very weak emission at the deep level around 2.5eV at room temperature. Accordingly the synthesized ZnO films indicate a good optical property and low deep level defect density. In addition, we are concerned about annealing effect at high temperature and buffer layer for reduction lattice mismatch stress, and this work is entertained advance cystallinity and optical property.

9:55 AM Break

10:20 AM Invited
Morphological Development of Si_xGe_{1-x} Thin Films Grown on Strain-Modified Si(100) Surfaces Using Molecular Beam Epitaxy: *Douglas Swenson*[1]; Qingfang Yao[1]; [1]Michigan Technological University, Dept. of Matls. Sci. & Eng., 1400 Townsend Dr., Houghton, MI 49931 USA

Quantum cellular automata (QCAís) represent a potential new architecture for high-speed computation. The basic unit of this computing paradigm comprises four semiconducting quantum dots arranged in a nanometer-scale square array. Recently, we have determined that the ìbuilding blocksî of the QCA architecture may be formed via self-assembly using molecular beam epitaxy on Si(100). This is accomplished by a three step fabrication procedure: deposition of nanoscale, SiC particles on the Si surface, followed by Si buffer layer growth and finally deposition of a Si_xGe_{1-x} thin film. The buffer layer develops square pits associated with the underlying SiC particles, and the Si_xGe_{1-x} layer spontaneously forms {105} faceted rectangular islands that decorate the edges of the buffer layer pits. Here, we describe film morphologies that develop over a range of Si_xGe_{1-x} compositions (0.33≤x≤0.80). Related, progressively more highly organized structures are found with increasing Ge content. The morphological changes relate strongly to thermodynamic factors, and may be rationalized in terms of the interplay of surface energy and strain energy.

10:40 AM Invited
Interfacial Reaction Between Immiscible Ta (W) and Cu Examined by Inter-Atomic Potential Through Molecular Dynamics Simulations: L. T. Kong[1]; H. R. Gong[1]; W. S. Lai[1]; *B. X. Liu*[1]; [1]Tsinghua University, Matls. Sci. & Eng., Beijing 100084 China

Concerning the barrier function of refractory metal Ta (W) in Cu metallization, molecular dynamics simulations were carried out to examine the interfacial reactions of the Cu/Ta (W) system based on the respective n-body potentials, which were constructed under an embedded atom method and testified to be realistic. The simulation results revealed that interfacial diffusion did take place at an imperfect Cu/Ta interface at 800 K, which is in excellent agreement with direct observations in the cross sectional Ta/Cu samples. In the Cu/W system, however, no any cross-interface diffusion was detected at a perfect Cu/W interface within a temperature range of 300-900 K and such diffusion was still frustrated, even though a disordered interlayer was preset at the interface. It is therefore of interest to pursue experimental study to test if the refractory metal W could serve as diffusion barrier in Cu metallization for replacing currently used Ta.

11:00 AM
Microstructural Evolution of Sputtered Ni3Al and NiMn Thin Film: *Y. Yang*[1]; P. F. Ladwig[1]; Y. A. Chang[1]; [1]University of Wisconsin-Madison, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706-1595 USA

Films of chemically disordered fcc Ni3Al and NiMn were synthesized by r.f. magnetron sputtering. EPMA measurements were used to measure the film compositions. Differential scanning calorimetry(DSC) was then used to identify the microstructural evolution during the subsequent heating process. A similar trend was found in both Ni3Al and NiMn thin film. Glancing angle X-ray diffraction and transmission electron microscopy were used to examine the microstructures of the films corresponding to temperatures before and after the occurrence of each DSC peak.

11:15 AM
Phase Equilibria and Transformations in Cu-Ge Alloys: Sumanth Jagga[1]; *Vijay K. Vasudevan*[2]; [1]Convergys, Inc., Cincinnati, OH 45202

USA; [2]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA

Alloys based on the Cu-Ge system, especially those on the ε_1-Cu_3Ge compound, have considerable potential for application as interconnects in microelectronic devices because of their attractive electrical properties. Though the thermodynamics of this system has been assessed based on available data, knowledge of phase equilibria, structures of phases and phase transformations is far from complete. The present study was undertaken to address these aspects. Arc-melted cigars of five Cu-Ge alloys ranging in composition from 10 to 30 at.%Ge were prepared, homogenized and then subjected to a series of high temperature treatments followed by quenching in various media. Transformations and reaction temperatures were studied by DTA and the microstructures of oth as-cast and heat treated samples were characterized by XRD, OM, SEM, EPMA and TEM. The kinetics and temperature dependence of transformations during continuous cooling were studied using a novel, computer-controlled in situ temperature and electrical resistivity measurement system, coupled with post-mortem analysis of the microstructures. Reaction start and finish temperatures, continuous cooling diagrams, enthalpies and driving forces associated with the ε-ε_2 to ε_1 phase transformation were determined for alloys containing 20 to 30 at.%Ge. New results related to the structure of the high-temperature phases and the nature of the transformation to ε_1 were obtained. These results will be presented and discussed.

11:30 AM
The Relationship Between Phase Stability and Thermoelectric Properties of TiNiSn Half-Heusler: *Takahiro Katayama*[1]; Sung Wng Kim[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Interdisciplinary Grad. Sch. of Sci. & Eng., Dept. of Matls. Sci. & Eng., 4259 Nagatsuta, Midori-Ku, Yokohama, Kanagawa 226-8502 Japan

Half-Heusler type compounds have been studied for promising thermoelectric materials. We have focused on TiNiSn, with or without doping Hf, Zr, Si, or Pt. Nominally stoichiometric TiNiSn alloys were prepared by using arc-melting and subsequent annealing at 1073K for 2 weeks. The thermoelectric properties such as thermoelectric power, electrical conductivity and thermal conductivity were measured in a temperature range from 300 to 900K. As-cast samples show semi-metallic transport properties while annealed samples exhibit semiconductor behavior. Microstructures of TiNiSn alloys basically consist of non-equilibrium four-phase; half-Heusler TiNiSn, Heusler $TiNi_2Sn$, metallic Ti_6Sn_5 and Sn solid solution. The volume fraction of half-Heusler TiNiSn phase significantly increases by annealing. We have revealed that coexisting metallic phases lower the thermoelectric properties of half-Heusler TiNiSn. Doping of impurities strongly affects not only thermoelectric properties but also phase stability. The thermal conductivity of doped alloys decreases due to the point-defect phonon scattering.

Surface Engineering in Materials Science - II: Nanotechnology and Property Evaluation I

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA; A. Agarwal, Plasma Processes, Inc., Huntsville, AL 25811-1558 USA; Narendra B. Dahotre, University of Tennessee-Knoxville, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; C. Suryanarayana, University of Central Florida, Mechanical, Materials & Aerospace Engineering, Orlando, FL 32816 USA

Monday AM — Room: 7A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Sudipta Seal, University of Central Florida, AMPAC & MMAE, Orlando, FL 32816 USA; Yip Wah Chung, Northwestern University, Matls. Sci. & Eng., Evanston, IL 60208 USA

8:30 AM Invited
Nanolayer Coatings for Hard Disk and Demanding Tribological Applications: *Yip-Wah Chung*[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Coating properties and performance can be tailor-designed by proper control of nanostructure and process conditions. This paper presents two examples: one-nm-thick nitrogenated carbon (CNx) nanolayers as protective overcoats in extremely high density hard disk systems and the development of multilayer coatings for wear protection at elevated temperatures. Synthesis of these coatings was done by magnetron sputtering. These studies demonstrate that one can produce atomically smooth one-nm thick CNx overcoats with acceptable corrosion performance. In addition, we show that superhard coatings based on TiN/SiNx and TiB2/TiC multilayers with high thermal stability, low internal stress and high wear resistance can be synthesized by proper choice of nanolayer thickness and process conditions.

9:00 AM
Surface Modification and Magnetism in Nanostructured Materials: *Srikanth Hariharan*[1]; [1]University of South Florida, Dept. of Physics, 4202 E. Fowler Ave., PHY 114, Tampa, FL 33620 USA

Magnetic nanostructures hold tremendous potential as basic building blocks in spin-electronic devices and high-density data storage. Since these structures are often formed as clusters through various synthetic methods, it is important to understand the collective dynamic properties in such materials. Precise mapping of fundamental parameters like the anisotropy and switching fields over a wide range in temperature and magnetic fields, is essential to understand the influence of the relaxation, interactions and other phenomena that govern the dynamic magnetic properties in these systems. We have studied the static and dynamic magnetic properties of as-prepared nanoparticles (Fe, Co, gama-Fe2O3, MnFe2O4) and particles dispersed in a matrix (like polystyrene, SiO2). The systems ranged from polymerized magnetic nanopowders synthesized using a microwave plasma method to highly monodisperse nanoparticles prepared by reverse-micelle techniques. The magnetic anisotropy and switching fields in these materials were systematically tracked over a wide range in temperatures and fields using a Physical Property Measurement System and a novel resonant RF method based on a tunnel-diode oscillator (TDO) operating at 10 MHz. We find subtle variations in the switching, anisotropy and approach to saturation that are different in the particles embedded in a dielectric matrix. The role of surface modification and matrix-mediated interactions will be discussed. We will also report on our progress in searching for novel co-operative magnetic phenomena in co-ordination polymers synthesized using supramolecular chemical assembly. This work is supported by NSF through grant #NSF-ECS-0102622.

9:20 AM
Investigation of the Interface Microstructure of the Enamel/Hot-Rolled Steel by TEM: *Xiao Bing Yang*[1]; Animesh Jha[1]; Rik Brydson[1]; Bob C. Cochrane[1]; [1]University of Leeds, Dept. of Matls., Leeds LS2 9JT UK

In the enamelling of steel, NiO plays an important role in the development of the adhesion of the enamel to the steel. This paper presents the results of the study of interface microstructure of the samples pretreated with and without NiO precoat. Microstructural characterization of the enamel-steel interface was carried out using transmission electron microscopy (TEM). Plan-view thin foil specimens were produced to examine the microstructural features of interface. The presence of different metal oxides and their microstructural morphological features at the interface are analyzed and explained in term of relevant chemical reaction. On the basis of the above results, a mechanism concerning the role of NiO in promoting adherence of the enamel-steel has been proposed.

9:40 AM
Sol-Gel Synthesis of Sterically Stabilized Zirconia Nanoparticles: *Satyajit Shukla*[1]; Sudipta Seal[1]; [1]University of Central Florida, Dept. of Mechl. Matls. Aeros. Eng., 4000 Central Florida Blvd., Eng #381, Orlando, FL 32816 USA

Submicron-sized, monodispersed and non-agglomerated zirconia particles as well as zirconia nanoparticles have been synthesized using sol-gel technique involving controlled hydrolysis of dilute solutions of zirconium alkoxide. The zirconia nanoparticles synthesized using the present technique are sterically stabilized from aggregation by employing an organic polymer viz. Hydroxypropyl Cellulose (HPC). The effect of various process parameters viz. R value (water:alkoxide ratio), HPC concentration, its molecular weight on zirconia nanoparticles size and their phase evolution behavior is systematically investigated. The adsorption behavior of HPC polymer on the zirconia nanoparticles surface is also studied in detail. Various characterization techniques such as XPS, XRD, TEM, SEM and EDS are employed to characterize the synthesized zirconia nanoparticles.

10:00 AM
Adhesion of Thin Ductile Films Using Stressed Overlayers and Nanoindentation: *Megan J. Cordill*[1]; Neville R. Moody[2]; David F.

Bahr[1]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., PO Box 642920, Pullman, WA 99164-2920 USA; [2]Sandia National Laboratories, PO Box 969, MS 9404, Livermore, CA 94551-0969 USA

Compressively stressed tungsten films can be deposited upon ductile films to generate delamination of the underlying films. As the thickness and stress are varied using different sputtering conditions, the morphology of the delamination can change. Two groups of experiments were used to validate the stressed overlayer method (SOL) of film delamination, one with tungsten overlayer, the other with the tungsten on Pt/Ti films on silicon dioxide. Overlayer films with a high compressive stress (1-2GPa) spontaneously buckled forming telephone cord and straight buckles. Tungsten films with a lower compressive stress (less than 1GPa) required nanoindentation tests to induce buckling. The interfacial fracture toughness and phase angle of loading were calculated for both systems. The SOL method was shown to be a valid technique to determine the interfacial toughness of the Pt/Ti/silica system. This work is supported by US DOE Contract DE-AC04-94AL85000.

10:20 AM Break

10:40 AM Invited
Zirconia-Based Nanolaminate Coatings: *Carolyn Rubin Aita*[1]; [1]University of Wisconsin-Milwaukee, AceLab/Matls. Dept., Col. of Eng. & Appl. Sci., PO Box 784, Milwaukee, WI 53201 USA

In recent years, nanostructured ceramic coatings have emerged as technologically important materials chiefly because they can be tailored to exhibit unique properties and behavior not achievable in bulk. This paper addresses the design, synthesis, and characterization of tailored structures in which two or more ceramics are combined in a multilayered nanocomposite: a nanolaminate. First, we give a broad overview of the ceramic nanolaminates. We then use the sputter-deposited zirconia-alumina, zirconia-yttria, and zirconia-titania nanolaminates as model systems to discuss intralayer phase selection, interface characteristics of as-grown coatings, and architectural stability of under adverse environmental conditions. Lastly, we will show how these nanolaminates can be used as components in multifunctional ìsmartî coatings for mechanical and corrosion protection for biomedical applications. Support acknowledged under NSF Grant 9988892.

11:10 AM
An Assesement of the Residual Stresses in Low Pressure Plasma Sprayed Coatings on an Advanced Copper Alloy: *Sai V. Raj*[1]; Louis J. Ghosn[2]; Arvind Agarwal[3]; Thomas P. Lachtrupp[4]; [1]NASA Glenn Research Center, Matls. Div., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [2]Ohio Aerospace Institute, Struct. Div., 21000 Brookpark Rd., MS 49-7, Cleveland, OH 44135 USA; [3]Plasma Processes, Inc., 4914 D Moores Mill Rd., Huntsville, AL 35811 USA; [4]Lambda Research, 5521 Fairlane, Cincinnati, OH 45227-3401 USA

An advanced Cu-8(at.%)Cr-4%Nb alloy developed at NASAís Glenn Research Center, and designated as GRCop-84, is currently being considered for use as combustor liners and nozzles in NASAís future generations of reusable launch vehicles (RLVs) under its GEN 2 and GEN 3 programs. Despite the fact that this alloy has superior mechanical and oxidation properties compared to many commercially available copper alloys, it is felt that its high temperature and environmental resistance capabilities can be further enhanced with the development and use of suitable coatings. Thermal modeling were conducted for several coating top coat-bond coat combinations to assess their suitability as a thermal barrier. The residual stresses developed as a result of depositing these coatings by low pressure plasma spraying (LPPS) were theoretically assessed by modeling their development during cool down after spraying. The residual stress depth profiles of the as-sprayed specimens were measured from the free surface to the interior by x-ray diffraction, where the measurements were made at each depth after removing a suitable amount of material by electropolishing.

11:30 AM
Influence of Flow Rate, Deposition Temperature and System Pressure on TiO_xN_y Film Morphology: *Siddhartha K. Pradhan*[1]; Phillip J. Reucroft[1]; [1]University of Kentucky, Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA

The morphological features of MOCVD TiO_xN_y films are characterized to evaluate the effect of various process parameters. By increasing the TIP(titanium isopropoxide) and ammonia flow ratio the cluster shape changes from angular to rounded. Dilution of flow results in finer elongated clusters. By increasing the flow rate at fixed precursor and ammonia ratios, cluster shapes change from angular to lenticular and cluster size decreases. Deposition at higher temperatures results in bigger clusters, eventually resulting in particle-deposition at 650°C and above. Deposition of particles at high temperature is due to the collective influences of total flow rate, system pressure and temperature.

11:50 AM
A Hybrid Parameter for the Characterisation and Specification of Surface Texture: *Pappula Laxminarayana*[1]; *V. S.R. Murti*[1]; [1]Osmania University, Mechl. Eng., Univ. Col. of Tech., Hyderabad, Andhra Pradesh 500 007 India

The inadequacy of specifying surface texture by the average height Ra of its roughness profile is universally acknowledge owing to its inability to identify the spatial characteristics. Auto correlation function ACF can discriminate between differing spatial structures by its decay properties. Treating the digitized values of ordinates from the roughness profile as a time series, its ACF can be computed. A combination of correlation length from ACF and Ra, in the form of their ratio, leads to a hybrid parameter for the specification of surface texture. Higher this index, denoted by Rx, better is the surface finish. This paper presents, such characterisation of two widely differing surfaces from turning and electrodischarge machining (EDM) and identifies the influence of dominant process parameters on the resultant surface texture characteristically they observed to be feedrate in turning and pulse current and frequency in EDM.

Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties: Structure-Property Relations in Advanced Materials & Point Defects, Dislocations and Deformation of Metals, Ceramics and Intermetallics - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Amit Misra, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Harriet Kung, Los Alamos National Laboratory, Materials Science & Technology Division, Los Alamos, NM 87545 USA; Stuart A. Maloy, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Michael Nastasi, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Ricardo Schwarz, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Monday AM — Room: 10
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: John P. Hirth, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Amit Misra, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

8:30 AM Invited
The Magic of Plutonium and its Alloys: *Siegfried S. Hecker*[1]; [1]Los Alamos National Laboratory, MST-DO, MS G754, Los Alamos, NM 87544 USA

Plutonium is extraordinarily unstable with temperature, pressure, chemical additions, and time. The addition of a few atomic percent gallium retains the face-centered cubic δ-phase to room temperature, avoiding transformation to a monoclinic phase with a huge volume contraction. Plutonium sits near the middle of the actinide series, which marks the emergence of 5f electrons in the valence shell. Right at plutonium the 5f electrons are caught in an abrupt transition between being bonding and being localized (chemically inert). In fact, in the δ-phase they appear to be in a unique state of being neither fully bonding nor localized, which leads to novel electronic interactions, complex structures and fascinating properties. These interactions will be compared to two other unstable elements in the periodic table, namely iron and cerium, to get a better appreciation for the peculiarities of plutonium.

9:15 AM Invited
Microstructural Design of Advanced Materials: *Gareth Thomas*[1]; [1]University of California-Berkeley, Dept. Matls. Sci. & Eng., 561 Evans Hall, Berkeley, CA 94720-1760 USA

Due to the many advances in theory and experiment, it is now possible to ìtailor-makeî materials to specified physical properties-strength, toughness, functional. This development requires a thorough understanding of the iteration between processing, microstructure, compositional distribution and properties, as well as of the appropriate phase and kinetic diagrams. In this sequence the role of electron microscopy and microanalysis has been dominant, and this talk will draw upon these examples: 1. Nano-microstructural design of low carbon structural steels for the infrastructure. 2. Joining of incompatible ceramics: Al2O3-Si3N4 by polytypoids. 3. Grain boundary design for polytypoidal superconducting ceramics. The support of the US Office of Basic Energy Sciences, Div. Mat; Sci; & Engin. USDoE, under contract No. DE-AC03-76SF00098 is gratefully acknowledged.

9:45 AM Invited
Nanoparticles and Nanofilms: *C. Barry Carter*[1]; Christopher R. Perrey[1]; [1]University of Minnesota, Dept. of Cheml. Eng. & Matls. Sci., 410 Amundson Hall, 421 Washington Ave. SE, Minneapolis, MN 55455 USA

In the 1-100 nm size regime, the properties of materials can differ significantly from those of their bulk counterparts. As commercial applications begin to utilize these size-dependent properties, a fundamental understanding of the structure and chemistry of nanoparticles and nanofilms is necessary. Because of the inherently small size of nanoscale structures, transmission electron microscopy (TEM) is a necessary tool in this investigation. Traditional methods of sample preparation for the TEM do not provide a large electron-transparent area and induce sub-surface damage, destroying the nanoscale structure. Using novel specimen preparation techniques, such as the use of the focused ion beam tool, the structure and chemistry of nanoscale structures have been investigated. Issues of interest include the preservation of the film/substrate interface for mechanically different materials, preservation of the internal microstructure, and the defect structure of nanoparticles. Both individual nanoparticles and nanoparticle films have been studied, illustrating the effectiveness of these techniques.

10:15 AM Break

10:45 AM Invited
Determination of Pipe Diffusion Coefficients in Sapphire by Annealing of Dislocation Dipoles: *Arthur H. Heuer*[1]; [1]Case Western Reserve University, Matls. Sci. & Eng., 10900 Euclid Ave., White 418, Cleveland, OH 44106-7204 USA

The break-up of dislocation dipoles into prismatic dislocation loops during annealing plastically deformed samples of undoped and 30ppm MgO-doped sapphire (α-Al_2O_3) single crystals was monitored using transmission electron microscopy (TEM). The dipole break-up occurs sequentially, i.e., prismatic dislocation loops are pinched off at the end of a dislocation dipole. This pinch-off process is primarily controlled by pipe diffusion, and the pipe diffusion coefficients were estimated by monitoring the kinetics of the dipole break-up process, using ex situ annealing of TEM foils, interspersed by periodic TEM examination. We found an activation energy of 4.4 ± 0.2 eV and a pre-exponential of $9.2 \pm 5.2 \times 10^{-4}$ for an undoped crystal and an activation energy of 4.2 ± 0.2 eV and a pre-exponential of $4.3 \pm 3.1 \times 10^{-4}$.

11:15 AM Invited
Galena Powder Preparation: Dislocations and Make-Up Manufacturing in Ancient Egypt: *Jacques Castaing*[1]; Philippe Walter[1]; Patrick Veyssiere[2]; [1]C2RMF-CNRS, UMR 171, 6 Rue des Pyramides, 75041 Paris, Cedex 01 France; [2]ONERA-CNRS, Lab. díetude des Microstructures, BP 72, Chatillon, Cedex 92322 France

Transmission electron microscopy has been used to examine grains of galena (PbS) milled in the laboratory and collected from powders found in ancient Egypt burial objects. The microstructure of coarse particles consists of dislocation networks created by plastic deformation of PbS during milling. Preparation conditions have a strong influence on dislocations arrangements (reactions, interactions, ...). Energetic ball milling produces a large variety of particle sizes from 10 nm to several micrometers, with grains containing very high dislocation densities. Thin foils from archeological specimens are difficult to prepare because they generally include various lead compounds. Dislocations introduced in PbS, by ancient time processing, present many analogies with those found in modern specimens, confirming the assumptions concerning make-up manufacturing.

11:45 AM Invited
Transmission Electron Microscopy of Defects in Transition-Metal Silicides: *Haruyuki Inui*[1]; Masaharu Yamaguchi[1]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan

Many transition-metal disilicides have attracted considerable interest as possible candidates not only for high-temperature structural applications but also for applications in microelectronics and thermoelectric cooling and power generation. TEM is a powerful tool in investigating these silicides, since crystal defects such as vacancies, dislocations and planar defects in many cases play a decisive role in determining the macroscopic properties of our concern. In the present paper, we will present the results made with various TEM techniques on (1) dislocations in structural silicides such as MoSi2, NbSi2 and CrSi2, (2) twins (and enantiomorph identification) in thin films of silicides for microelectronics applications such as MoSi2, TaSi2 and TiSi2 and (3) vacancies (their ordered arrangement) in thermoelectric silicides based on ReSi2-X. HRTEM, CBED and HAADF-STEM are respectively used for investigating each of these items. Implications will be made on defect microstructure-property relationships in these transition-metal disilicides based on the TEM observation results.

The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research: Advanced and Novel Microscopy Techniques

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Japan Institute of Metals, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Julia R. Weertman, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Katherine T. Faber, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Morris E. Fine, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Wayne King, Lawrence Livermore National Laboratory, San Ramon, CA 94583-2496 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Ben Mori, Tokyo 168-0081 Japan

Monday AM — Room: 9
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: J. Weertman, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA; J. R. Weertman, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

8:30 AM Opening Remarks Overview of Professor Meshiiís Career

8:40 AM Invited
Electron and Scanned Probe Microscopies in Nanotechnology: The Discovery, Structure and Properties of Peapods: *David E. Luzzi*[1]; [1]University of Pennsylvania, Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104-6272 USA

Consistent with the history of human technological progress, recent advances in nanoscience and nanotechnology rely upon our ability to ìseeî what it is that we produce. In this talk, the importance of electron microscopy and scanned probe microscopy to the nanotechnology revolution will be discussed. The discovery, synthesis and characterization of nanoscopic peapods within my research group, is a direct example of the important synergies between these two techniques. Our work on peapods, nanotubes filled with 1-D chains of molecules, will be used to illustrate the main thesis of the talk. Results from electron microscopy and scanned probe microscopy studies will be presented along with other data. Finally, the theory and experimental evidence for strong anisotropy in the response of carbon nanotubes to electron irradiation will be presented.

9:10 AM
Examination of Deformation Defects in Bulk Crystals Using Electron Channeling Contrast Imaging: *Martin A. Crimp*[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

The crystallographic characterization of dislocations and deformation twins associated with plastic deformation is commonly carried out by thin foil examination in transmission electron microscopy (TEM) using diffraction contrast analysis. However, the thin foil approach has some limitations including difficulties in sample preparation, relatively small viewable areas, artifacts associated with thin foils, and difficulties in carrying out in-situ studies. As an alternative approach, electron channeling contrast imaging (ECCI) offers the ability to image and characterize crystallographic defects in the near surface region of bulk specimens by using a field emission gun scanning electron microscope (FEG-SEM). In this method, selected area channeling patterns (SACPs) are used to set up imaging conditions in a manner analogous to using selected area diffraction patterns in TEM. Because bulk samples are used in ECCI, the technique is much more amenable to in-situ studies than TEM. This talk will review the electron channeling phenomena and image formation in ECCI, and will outline the experimental parameters necessary to image dislocations and microtwins. A number of examples will be presented that illustrate the capabilities and advantages of using ECCI for defect imaging and analysis including the examination of defect structures at crack tips and edges, the study of defect generation in in-situ loaded samples, and the analysis of deformation transfer and nucleation of microcracks at grain boundaries. This work has been supported by the Air Force Office of Scientific Research grant #F49620-01-0116, the National Science Foundation under #DMR 9257826, the Office of Naval Research grant #N00014-94-0203, and the Michigan State University Composite Materials and Structures Center.

9:30 AM
The Benefits of Energy-Filtering in Weak-Beam Microscopy: *Michael L. Jenkins*[1]; Stephen M. Martin[1]; Crispin J.D. Hetherington[1]; Mark A. Kirk[2]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK; [2]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., Argonne, IL 60439-4838 USA

Under typical weak-beam imaging conditions, more than 50% of the image intensity is contributed by inelastically-scattered electrons with energy losses greater than about 10 eV. In the present work we have explored systematically the benefits of removing these inelastically-scattered electrons by energy filtering. Digital weak-beam images were obtained of long dislocations in Ni_3Ga using a Gatan Imaging Filter attached to a Jeol 3000F FEGTEM. The image quality was assessed in terms of three parameters: the image peak width; the peak-to-background ratio; and the signal-to-noise ratio. All three of these measures were significantly improved in zero-loss energy-filtered images compared with unfiltered images taken under the same imaging conditions. The improvement was most marked in thick areas of foil (> 100 nm), where unfiltered images were badly degraded by chromatic aberration. However, energy-filtered images were of comparable quality to those obtainable in thin areas of foil (< 50 nm).

9:50 AM
Study of Dislocations in Copper by Weak Beam, Stereo, and In Situ Straining TEM: *Rodney J. McCabe*[1]; Amit Misra[1]; Terence E. Mitchell[1]; [1]Los Alamos National Laboratory, MST-8, Struct./Prop. Relations, MS G755, Los Alamos, NM 87545 USA

Weak beam, stereo, and in situ straining TEM were used to study dislocations in copper. Stereo-TEM coupled with in situ straining TEM was used for tracking dislocation motion and interactions in 3D in low dislocation density copper foils. The information obtained from these experiments is useful for understanding dislocation behavior and as input and validation for discrete dislocation dynamics simulations. At higher strain levels, medium to high stacking fault FCC metals form structures of cell blocks separated by dense dislocation walls (DDWs). Little work has previously been done to characterize the dislocation content of DDWs, primarily because the high dislocation density and change in crystal orientation associated with these structures make imaging of the individual dislocations difficult. Weak beam TEM in conjunction with stereo-TEM was used for analysis of the dislocation content of DDWs.

10:10 AM
The Dynamics of Interfacial Evolution in Three Dimensions: Coarsening of Dendritic Microstructures: R. Mendoza[1]; Dimitris Kammer[1]; J. Alkemper[1]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Eng., Cook Hall, 2225 N. Campus Dr., Evanston, IL 60208 USA

Mike Meshii has had a long-standing interest in phase transformations and microstructural evolution. One of the remaining challenges in the area is the visualization and quantitative measurement of the three dimensional morphology of a multiphase microstructure. Recent advances in experimental technology now allow for the routine analysis of metallic microstructures in three dimensions. Visualization is accomplished using a computer to reconstruct the microstructure from a series of planar sections. Using this reconstruction the microstructure is characterized via the probability density of the mean and Gaussian interfacial curvatures, the probability density of the orientation of the interfacial normals, and the genus. As an illustration of this approach, the morphological evolution of dendritic microstructures in directionally solidified Al-Cu and Pb-Sn alloys during coarsening will be presented.

10:30 AM Invited
Atomic-Level Studies of Grain Boundaries: *Karl Leonhard Merkle*[1]; [1]Argonne National Laboratory, Matls. Sci., MSD/Bldg. 212, Argonne, IL 60439 USA

Grain boundary properties and structure are closely related and ultimately determined at the atomic level. In this paper high-resolution transition electron microscopy (HREM) is used to study the atomic-scale structure of grain boundaries. Recently this technique has also has been applied to in-situ observations of grain boundary migration. HREM has been essential to our understanding of the atomic-scale nature of high-angle grain boundaries. We demonstrate this via examples from investigations in metals and ceramic oxides. Major issues addressed by HREM include the role of the grain boundary plane in structure and energetics of grain boundaries, types of relaxations at grain boundary cores, their dependence on interatomic potentials, and the role of grain boundary dissociations.

11:00 AM
Temporal Evolution of Microstructures of Model Nickel-Base Superalloys on a Nanoscale: Experiments and Simulations: *David N. Seidman*[1]; Kevin E. Yoon[1]; Chantal K. Sudbrack[1]; Zugang Mao[1]; Dieter Isheim[1]; Ronald D. Noebe[2]; Pascal Bellon[3]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [2]NASA Glenn Research Center, Cleveland, OH 44135 USA; [3]University of Illinois at Urbana-Champaign, Matls. Sci. & Eng., Urbana, IL 61801 USA

The temporal evolution of microstructures created by solid-state decomposition in multicomponent and multiphase model nickel-base superalloys is studied on a nanoscale employing three-dimensional atom-probe microscopy (3DAP), differential thermal analysis, conventional and high-resolution electron microscopies, kinetic Monte Carlo (KMC) simulations, ThermoCalc, and Dictra. The confluence of 3DAP and KMC simulation techniques allows us to compare directly the experimental 3DAP results with the computational results of the KMC simulations for similar numbers of atoms. A detailed atomistic picture of the temporal evolution of partitioning of elements between phases, segregation at heterophase interfaces, compositions of the matrix and precipitate phases, and precipitate dimensions and morphologies. Detailed 3DAP and KMC results are given for two model nickel-base superalloys (Ni-Al-Cr), with low and high supersaturations of aluminum (concentrations of Al are 5 and 10 at.%), which are compared with one another in detail.

11:20 AM
Connectivity of Grain Boundary Networks: *Christopher Schuh*[2]; Mukul Kumar[1]; Wayne King[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., Livermore, CA 94550 USA; [2]Massachusetts Institute of Technology, Cambridge, MA 02139 USA

It has been demonstrated that mechanical properties of FCC metals and alloys can be improved by exercising control over the population of grain boundary types in the microstructure. The studies also suggest that such properties tend to have percolative mechanisms that depend on the topology of the grain boundary network. Grain boundary engineering investigations have been facilitated by the emergence of SEM-based automated electron backscatter diffraction (EBSD) that enables the characterization of statistically significant datasets of interface crystallography. Recent TEM and EBSD investigations have revealed the mechanism for the introduction of new low energy (special) boundaries into the microstructure that disrupt the spatial connectivity of the random grain boundary network. The EBSD datasets have been analyzed to quantify microstructures in terms of grain boundary character and triple junction distributions. The percolation thresholds obtained for these topologically constrained networks will be compared with the case of random percolation theory.

11:40 AM
Bismuth Induced Embrittlement of Copper Grain Boundaries: *Matthew F. Chisholm*[1]; Gerd Duscher[2]; [1]Oak Ridge National Laboratory, Solid State Div., Bldg. 3025, MS 6030, PO Box 2008, Oak Ridge, TN 37831 USA; [2]North Carolina State University, Matls. Sci. & Eng. Dept., 2156 Burlington Nuclear Lab., Raleigh, NC 27695 USA

Catastrophic brittle fracture of crystalline materials is one of the best documented but least understood fundamental phenomena in materials science. A common feature of most materials that exhibit brittle fracture is the segregation of impurity elements to grain boundaries. Embrittlement of copper by bismuth is the classic example of this phenomenon. In this study, we use a combination of atomic resolution Z-contrast imaging, electron energy-loss spectroscopy and density functional theory to investigate the geometric and electronic structure of a copper grain boundary with and without bismuth. We are able to resolve the atomic arrangement of the bismuth doped boundary, detect impurity-induced changes in the electronic structure and calculate the charge density in the vicinity of Bi impurities. These results suggest that the copper atoms that bond to the segregated bismuth become embrittled by taking on a zinc-like electronic structure.

The MPMD Fourth Global Innovations Symposium: Energy Efficient Manufacturing Processes: Advances in Thermal Processing

Sponsored by: Materials Processing and Manufacturing Division,
Program Organizers: Toni G. Marechaux, National Research Council, National Materials Advisory Board, Washington, DC 20418 USA; Iver E. Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Chris Cockrill, DOE Seattle Regional Office, Seattle, WA USA

Monday AM — Room: 5A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Toni Grobstein Marechaux, National Research Council, Natl. Matls. Advisory Board, Washington, DC 20418 USA; Iver E. Anderson, Iowa State University, Ames Lab., Ames, IA 50011 USA

8:30 AM Opening Remarks

8:35 AM Keynote
Sea Changes in Manufacturing and Materials: *Denise F. Swink*[1]; [1]US Department of Energy, Washington, DC 20585 USA

To address the technological needs of the anticipated revolution of energy consumption in manufacturing industries, revolutionary approaches to materials processing and product development and design are required. Whole new paradigms of development, design and commercialization of products are, consequently, required also. Ms. Swink will share opportunities and challenges we face in both the near and long term to responsively and competitively be proactive to these changes.

9:05 AM Invited
Fluidized Beds: An Energy Efficient Alternative to Conventional Heat Treatment Operations: D. Apelian[1]; *M. Makhlouf*[1]; J. Rosendahl[2]; C. Bergman[2]; [1]Worcester Polytechnic Institute, Metal Procg. Inst., 100 Institute Rd., Worcester, MA 01609 USA; [2]Technomics Corporation, 17200 Medina Rd., Plymouth, MN 55447 USA

Heat treatment of metallic alloys is often essential in order to achieve improved mechanical properties. However, the conventional heat treatment process remains a major constraint to production flow in manufacturing operations since it must be performed off-line in labor-intensive batch processing systems and thus may require cycle times as long as 30 hours. Fluidized beds have been demonstrated to be a far more efficient means of energy transfer than convection furnaces and have been shown to reduce the heat treatment cycle time to about 4 hours. However, typical fluidized bed units are batch systems; therefore, further reductions in cycle time may be possible by using in-line continuous fluidized beds. In this talk, we will review the fundamentals of fluidized beds, highlight an in-line continuous fluidized bed that is currently under construction, and examine the application of fluidized beds to the heat treatment of metallic components with emphasis on the heat treatment of aluminum alloy components for automotive applications.

9:30 AM Invited
Numerical Evaluation of the Impacts of Burner Operations on the Thermal Efficiency of Industrial Furnaces: *Shen-Lin Chang*[1]; *Chenn Q. Zhou*[2]; Kevin Scheeringa[2]; [1]Argonne National Laboratory, Energy Sys. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Purdue University-Calumet, Dept. of Eng., 2200 169th St., Hammond, IN 46323 USA

A computational fluid dynamics code developed at Argonne National Laboratory was used to simulate industrial furnaces. The code was derived from fundamental principles of mass, momentum, and energy conservation. A furnace flow simulation includes turbulent mixing, combustion reaction, radiation heat transfer, and pollutant kinetics. Temperature, velocity, and other flow properties distributions are calculated based on furnace geometry and burner operating conditions. The code validated with experimental data collected from industrial furnaces, was used to evaluate the impacts of burner operation conditions on the energy efficiency of furnaces. Preliminary results indicate that burner injection velocity affects the flow penetration and the species mixing; burner injection angle has a significant impact on the flow patterns and heat transfer; and the equivalence ratio has an effect on the temperature and pollutant concentrations. The study demonstrates that CFD can be a useful tool for analyzing the combustion flow of an industrial furnace.

9:55 AM Break

10:10 AM Invited
Nitrocarburising Saves Energy and Cost: *Paul F. Stratton*[1]; Akin Malas[2]; Keith Bennett[3]; [1]BOC, Northern Techl. Ctr., Rother Valley Way, Holbrook, Sheffield S20 3RP UK; [2]Birlesik Oksijen A.S., Gebze Organize Sanayi Bolgesi, 300, Sokak PK 80, Gebze Kocaeli 41400 Turkey; [3]KMB Metallurgical, 56 Clayton Hall Rd., Cross Hills, W. Yorkshire BD20 7TB UK

Nitrocarburising can often be used as an alternative to conventional thermochemical treatments such as carburising, carbonitriding and nitriding. The properties produced are always at least as good, and in many cases enhanced, compared to high temperature thermochemical processes. Because nitrocarburising is generally carried out at lower temperatures and/or for shorter times than the conventional alternatives, there can be substantial energy savings. The enhanced properties, particularly improved resistance to corrosion and seizure, often mean that there is no need for any further treatments, saving yet more energy and cost. Some examples of the application of the range of optimized nitrocarburising processes offered by BOC on license worldwide are described, together with the properties they produce and their potential for energy savings.

10:35 AM Invited
An Integrated Heat Treatment Model for Aluminum Castings: *Richard D. Sisson*[1]; John E. Morral[2]; Yeming Kevin Rong[1]; Harold Brody[2]; [1]Worcester Polytechnic Institute, Matls. Sci. & Eng., 100 Institute Rd., Worcester, MA 01609 USA; [2]University of Connecticut, Dept. of Metall. & Matls. Eng., Storrs, CT 06269 USA

An integrated system of software, databases, and design rules to enable quantitative prediction and optimization of the heat treatment of aluminum castings to increase quality, increase productivity, reduce heat treatment cycle times and reduce energy consumption is currently being developed. It has been demonstrated, empirically, that the solutionizing time for a cast aluminum alloy can be reduced from 12 to 2 hours with no loss of quality or properties. This magnitude of heat treatment cycle time reduction will result in increased productivity and/or reduced energy consumption of greater than 50%. Validated databases for multicomponent alloys and predictive models will enable comparable results to be achieved for a wide range of alloys and applications. This paper will present the results to date of this project.

11:00 AM Invited
Thermodynamic Analyses of Energy Utilization and Pollutant Control in Secondary Aluminum Melting Furnaces: *Tianxiang Li*[1]; Mohammed Hassan[1]; Kazu Kuwana[1]; Kozo Saito[1]; Srinath Viswanathan[2]; Paul King[3]; [1]University of Kentucky, Dept. of Mechl. Eng., 151 RGAN Bldg., Rose St., Lexington, KY 40506 USA; [2]Oak Ridge National Laboratory, US Dept. of Energy, Oak Ridge, TN 37831-6083 USA; [3]US Department of Energy, Albany Rsrch. Ctr., Albany, OR 97321-2198 USA

This paper presents thermodynamic analyses of energy utilization according to the energy balance in reverberatory, aluminum melting furnaces, from which the overall energy efficiency and major heat losses are derived. The 2nd law efficiency is introduced to the aluminum melting based on the availability concept. The methods that can increase energy efficiency and reduce pollutant emissions from aluminum furnaces are also addressed.

Waste from Metal Plating Industries - I

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; Rodolfo Solozabal, Inasmet, Donostia, San Sebastian E-20009 Spain

Monday AM — Room: 1B
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Junji Shibata, Kansai University, Dept. of Cheml. Eng., Osaka 564-8680 Japan; Hideki Yamamoto, Kansai University, Dept. of Cheml. Eng., Osaka 564-8680 Japan

8:30 AM Invited
Electrolytic Recovery of Metals from Electroplating Effluents with Simultaneous Destruction of Organic Additives at Boron Doped Diamond Electrodes: *Marc A. Verhaege*[1]; Emiel Wettinck[1]; Christa Sonck[1]; Michel Moors[1]; [1]Ghent University, Lab. of Non-Ferrous Metals, Technologiepark 914, Zwijnaarde B-9052 Belgium

Many baths used in the surface treatment and metal finishing industry contain, besides metals, organic additives (brighteners, levelling agents, complexing agents) which can disturb the effluent treatment process mainly due to their complexing capacity for metals. This inhibits the precipitation of metals, a commonly used practice in metal removal effluent treatment plants. In this paper an electrolytic cell is described containing Boron Doped Diamond (BDD) Electrodes, produced by coating Niobium with a BDD layer generated by HFCVD (Hot Filament Chemical Vapor deposition), to treat organics containing plating rinse waters. At the cathode the bulk of the metal ions are reduced and plated out, at the anode the organic additives are oxidised into non-complexing species or completely destroyed, depending on the residence time of the effluent in the electrolysis cell and on the applied current density. Thus, simultaneously, the metal ion content of the effluent is drastically reduced and the TOC (Total Organic Carbon) of the solution is seriously lowered, resulting in lower BOD (Biological Oxygen Demand) and COD (Chemical Oxygen Demand). The treatment of two types of solutions has been studied: a copper-EDTA rinse water (electroless copper plating) and a bright nickel plating bath rinse. It is shown that over 95% of the metal content can be removed, complete decomplexing of EDTA containing solutions (copper plating) is possible and practically all organic matter (99% of TOC) in the nickel plating rinse, at a current density of 2 A/sqdm, is removed. Depending on the goals to be reached (only decomplexing or total TOC/COD removal) current efficiencies of respectively 95 to 55% can be obtained.

9:00 AM Invited

Application of Solvent Extraction and Solvent Impregnated Support to the Treatment of Effluents from Electroless Nickel Plating Processes: *Mikiya Tanaka*[1]; [1]National Institute of Advanced Industrial Science and Technology (AIST), 16-1 Onogawa, Tsukuba, Ibaraki 305-8569 Japan

Recovery of nickel from spent electroless nickel plating baths by solvent extraction has been investigated. Hydroxyoxime extractant such as LIX84I is effective to extract nickel at the pH more than 6. Acidic organophosphorus extractants such as PC88A and Cyanex272 are effective to remove impurity metals (iron and zinc) before extracting nickel. Removal of nickel from the rinse water has been also studied. Solvent impregnated supports with resin and fiber as supports are applied using D2EHPA as extractant. In the batch operation, nickel can be removed with high efficiency without pH adjustment. In the column operation, however, the nickel-D2EHPA complex seems to be re-dissolved into the aqueous phase.

9:30 AM

Fundamental Study for Recovery of Ni2+ from Waste Solution in Ni2+ Non-Electric Plating: *Junji Shibata*[1]; Norio Yoshikawa[1]; Norihiro Murayama[1]; Hideki Yamamoto[1]; [1]Kansai University, Dept. of Cheml. Eng., Yamate-cho, Suita-shi, Osaka 564-8680 Japan

Waste solution from Ni2+ non-electric plating liquor contains 4g/dm3 Ni2+, 70g/dm3 Na+, 80g/dm3 HPO32-, chelating agent and so on. Solvent extraction, cementation, ion exchange resin and precipitation methods are possible to use for the treatment of this kind of solution. In this study, solvent extraction of Ni2+ was investigated using two kinds of extractants to clarify the relation between extraction percent and pH for various extractant mixtures. As nickel ion has an octahedral structure with ligands, synergistic effect for Ni2+ extraction is considered to take place in the solvent extraction using two types of extractants. The investigation on the synergistic effect of Ni2+ extraction was carried out in case of various extractant systems.

9:50 AM

Recovery of Ni in Wastewater from Metal Plating Industries by Application of Ion Exchange Moving Bed: *Hideaki Tokuyama*[1]; Shougo Maeda[1]; Susumu Nii[1]; Fumio Kawaizumi[1]; Katsuroku Takahashi[1]; [1]Nagoya University, Dept. of Cheml. Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

Closed use of water is eagerly required in industry for reduction of environmental pollution as well as for effective use of resources. Acidic wastewater containing much amounts of heavy metals is generated in the process of metal surface treatment. Recovery of heavy metal from wastewater leads to reuse the heavy metals and water. Ion exchange process has an advantage over extraction or precipitation to recover heavy metal, since it is free from accumulation of reagents or solvents in the treated water. In this study ion exchange moving bed apparatus has been developed and its feasibility in countercurrent operation has been examined to separate Ni from model solutions of wastewater prepared by mixing HCl and NiCl2. Ion exchange resin used was a strong acid cation resin DIAION SK1B. The ion exchange equilibrium and diffusivity in resin particle have been also discussed and they depend on the concentration of HCl.

10:30 AM

Recovery of Pd from Waste Solution Containing Pd Catalyst Used in Copper Non-Electric Plating: *Junji Shibata*[1]; Muneto Kobayashi[2]; Hideki Yamamoto[1]; Norihiro Murayama[1]; [1]Kansai University, Dept. of Cheml. Eng., Yamate-cho, Suita-shi, Osaka 564-8680 Japan; [2]Techno Supply, Suita 564-0061 Japan

Through hole copper plating is performed to make multi-layer printed circuit board. Through hole plating is first carried out by Pd catalyst treatment, followed by copper non-electric plating. The amount of Pd catalyst used in one plant is about 0.25-2m3/month (8g/dm3 PdCl2 base) depending on the plant scale, and the cost of the catalyst reaches 17,000□135,000 US$/month. In this study, the flow and material balance of Pd in Pd catalyst treatment process was investigated. About 60% of Pd catalyst adheres on the through hole and other part of setup, while 25% is released into the washing liquor. Pd in the washing liquor should be collected by a suitable method like a pressure filtration using diatomite. The material balance of Pd in Pd catalyst treatment process and the recovery method of Pd from waste solution containing Pd catalyst.

10:50 AM

Removal of Impurity Metal Ions from Waste Plating Solutions by Using Orange and Apple Residues: *Katsutoshi Inoue*[1]; Kedar Nath Ghimire[1]; Kenjiro Makino[2]; [1]Saga University, Dept. of Appl. Chem., Honjo 1, Saga 840-8502 Japan; [2]Yamasoh Micron, Inc., 1-21-12, Uenoshibamachi, Sakai 593-8301 Japan

There is a growing interest of using low cost adsorbents for the treatment of heavy metals generated from various sources including metal plating industry as well. Adsorption tests of lead(II), copper(II) and zinc(II) on alginic acid and pectic acid gels with active carboxylic functional groups indicated that they strongly and selectively adsorb lead and ferric iron over other metals such as zinc and copper. In view of such regard, instead of using pure pectic acid, the pectin components in the crude orange and apple wastes have been utilized by saponificating with calcium hydroxide to obtain abundant carboxyl functionalities for the separation of various metal ions. Adsorptive removals of metal ions were investigated on such saponificated gels. The order of selectivity on saponificated gel was as follows: Pb>Fe>Cu>Cd>Zn>Mn. The exchangeable cations of the saponificated gels were as high as 2.64 mol/kg. Experimental results revealed that saponificated orange and apple gels are suitable for the adsorptive removal of impurity metals from the waste plating solutions for their regeneration.

11:10 AM

Distillery Waste Biomass as a Biosorbent for Removal of Lead: Sarabjeet S. Ahluwalia[1]; *Dinesh Goyal*[1]; [1]Thapar Institute of Engineering and Technology, Dept. of Biotech. & Environl. Scis., Patiala 147-004 India

Removal of lead, nickel, zinc and chromium ions from aqueous solutions by dried waste biomass of Saccharomyces cerevisiae from distillery plant was studied. The removal was in the order of Pb>Ni>Zn>Cr. Adsorption of lead by non-living biomass (20g/100ml of 20ppm metal solution) was 95%, which was strongly affected by the pH in the range of 4-5, whereas temperature had no significant effect. The Fourier transform infrared spectroscopy of native biomass, lead loaded biomass and lead-eluated biomass obtained after desorption by 0.1N-HCl, showed different types of functional groups on the surface of the cell wall are involved in biosorption of Pb and C-H (aliphatic) group were found to play an important role in metal biosorption. In a continuous up flow reactor system 80% removal of lead ions was observed at a flow rate of 100ml/hr with 13hr HRT (hydraulic retention time) when 20ppm lead solution was fed. After first desorption cycle with 0.1N-HCl, 100% removal of lead from aqueous solution was observed under the same condition upto 72hr and thereafter 82% removal occurred. In the second desorption cycle only 50% removal took place. The work has significance as it can have large-scale application for lead removal from industrial effluents by using waste biomass.

11:30 AM

Recycling of Chromium by Solvent Extraction: *Shannon R. Wilson*[1]; [1]Heritage Environmental Services, LLC, Hertiage Tech. Grp., 7901 W. Morris, Indianapolis, IN 46231 USA

A number of methods were looked at for recovery of chromium ions from rinse water for recycle back to the process for a particular plant. The most feasible for the situation would be a solvent exchange process. Bench-scale tests were completed using various extractants with some success. Some of the extractants used were converted to various salt forms, and the effect of chromate loading capacity was determined for each form prepared. By use of a particular salt form of an extractant and a modifier, chromate was effectively and efficiently loaded and stripped from the organic phase for recycling back to the process tanks.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Plenary

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee, Co-Sponsors: ASM International; AsociaciÛn Argentina de Materiales/Materials Research Society of Argentina; AssociaÁ„o Brasileira de Metalurgia e Materiais; Associazione Italiana di Metallurgia; Australasian Institute of Mining and Metallurgy; Chinese Metals Society; Czech Society for New Materials and Technologies; Dansk Metallurgisk Selskab/Danish Metallurgical Society; Deutsche Gesellschaft fuer Materialkunde; Dowa Mining Co., Ltd.; European Journal of Mineral Processing and Environmental Protection; Federation of European Materials Societies; Finnish Association of Mining and Metallurgical Engineers; FLOGEN Technologies, Inc.; Furukawa Co., Ltd., Institute of Materials (UK); Institution of Mining and Metallurgy; Instituto Argentino de Siderurgia; Instituto de Ingenieros de Minas de Chile; Iron & Steel Institute of Japan; Iron & Steel Society; Japan Institute of Metals; Korea Zinc Co. Ltd., Korean Institute of Metals & Materials; Metallurgical Society of the Canadian Institute of Mining, Metallurgy and Petroleum; Mining and Materials Processing Institute of Japan; Mitsubishi Materials Corporation; Mitsui Mining and Smelting Co., Ltd.; Nippon Mining & Metals Co., Ltd.; Non-ferrous Metals Society of China; Outocumpu Oyj, Finland; Slovak Metallurgical Society; Slovensko drustvo za materiale/Slovenian Society of Materials; SociÈtÈ FranÁaise de MÈtallurgie et de MatÈriaux; Society for Mining, Metallurgy, and Exploration; South African Institute of Mining & Metallurgy; Sumitomo Metal Mining Co., Ltd.; Toho Zinc Co., Ltd.

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Monday AM	Room: 6D
March 3, 2003	Location: San Diego Convention Center

Session Chairs: Hong Yong Sohn, University of Utah, Dept. of Metallurgl. Eng., Salt Lake City, UT 84112-0114 USA; Florian Kongoli, FLOGEN Technologies Inc., Montreal, Quebec H3S 2C3 Canada

8:30 AM Hong Yong Sohn: Opening Remarks

8:45 AM Plenary

Lifetime Achievements of Prof. Akira Yazawa: *Kimio Itagaki*[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

The lifetime achievements of Prof. Yazawa, one of the most quoted authors in pyrometallurgical reference books worldwide, are respectfully highlighted and summarized with a birdís-eye view. His research works, published in more than 300 technical papers in the last 50 years, started with the mutual dissolution of copper matte and iron silicate slag, continued with a variety of subjects related to slags, alloys, matte, speiss, aqueous solutions, alcohols and algea and are still continuing in several fields including the thermodynamic calculation of the dioxine formation. This colorful activity, recognized worldwide with many awards, prizes and honors, is categorized according to various disciplines such as thermochemistry, chemical metallurgy, process metallurgy and extractive metallurgy of numerous nonferrous metals. His close collaboration and cooperation, in research and education, with many domestic and international fellows, research associates, students and metallurgical engineers is also highlighted as a great achievement of Prof. Yazawa especially when it is noted how wide and deep this relationship and friendship developed and continuously grew over the years.

9:20 AM Plenary

Contribution of Copper Smelting Technology to Preserving Global Environment: *Akira Nishikawa*[1]; [1]Mitsubishi Materials Corporation, Tokyo 100-8117 Japan

During the twentieth century, the copper smelting industry achieved dramatic expansion corresponding to the vast growth of global economy. This was largely due to innovations in pyro-metallurgical processes, such as autogenous smelting and continuous smelting and converting technologies. However, as copper production capacity increased, its impact on the environment also could not be ignored, and thus with increasing global environmental awareness, many smelting operations either had to renew or rebuild their facilities. In addition to these changes, in places like Japan and Europe, with scarce primary resources, the treatment and recovery of metals from items such as car shredder dust, used electrical appliances and electronic devices, on top of scraps traditionally treated, is having a profound effect on the reduction and neutralization of waste. Thus compared to the days when copper smelting focused on the environmental control in order to break with ìSmoke Stack Industryî, copper smelting today is making an important contribution to preserving the global environment.

9:55 AM Plenary

Impurity Capacity of Non-Ferrous Metals Production Slags: *Ramana G. Reddy*[1]; [1]The University of Alabama, Ctr. for Green Mfg., Dept. of Metallurgl. & Matls. Eng., Tuscaloosa, AL 35487 USA

A Modeling and Experimental study on impurity capacity and distribution between mattes and slags in sulfide smelting of non-ferrous metals was discussed. A Thermodynamic model for prediction of impurities capacities in slags and their distribution in mattes and slags was developed. The impurities capacities (i.e. sulfur and arsenic) of slags were calculated a priori using modified Reddy-Blander model. The capacities predictions were made in a wide range of matte and slag compositions, PSO_2 and temperatures in copper, nickel and lead smelting conditions. The calculated results for sulfur and arsenic capacities are in good agreement with the available experimental data. Application of this model for the prediction of Sb, Bi and other impurities capacities and distribution ratios in mattes and slags, and removal of impurities in several industrial smelter processes is discussed.

10:30 AM Break

10:40 AM Florian Kongoli: Plenary Remarks

10:55 AM Plenary

Application of Sodium Carbonate Slag to Copper Refining: *Chikabumi Yamauchi*[1]; [1]Chubu University, Grad. Sch. of Eng. & Sch. of Eng., 1200 Matsumoto-cho Kasugai-shi, Aichi-ken 487-8501 Japan

An outline of the following will be given. (1)Prof. Akira Yazawa as our pride. (2)Predominance of the application of sodium carbonate slag to copper refining in comparison with that to pig iron refining. (3)Not experimentally direct measurement of the distrbution ratios of impurity elements such as As, Sb, etc. between sodium carbonate slag and molten copper, but indirect calculation of them by using thermodynamic data which were measured by our own experiments. (4)Reasons why such an approach was accepted. (5)Principle of the calculation. (6)Examples of results and discussion. (7)Approach for reducing the contents of impurity elements to their desired levels(Comparison of the calculated values with those obtained by experiments). (8)Kinetic aspects.(9)Application of the slag to a practical operation. (10)future view.

11:30 AM Plenary

Non Ferrous MetalsñThe Challenges in Production and Technology Tranfer: *Juho M‰kinen*[1]; [1]Outokumpu Oyj, Riihitontuntie 7B, PO Box 140, Espoo 02200 Finland

Non-ferrous metals and metals in general are considered as being non-renewable natural resources. This is naturally true, but the predictions that the world would within a short period of time run out of metals have proven to be highly exaggerated. Metals are needed because of their excellent and irreplaceable properties. The consumption of metals, with the exception of carbon steel, is steadily growing by 1-3% annually, and that of stainless steel as much as 3-6%. This is a general trend, which it is periodically disturbed by cyclical variations. In recent years so-called ìNEW ECONOMYî has become prominent in the public media and the market value of the traditional industry has been well below its real value and importance. Nevertheless, the modern society requires metals today and also in the future, and the companies using the most modern technology in a responsible way and innovative solutions not only in technology, but in everything they do will survive. Outokumpu Oyj is one of the leading metals and te chnology companies in the world in stainless steel, copper, zinc, and sales of technology in metals and minerals businesses. Outokumpu applies its knowledge of metals and metals processing to generate value for its customers and shareholders. Everything we do, we do in an economically, environmentally and socially responsible way.

12:05 PM Plenary

The Role of Lead Smelting at Korea Zinc: *Chang-Young Choi*[1]; [1]Korea Zinc Company, Ltd., Young-Poong Bldg., 142 Nonhyon-dong Gangnam-ku, Seoul 135-749 S. Korea

Korea Zinc Company commenced zinc production in 1978 with a capacity of 50,000 tpa. Since then, continuous investments have been made and resulted in Korea Zinc now being the biggest zinc and lead producer in the world. In the course of increasing zinc production capacity, the company management realized that the company needed a lead smelter to prevent the solid waste disposal problems by treating the lead containing solid waste. This paper begins by briefly stating the objectives and the roles of the lead smelter at Onsan. It describes the QSL process and the TSL process, and further extends the description of the TSL process, through which harmless inert solid waste could be produced for secure final disposal. It is also mentioned that the TSL process plays an important role in the integration of zinc and lead production. Throughout the paper, a positive attitude in handling the environmental issues in the non-ferrous smelting industry is emphasized. Finally, a solution for the solid waste disposal problem in the zinc and lead industry is proposed, with which the environmental issues can be an opportunity rather than a crisis. The current operation at Onsan suggests the future direction of the non-ferrous smelting technology that all of the zinc and lead producers should pursue.

Notes

Advances in MEMS and Optical Packaging - II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Sungho Jin, University of California-San Diego, Department of Mechanical & Aerospace Engineering, La Jolla, CA 92093-0411 USA; Darrel R. Frear, Motorola, Tempe, AZ 85284 USA; Andrei M. Shkel, University of California-Irvine, Department of Mechanical & Aerospace Engineering, Irvine, CA 92697-3975 USA; Martin Weiser, Honeywell Electronics Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Monday PM Room: 15B
March 3, 2003 Location: San Diego Convention Center

Session Chairs: Darrel R. Frear, Motorola, Tempe, AZ 85284 USA; Martin Weiser, Honeywell Electronics, Plated & Discrete Products, Spokane, WA 99216 USA

2:00 PM Invited
The Challenges and Constraints of Packaging Optical MEMs: *Mark Paczkowski*[1]; [1]Lucent Technologies, Bell Labs., 600 Mountain Ave., Murray Hill, NJ 07974 USA

Microelectromechanical sytems (MEMs) are beginning to play an important role in the transmission of optical signals. The number of network elements containing MEMs is growing daily and includes components such as variable optical attenuators, wavelength tunable lasers, reconfigurable add-drop and optical cross-connects. The method by with the MEMs technology is applied varies and includes both 2D and 3D configurations. The advantages associated with using MEMs structures in these optical devices include the high degree of integration and density that can be achieved using either surface or bulk silicon micro-machining techniques and an improved mechanical reliability of the micro-machined parts over conventional macro-machined parts. While new micro-fabrication techniques of three-dimensional microelectromechanical devices presents a platform to create new optical components, there are huge challenges in the packaging of these devices in order to make these components cost effective. In this talk, I will discuss the issues in packaging optical MEMs devices and some of the approaches taken to make these components practical.

2:30 PM Invited
Thermally Compensated 3-Port Packages Used in DWDM Modules: *Michael Uschitsky*[1]; [1]Corning, Inc., Adv. Photonic Tech., 17595 Mt. Herrman St., Fountain Valley, CA 92708 USA

This paper discusses an innovative packaging process of 3-port filtering devices used in dense wavelength-division multiplexing (DWDM) modules. The developed process meets the contradicting requirements for the precision alignment and in-situ adhesive bonding of dissimilar glass optical components and metal protective units. Thermally compatible adherent and adhesive materials, including high expansion glasses, moderate expansion alloys and the filled adhesives with the coefficient of thermal expansion matching these glass and metal adherents provide thermally compensated filtering in the bonded assembly. With use of UV/heat curable epoxies, the process includes UV initiation of the adhesive bond (1), thermally assisted stress relaxation cycling (2), a heat cure (3), and a final post-cure (4), all of which improves the microstructure of the bond. Low-temperature soldering is used to encapsulate the assembly in the metal enclosure. Manufacturing these packages and exposing them to severe moisture.

3:00 PM Invited
Alignment and Joining Processes in Optoelectronic Packages: *Nagesh R. Basavanhally*[1]; [1]Lucent Technologies, Bell Labs., 600 Mountain Ave., Murray Hill, NJ 07974 USA

Electronic packaging involves maintaining the functionality of semiconductor devices by providing mechanical support and electrical interconnections, while protecting the device from the operating environment. The choice of material and joining processes used in packaging are based on electrical, thermal and chemical issues. In optoelectronic packaging, in addition to the above-mentioned challenges, one has to deal with an optical port that channels the optical signal in and out of the device. The optical interconnection, be it through an optical fiber, a waveguide, or free-space, requires mechanical alignment with the device This alignment, which can be active or passive, can have a degree of tolerance ranging from sub-micron to a few microns. Therefore, the challenge is in choosing the appropriate material and joining processes, so as to maintain the required alignment during entire lifetime of the product. Typically, the joining processes include the use of adhesives, soldering and welding, and oxide bonding. The choice of method depends on various requirements, such as alignment tolerance, optical path impairment, strength, hermeticity, etc. In this paper, we will present different joining techniques used in optoelectronic and MEMS packaging using practical examples.

3:30 PM Invited
Packaging of Nano-Structured MEMS Microtriode Devices: *Leon Chen*[1]; Sungho Jin[1]; [1]University of California-San Diego, Dept. of Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92130-0411 USA

Combining the world of MEMS with that of nano can lead to new devices with unique and advanced functionalities. Carbon nanotubes are fascinating new nano materials with many interesting physical, chemical and electronic properties and potential new applications. We have incorporated nanotubes into the MEMS structure to create on-chip, miniature vacuum tubes of microtriodes that can be useful for high frequency microwave communications. A proper bonding and assembly processes were essential in the packaging of such devices, especially for the purpose of providing reliable electrical connections for evaluations of electron field emission behavior and triode amplifying characteristics. In this talk, various bonding approaches and electrical performances of the devices will be described.

4:00 PM Break

4:20 PM
Development of Free Standing Metal/Polymer Films for Packaging Air-Coupled Acoustic Microsensor MEMS: Yan Liu[1]; *Matt OiKeefe*[1]; Asye Beyaz[2]; Chris Singleton[2]; Tom Schuman[2]; [1]University of Missouri-Rolla, Metallurgl. Eng., 1870 Miner Cir., 292 McNutt, Rolla, MO 65409 USA; [2]University of Missouri-Rolla, Chem. Dept., 1870 Miner Cir., 142 Schrenk Hall, Rolla, MO 65409 USA

Synthesis and fabrication of free-standing enclosures for packaging air-coupled, acoustic microsensor systems that allows for acoustic waves from all directions to reach a MEMS microphone with minimal signal attenuation are being evaluated. Bi-layers of metal/polyaniline (PAni) have been fabricated by sputter deposition of aluminum, iron, copper and titanium onto free standing films of PAni in its emeraldine base (EB) form. Characterization of these films via scanning electron microscopy, transmission electron microscopy, and four-point probe resistivity measurements has been done to investigate the morphology, microstructure, and electrical properties of the samples. Chemical bonding and interaction of the metal atoms with EB films of PANi have been investigated using Auger electron and X-ray photoemission spectroscopy. Results from the study indicate that the type of metal influences the interactions and properties of the metal/polyaniline interface and free-standing films. Preliminary acoustic testing results indicates that there is minimal acoustic energy absorption below 2000 Hz.

4:40 PM
The Effect of Thickness on Strength and Ductility of Ni Thin Foils: *Nichole Kristin Whitney*[1]; Fereshteh Ebrahimi[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, Gainesville, FL 32611 USA

Micro-electro-mechanical-systems (MEMS) are combinations of structures, actuators, sensors, and electronics on a silicon substrate made possible by micro-fabrication technology. To increase reliability of these micro-sized elements, it is necessary to determine the mechanical properties of free standing thin foil materials. There have been indications in the published literature that the tensile properties of metallic thin foils may depend on the thickness. Nickel and its alloys are candidate materials to be used in MEMS. The objective of this study was to evaluate the tensile stress-strain relationship of Ni 200 samples with varying thickness ranging between 58 micrometers up to 1.5 millimeters. The Nickel samples were tested in annealed and cold worked conditions. This paper discusses the effects of grain size to thickness ratio and sample misalignment on the tensile strength and elongation of nickel foils.

5:00 PM
Evolution of Microstructures During Cu Wafer Bonding: *K. N. Chen*[1]; A. Fan[1]; C. S. Tan[1]; R. Reif[1]; [1]Massachusetts Institute of Technology, Microsys. Tech. Lab., 60 Vassar St., Rm. 39-623, Cambridge, MA 02139 USA

This paper summarizes all material results on copper wafer bonding for three- dimensional integrated circuits. Material issues including interfacial morphologies, grain structure and oxide composition were observed in Cu-Cu bonded layer under different process parameters. By changing the bonding temperature, time, pressure and approaches, the strength of the bonded layer can be improved effectively. When the wafers are bonded at 400°C for 30 min following by 30 min nitrogen annealing, the bonded interface cannot be observed anymore. Grain growth and interface diffusion are possible reasons for this result. In addition, the oxygen compositions at different location in the bonded layer are all lower than 3 wt. %. No particular high oxygen composition is observed in the bonded interface area. This phenomenon also suggests the strong interdiffusion at interface during bonding and annealing. Possible mechanisms are proposed to explain these phenomena.

Alumina and Bauxite: Precipitation and Alumina Products

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: David Kirkpatrick, Kaiser Aluminum & Chemical Group, Gramercy, LA 70052-3370 USA; Steve Rosenberg, Worsley Alumina Pty, Ltd., Process Chemistry Group, Collie, West Australia 6225 Australia; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday PM — Room: 3
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: David Kirkpatrick, Kaiser Aluminum & Chemical Group, Gramercy, LA 70052-3370 USA; Steve Rosenberg, Worsley Alumina Pty Ltd., Process Chem. Grp., Collie, W. Australia 6225 Australia

2:00 PM Introductions

2:15 PM
Effect of Temperature on the Agglomeration of Fine Particles of Hydrate: *Xie Yanli*[1]; [1]Northeastern University, Sch. of Metall. & Matls., PO Box 117, Shenyang, Liaoning 110004 China

Agglomeration, one of the most important mechanisms in seed precipitation of sodium aluminate liquors, is the key method to enhance the coagulation of fine particles and get coarse hydrate particles. Therefore, it is necessary to utilize the mechanism of agglomeration to satisfy the requirement of producing sandy alumina; while temperature is the main factor which influence the agglomeration but some research results demonstrated that agglomeration could happen only if the temperature was higher than 75C, however, the highest precipitating temperature in China is lower than 65C. In order to guide the production and use the mechanism efficiently, the effect of temperature on agglomeration was studied in laboratory batch isothermal precipitator. The results illustrated that there existed an optimum agglomerating temperature for every solution that decreased with the increasing of the initial supersaturate of the liquor. And the optimum temperature for Chinese industrial liquor is higher than 75C.

2:40 PM
Crystallisation Rate Behavior of Aluminium Hydroxide as a Function of Solution Concentrations: *Andrea Ruth Gerson*[1]; *Huixin Li*[1]; Jonas Addai-Mensah[1]; John Thomas[2]; [1]University of South Australia, Ian Wark Rsrch. Inst., Mawson Lakes Campus, Adelaide, S. Australia 5095 Australia; [2]University of South Australia, Sch. of Physics & Elect. Sys. Eng., Mawson Lakes Campus, Adelaide, S. Australia 5095 Australia

Dynamic light scattering measurements on NaOH/Al solutions of molar ratio 1.22 showed that the particle growth rate was most rapid for a solution of 3 M NaOH and slowed on increasing and decreasing NaOH concentration. For fixed 4.1 M Al solutions, the growth rate was also found to decrease on increasing NaOH concentration (5.0 to 7.0 M). Therefore the growth rate is determined by the NaOH concentration, at least above 3 M NaOH, rather than the Al concentration. Nucleation induction times, measured by UV-vis, for the NaOH/Al 1.22 molar ratio solutions, increased below 2.0 M NaOH and above 5.0 M NaOH. It is of interest that both the induction time increases and particle growth rate decreases at low NaOH concentrations even though the relative supersaturation increases with decreasing NaOH. These trends are thought to be indicative of a change in NaOH concentration-dependent particle growth mechanism.

3:05 PM
Preparation of Nanosized Alumina Powder by Phase Transfer: *Zhao Hengqin*[1]; Li Jie[2]; Lai Yanqing[2]; Liu Yexiang[2]; [1]Zhengzhou Institute of Multipurpose Utilization of Mineral Resources, CAGS, No 328, Longhai W. Rd., Zhengzhou, Henan 450006 China; [2]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

Though liquid phase method is a simple process to prepare nanosized alumina powder, difficulties in separating prepared nanosized powder from liquid phase, more evident in China due to backward of separation equipment, make it very hard to produce nanosized alumina powder in industrial scale by liquid phase method. With $NH_4Al(SO_4)_2$ and NH_4HCO_3 as raw material to prepare nanosized alumina powder, nanosized alumina powder with particle diameter ranging from 20nm to 100nm was obtained by phase transfer in which white oil was used as organic phase and SPAN as surfactant. The particle diameter of the prepared nanosized alumina powder was determined by TEM. Due to the fact that nanosized precipitate was surface modified and transferred into organic phase with smaller volume than water, separation of nanosized precipitate was easily realized and industrialized.

3:30 PM
A Method for Evaluating Seed Balance Parameters in Alumina Refinery Seed Classification Systems: *Walter M. Bounds*[1]; [1]2583 Woodland Ridge Blvd., Baton Rouge, LA 70816 USA

An important consideration in controlling alumina refinery seed classification systems is ensuring that quantity and particle size distribution for seed produced matches with that for seed charged. When this condition is not satisfied, the system may not be stable, resulting in changing seed inventory quantities and particle size distribution. In this paper, information from a previously reported method for evaluating operating characteristics of seed classifiers is utilized to simulate a classification system and determine seed charge quantities and particle size distribution which balance with seed produced. Out-of-balance cases are described to evaluate consequences, and balanced cases are developed to illustrate the method.

3:55 PM Break

4:15 PM
Research on the Mechanism and Optimum Adding Method of Additives in Seed Precipitation: *Xie Yanli*[1]; [1]Northeastern University, Sch. of Metall. & Matls., PO Box 117, Shenyang, Liaoning 110004 China

To get high solution productivity and obtain sandy alumina is the main goal for alumina refinery although the two parameters is always contradict. While doping additives properly is one of the most efficient methods to produce coarse and high strength hydroxide alumina meanwhile not decreasing liquor productivity. The effect of different adding method of additives on precipitation ratio, particle size and strength of alumina hydroxide is studied in detail. It is found that additives have negative effect on precipitation when they were first mixed with seeds even though they could enhance the procedure efficiently when added to liquors first. Furthermore, the dispersant is another important factors influencing the effects of additives and oil dispersant is better than water, which can enhance the efficiency of additives and make them more stable. The mechanisms of different results from different adding methods were also studied by analyzing the data and SEM photographs.

4:40 PM
Evaluation of Superfines Particles in the Precipitation Circuit Agglomeration Phase: Jes´s Alcal·[1]; *Enio Rodriguez*[1]; [1]CVG-Bauxilum, Zona Industrial Matanzas, Puerto Ordaz Venezuela

The superfine particles (3-2 micron) of hydrate, generated in agglomeration and growth phase in the precipitation circuit have vital importance to guarantee calcined product final granulometry. At C.V.G Bauxilum the content of these superfine particles in the fine seed suspension has been increased 37% in the last three years. This increase of particles is related to the changes of diverse variables in precipitation. This paper relates the impact of the content of superfine particles in the fine seed since 1999 until 2002 on the agglomeration phase grain size and the output of fine particles in the last precipitator, finding out that the unstability of the agglomeration phase granulometry was fundamentally due to the increment of the superfines. It also maintains a special relationship with the thermal decrease done at interstages cooling system of the precipitation circuit growth phase.

5:05 PM
New Alumina Superrefractories Cements and the Using its for High-Temperature Composites: *N. Ilyoukha*[1]; V. Timofeeva[1]; [1]Academic Ceramic Center, 8 Frunze St., Kharkov 61002 Ukraine

The use of CA, BA, SrA based binders shows inferior properties, not good resistance to high temperature and melting materials. In this report the results of development of new alumina superrefractories cements and using its for high-temperature coating. The new cements produced by the solid state sintering, contents compounds having a high melting point was used as raw stuff. High-temperature composites and coating based on superrefractories cements are meant to protect units from influents of temperature more than 2000∞C and used for manufacturing monolithic lining, crucibles used in the melting of pure metals, including alloys on rare-earth melements, for closing one of ceramic modules of fire wall, inrefractory lining of quartzglasstanks, petrochemistry reactors, in burial of radiation fuel by extreme environments in nuclear reactors.

Aluminum Reduction–Potroom Operations Symposium: Potroom Optimization

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Alton T. Tabereaux, Alcoa Inc., Process Technology, Muscle Shoals, AL 35661 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday PM — Room: 6B
March 3, 2003 — Location: San Diego Convention Center

Session Chair: Alton T. Tabereaux, Alcoa Inc., Process Tech., Muscle Shoals, AL 35661 USA

2:00 PM
Resistance Modifier Based on Pot Temperature: *Dayan de Paula Neves*[1]; Ari Ferreira Silva[1]; [1]ALUMAR, BR-135, Km 18 Disto Indutrial de, Sao Luis, MA 65095-050 Brazil

Bath resistivity changes with temperature. High bath temperatures decrease bath resistivity and, other wise, low bath temperature increase bath resistivity. As computer process control considers a fixed resistance target, ACD will increase in hot pots, and decrease in cold pots. Hot pots are normally very stable, and a decrease in ACD can help lower the temperature faster and save some energy. Cold pots can be really unstable, decreasing pot performance. Normally, resistance control apply noise modifiers to make this pots stable. Cold temperature modifiers can prevent pot instability. The purpose of this work is to show how Alumar studied and developed its own control.

2:25 PM
Optimising Maintenance Activities of Mobile Equipment in a Smelter: *Mohammed Ismail*[1]; Dilip Koshy[1]; [1]Dubai Aluminium Company, Dubai 3627 UAE

Dubai Aluminium Company Limited (DUBAL) is one of the largest single site aluminium smelter in the western world, with a production capacity of 536,000 metric tonnes per annum (mtpa) of high quality aluminium and is located in Dubai in the United Arab Emirates spread over 480 hectares. The DUBAL site currently consists of six potlines, a 1450 MW power station, desalination plant, related support facilities and residential area. DUBAL has been certified to the ISO 9001 and 14001 standard. The Casthouse has also been certified to QS-9000. To support its operations, DUBAL has 530 Mobile Equipment (valued at US $16 million) ranging from simple personnel carriers and pick-ups to sophisticated fluoride feeders and Anode Pallet Transporters. This fleet is maintained by the Mobile Equipment Workshop which has over the last two years made quantum leaps in continuous improvement initiatives that have achieved: (1)Fleet availability increasing from 80% to current rate of 96%. (2)Vehicle over 48 hours dropping from 39 in 2001 to as low as zero till June this year. The above has been achieved with a reduction in the operating cost of mobile equipment (50% savings in maintenance materials) and reduction in overtime (by 40%) over a period of 15 months. A summary of the improvements are: Customer focus through pro-active maintenance. Rationalizing and rescheduling the periodic maintenance of all equipment based on actual running hours rather than a time based preventive maintenance. Outsourcing activities that are more cost effective if performed by contractors. Major focus on cost reduction with all the staff analysing the cost of spare parts specially since all the manufacturers are either in Europe or Japan. Way Forward meetings which continuously review the shop maintenance activities. Planning the replacement based on previous experience and actual running hours of the equipment and future major overhauls.

2:50 PM
Carbon Dust Reduction on Soderberg Pots: *Ciro R. Kato*[1]; *Leonardo Paulino*[1]; Anna K. Resende[1]; Agnello J. Borim[1]; Jose G. Freitas[1]; Walfredo P. Filho[1]; [1]Alcoa Aluminio SA, Rodovia Pocos, Andradas, Km 10, Pocos de Caldas, MG 37701-970 Brazil

It is well known that Sˆderberg cells-performance is extremely dependent on the quality of the primordial component of the aluminum electrolysis process: the ANODE. Alcoa AlumÌnio in PoÁos de Caldas has been directing resources in order to improve anode quality throughout the last 5 years. Some examples of the investments in this area are: Anodes Enlargement in height and width; Implantation of Stud Cleaning Equipment (Shot blasting); Implementation of Studs-quality control (segregation and replacement); Optimization of Green Anode Paste production; Potrooms operational practices optimization. Starting in 1997 with the anodes enlargement program, that allowed an increase in aluminum production with lower anode current densities, to the acquisition of automated Stud Cleaning Equipment, the first step to the minimization of carbon dust was set. On year 2000 Alcoa PoÁos assisted by the Swiss R&D Carbon started up a task force to reduce process variability and optimize the paste production. Support from Alcoa technology groups also played an important role in this development. The results of paste production improvements led to a better anode formation which can be seen on some physical properties as Green Anode Density-GAD, Baked Anode Density-BAD, anode electrical resistivity and anode porosity. These parameters had a positive influence on cells overall performance mainly on anode consumption, number of anode problems and carbon dust generation. Besides that, good studs-quality management and significant changes on potrooms operational practices set the foundation to make great strides regarding carbon dust generation - which is one of the main solid wastes of Smelters. This presentation will explain how it was reduced carbon dust generation from 60 to 15 kg/tonAl, contributing to better operation and the achievement of superior Environmental, Health and Safety conditions in Potrooms.

3:15 PM Cancelled
An Introduction to Energy Optimisation Measures in Place at Madras Aluminium Company Limited: *S. Sasikumar*[1]; P. K. Sayeenathan[2]; [1]Madras Aluminium Company, Ltd., Pot Rm., Mettur Dam, Salem Dist., Tamilnadu 636 402 India; [2]Madras Aluminium Company, Ltd., Paste Plant, MetturDam, Salem Dist., Tamilnadu 636 402 India

3:40 PM Break

Aluminum Reduction Technology: Environmental - General

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA; Martin Segatz, VAW Aluminum Technology, Bonn D-53117 Germany

Monday PM — Room: 6B
March 3, 2003 — Location: San Diego Convention Center

Session Chair: Yogesh Sahai, The Ohio State University, Dept. of Matls. Sci. & Eng., Columbus, OH 43210-1179 USA

4:00 PM
Stakeholder Engagement Process in the EIA of an Aluminium Smelter: *Paul Lochner*[1]; *Warren Brooks*[2]; *Roland Pesch*[2]; [1]CSIR, PO Box 320, Stellenbosch 7599 S. Africa; [2]Pechiney, PAS 2005, BP 07, Voreppe 38341 France

Pechiney is planning to build a new aluminium smelter known as PAS 2005. Pechiney has identified South Africa as one of its preferred locations. In investigating the environmental feasibility of the proposed smelter, Pechiney has commissioned CSIR, together with Sandy & Mazizi Consulting (who carries out the public participation process) and a team of specialists, to conduct an Environmental Impact Assessment (EIA). The objective of the EIA is to provide decision-makers with relevant and objective environmental information to determine whether or not the proposal will support sustainable development and whether to accept or reject the EIA application. Pechiney and CSIR will present the different steps of the EIA, focusing on the technical studies undertaken in order to assess the predicted environmental impacts project; as well the comprehensive approach towards the interested and affected parties. Due to a specific public participation process, these should reflect a new standard for future EIAs.

4:30 PM
A New Anode Effect Quenching Procedure: *Pablo Navarro*[1]; Gustavo Gregoric[1]; Osvaldo Cobo[1]; Alfredo Calandra[1]; [1]Aluar Aluminio Argentino SAIC, R&D, PO Box 52, Puerto Madryn, Chubut U9120OIA Argentina

The most common methods for the automatic quenching of anode effects consist in tilting or pumping the anode system, or lowering it until it touches the metal pad. These methods did not render satisfactory results in Aluarís cells and forced us to rely on manual killing by green poling. A new AE quenching procedure was developed based on the principle that each pot technology has a characteristic anode-cathode distance in which a wave in the metal-bath interface develops very fast. In this case the wave is used to produce local short-circuits to the anodes, allowing a fast removal of the isolating layer and a replenishment of alumina in the interpolar volume. The procedure was tested in different pot technologies and showed very low values of anode effect overvoltage and duration, a minimum disturbance to the anode crust, and a high success rate, providing a significant reduction on the perfluorocarbon emissions.

5:00 PM

HF Emission from DUBALís Electrolysis Cell: *Najeeba Hassan Aljabri*[1]; *Koluman G. Venkatasubramaniam*[1]; Yousef Mohammed Ali Alfarsi[1]; [1]Dubai Aluminum Company, Ltd., Process Control, Jebel Ali, Dubai UAE

The overall goal of this paper is to gain better understanding of the environmental aspect of the aluminium smelting cell. It encompasses a study of the relative contributions of operational practices, duct flow, alumina feeding and ambient conditions to the overall HF generation and the different categories of emission. This study was taken up separately in the cell duct and the pot room roof through continuous HF emission monitoring on different cell technologies, operating amperages and correlated to operating practices, flow rate etc. Roof HF emissions are discussed in detail. The contribution to roof HF concentration from different operations was estimated for different buildings each housing different types of cell designs and compared. Airflow measurements were done at same time for open and simulated semi-open building to quantify the total roof HF emissions in kg/t Al. It was seen that anode setting, tapping and background emissions were key contributors to the total emission. During the duct emissio n study it was seen that bar break cells have lower duct HF emissions than point fed cells due to differences in crust condition and feeder holes.

Applications and Processing of Powder Metallurgy Refractory Metals and Alloys: Tungsten and Rhenium Base Alloys

Sponsored by: Structural Materials Division, SMD-Refractory Metals Committee, MPMD-Powder Materials Committee
Program Organizer: John J. Stephens, Sandia National Laboratories, Joining & Coating Department, Albuquerque, NM 87185-0889 USA

Monday PM	Room: 18
March 3, 2003	Location: San Diego Convention Center

Session Chair: Sherri R. Bingert, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM Intro Remarks

2:05 PM

Viable P/M Approaches for Manufacturing Rhenium Parts: *Murali Pandheeradi*[1]; Chengming Wang[1]; Charles A. Meglio[1]; Carole L. Trybus[1]; [1]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA

Rhenium parts made using powder metallurgy (P/M) methods have traditionally required additional processing such as extensive machining to meet the specifications. Two P/M techniques - Powder Injection Molding (PIM) and Cold Isostatic Pressing (CIP) - have been developed to enable the manufacturing of net or near-net shape rhenium parts for high temperature applications. While PIM offers a route to make small intricate rhenium parts, CIP is suitable for larger parts of relative shape complexity from simple to medium. The parts produced are shown to satisfy the density specifications with small enough grain sizes so that the mechanical properties are not adversely affected. Experiments are combined with quick analysis tools (developed for PIM) and finite element modeling (in the case of CIP) in the development of the two processing routes for rhenium. The discussion of rhenium PIM highlights binder selection, as well as the successful use of disposable metal-filled dies, while rhenium CIP emphasizes improvements over current practice. Selection of powder type for CIP and rhenium sintering behavior, which significantly impacts both the P/M methods for rhenium, are also discussed. Acknowledgement: This work was conducted by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation under contract No. N00014-00-C-0544 to the US Navy as part of the US Navy Manufacturing Technology Program.

2:35 PM

Observations on the Mechanical Behavior of Rhenium: *C. L. Trybus*[1]; R. W. Semelsberger[1]; T. A. Kennedy[1]; H. Dong[1]; [1]Concurrent Technologies Corporation, Johnstown, PA 15904-1935 USA

Rhenium presents a formidable challenge to the process engineer. It has limited workability at room temperature and forms oxides at elevated temperatures. Warm working rhenium under a protective atmosphere may promote better formability but there is insufficient knowledge of the flow stress, deformation mechanisms and microstructural evolution to effectively use this method. The objective of this work was to develop the required database to identify optimal parameters to form fully consolidated P/M rhenium. Compression testing was done under a slightly reducing atmosphere on rhenium plate processed to different grain sizes. Stable material deformation occurred at high plastic strains and moderate temperatures (~1000°C) with slow strain rates (< 0.01 sec-1). Material flow was marked by strain rate sensitivity parameters of 0.15 to 0.18 and nearly constant flow stress, about 50%-70% lower than ambient flow stress. True strains of ~ 0.5 were obtained without significant defects.

3:05 PM

Structural Evolution During Microwave Sintering of Fine Grained Tungsten: Ganesh Skandan[1]; *Robert Dowding*[2]; Mohit Jain[1]; Dinesh Agarwal[3]; [1]Nanopowder Enterprises, Inc., 120 Centennial Ave., Piscataway, NJ 08854 USA; [2]AMSRL-WM-MD, Aberdeen Proving Ground, MD 21005-5069 USA; [3]Pennsylvania State University, 207 Matls. Rsrch. Lab., University Park, PA 16802-4801 USA

Processing of tungsten at low cost into complex shapes, such as thin walled cones and hemispheres, is a challenge. Conventional sintering using high temperature furnaces leads to the warping due to non-uniform sintering in thin sections. We have approached this problem by using a fine grain tungsten powder as starting material and by microwave sintering. The advantage of using microwave furnace for sintering is that it uniformly delivers energy throughout the green compact. This eliminates the problem of non-uniform shrinkage if the compact is homogeneous. Microwave sintering also leads to reduction in soak time. We have achieved near theoretical densities for tungsten in less than 30 minutes. The focus of this presentation will be on microstrctural evolution in tungsten during microwave sintering, and the effect of processing parameter on the density and grain size on the final sintered part.

3:35 PM

Low Expansion Heavy Alloys: *Greg Rudd*[1]; [1]Spectra-Mat, Inc., 100 Westgate Dr., Watsonville, CA 95076 USA

Tungsten heavy alloys are used where machinable, ultra-dense materials are desired. They have found use as radiation shields, x-ray collimators, armor penetrating rounds, and weights. They have a low coefficient of thermal expansion (CTE) approaching that of tungsten and good thermal conductivity. This property also makes them desirable substrates to match the CTE of semiconductors (e.g., silicon, SiC, GaN, InP) in electronic, optical, and MEMS (Micro Electro Mechanical Systems) packages. Still lower CTEís are desired, however. We have investigated heavy alloy compositions based on Invar-type alloys that have CTEís even lower than tungsten, but with thermal conductivity similar to tungsten. We report the powder metallurgical processing, compositions and properties of these materials.

4:05 PM

Optimisation of Sintering Parameters for PM Tungsten Base Heavy Alloy: Vittal Mahender[1]; *V. S.R. Murti*[2]; *Pappula Laxminarayana*[2]; [1]International Advanced Research Centre, A.R.C.I., Balapur, Hyderabad, Andhra Pradesh 500 005 India; [2]Osmania University, Mechl. Eng., Univ. Col. of Eng., Hyderabad, Andhra Pradesh 500 007 India

Tungsten, a high strength-refractory material, is not amenable for processing by conventional methods and require powder metallurgy technique. However in PM parts the density is poor with lower hardness and strength. The present work employs liquid phase sintering and aims at optimising the sintering parameters to obtain maximum density, hardness and strength. The tungsten heavy alloy has the composition of 95% tungsten, 3.5% nickel and 1.5% iron. At the sintering temperature nickel and iron melt and provide a uniform coating on tungsten powder. The tests involved several iterations of different combinations of sintering temperature(1460∞C upwards in steps of 40∞C) and atmosphere (vacuum, inert gas, cracked ammonia and hydrogen) to get best possible density, hardness and strength. The optimum results occurred when sintering was done in vacuum and at a temperature of 1580∞C to obtain a density of 18gm/cc, hardness 370VPN and compressive strength of 1600MPa.

4:35 PM

Application of ECAE to Processing Tungsten Heavy Alloys: *Thomas M. Lillo*[1]; Yevgeny (Jenya) Macheret[2]; Robert L. Goetz[3]; [1]Bechtel BWXT, Idaho, Matls. Dept., MS 2218, PO Box 1625, Idaho Falls, ID 83415-2218 USA; [2]IDA, Sci & Tech. Div., 4850 Mark Center Dr., Alexandria, VA 22311 USA; [3]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA

Although tungsten possesses sufficiently high density, its deformation behavior is not attractive for penetrator materials applications. We employed ECAE of liquid phase sintered W-7Ni-3Fe alloy (WHA) in an attempt to develop microstructure for improved high velocity impact behavior. The severe plastic deformation nature of ECAE coupled with relatively low processing temperatures (~300°C) and limited ductility of the WHA initially caused billet fracture. Finite element modeling revealed large tensile stresses in the billet during ECAE processing. FEA modeling also showed that backpressure applied to the billet during processing would alleviate tensile stresses. The ECAE die was subsequently modified to provide the backpressure, and the WHA has been successfully processed. In this paper we present the results of the FEA and microstructural analysis of the processed billets.

5:05 PM
An Examination of the Interparticle Contact Area During Sintering of Tungsten: *David Mitlin*[1]; Randall M. German[2]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA; [2]The Pennsylvania State University, 0118 Rsrch. Bldg. W., University Park, PA 16802 USA

As a powder compact sinters its microstructure evolves. One way to quantify the scale of the microstructure is to consider the interparticle contact area. This study examines two known models for calculating the interparticle contact area; the classic two-sphere model and Voronoi cell model. Both models have particular assumptions about the microstructure that make them not applicable for treating densification to near full density with concurrent grain growth. We propose a modified Voronoi cell that accounts for an increasing grain size, making it applicable to a general case where grain growth occurs during sintering. The three models are compared to the interparticle contact area data, obtained by stereology techniques, for W-0.3wt.%Co sintered from green state to near full density.

Automotive Alloys 2003 - II

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA

Monday PM — Room: 5B
March 3, 2003 — Location: San Diego Convention Center

Session Chair: Subodh K. Das, Secat Inc., Coldstream Rsrch. Campus, Lexington, KY 40511 USA

2:00 PM
Effect of Entrapped Eutectic on the Ductility of Semi-Solid-Formed Al-7%Si-Based Alloys: *Jon T. Carter*[1]; Vjekoslav Franetovic[1]; [1]General Motors R&D, Matls. & Processes Lab., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

The fracture path in semi-solid-formed aluminum parts was found to pass through the primary alpha globules only if those globules contained entrapped eutectic. It was established that a certain amount of entrapped eutectic was needed to maximize ductility. This effect on ductility was explained by a model which involves the nucleation of microcracks by entrapped eutectic.

2:15 PM
Formability of Sheet Metal with Pulsed Electromagnetic and Electrohydraulic Technologies: *Sergey Fedorovich Golovashchenko*[1]; Vyacheslav Sabbaydinnovich Mamutov[2]; Vladimir Vladimirovich Dmitriev[1]; Andrew M. Sherman[1]; [1]Ford Motor Company, Mfg. Sys., MD 3135, 2101 Village Rd., Dearborn, MI 48124 USA; [2]St. Petersburg Technical University, Machines & Tech. of Metal Forming Dept., 29 Politekhnitcheskaya St., St. Petersburg 195251 Russia

In this presentation, results on sheet metal forming using pulsed electromagnetic and electrohydraulic technologies will be discussed. Pulsed electromagnetic forming is based on high-voltage discharge of capacitors through a coil. An intense transient magnetic field is generated in the coil and through interaction with the metal work-piece, pressure in the form of a magnetic pulse is built up to do the work. Pulsed electrohydraulic forming is a similar electrodynamic process, based upon high-voltage discharge of capacitors between two electrodes positioned in a fluid-filled chamber. Data on the formability of aluminum alloy 6111-T4 employing these two methods will be compared with data for traditional forming technologies. In addition, results on pulsed forming of sheets into open round die and conical die will be reported for: pure aluminum, aluminum alloys (Al-Cu-Mg-Mn and Al-Mn), copper, low carbon steel, stainless steel, titanium, Ti-Mn-Al alloy, nickel, and brass.

2:30 PM
Fatigue Performance and Microstructure of Hydroformed Aluminum Tubular Sections: *Alan A. Luo*[1]; John M. Tartaglia[2]; [1]General Motors Research & Development Center, Matls. & Processes Lab., 30500 Mound Rd., MC 480-106-212, Warren, MI 48090-9055 USA; [2]Climax Research Services, 51229 Century Ct., Wixom, MI 48393-2074 USA

This study investigated the fatigue characteristics and microstructure of hydroformed sections of 5xxx and 6xxx series aluminum alloys. Room temperature fatigue tests were conducted using tension-tension cycling (R=0.1) in load control with a sinusoidal waveform. The results show that despite their lower yield strength, the hydroformed 5xxx alloy sections have higher fatigue strength than the 6xxx material. The microstructures of the aluminum sections were characterized using optical microscopy and scanning electron microcopy (SEM). The fatigue fracture surfaces were also analyzed under SEM. The fatigue behaviors of the different aluminum alloys were related to their microstructural features such as grain size, orientation and second phase particles.

2:45 PM
Fold Formation in Aluminum Lost Foam Casting: *Qi Zhao*[1]; *Thomas W. Gustafson*[2]; *Mark Hoover*[3]; *Merton C. Flemings*[4]; [1]Metal Casting Technology, Inc., 127 Old Wilton Rd., Milford, NH 03055 USA; [2]General Motors Corporation, GM Powertrain at Metal Casting Tech., Inc., 127 Old Wilton Rd., Milford, NH 03055 USA; [3]General Motors Corporation, Casting Dvlp. & Validation Ctr., MC 486-629-235, 1629 N. Washington Ave., Saginaw, MI 48605-5037 USA; [4]Massachusetts Institute of Technology, 77 Massachusetts Ave., Cambridge, MA 02139-4301 USA

Various types of folds collected from both production and research environments were characterized via different means to understand fold formation mechanisms. Visual inspection (both real time and post-cast) and surface analysis (via XPS and AES) of defects indicated that all the folds analyzed, despite of differences in shape, size, color and occurrence, share the same formation mechanism. It is the variation in growth environment within and between castings that leads to complexity in fold appearance. A model of fold nucleation was proposed to describe the necessary and sufficient conditions for fold formation and to correlate fold occurrence with various process variables, such as mold fill rate, foam pattern decomposition behavior, casting/gating design features, coating performance in both heat and mass transfer, etc.

3:00 PM
Fracture Processes in Semi-Solid-Formed Al-7%Si-Based Alloys: *Vjekoslav Franetovic*[1]; Raymond C. Lints[1]; Jon T. Carter[1]; [1]General Motors R&D, Matls. & Processes Lab., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

The fracture surfaces on tensile bars machined from semi-solid-formed shafts of aluminum alloys 319 and 357 were examined in an SEM to determine the fracture morphologies. For the three metallurgical conditions examined (as-cast, T5, and T6) the crack had generally propagated through the interglobular eutectic, and occasionally through the alpha-aluminum globules. The fracture surface of the eutectic in the T6 condition was ductile, as indicated by dimple-rupture features. The fracture of the eutectic in both the as-cast condition and the T5 condition was a novel mixture of microductile (very small dimples) and quasi-cleavage. This fracture morphology is unique to semi-solid-formed alloys. Alpha globules never fractured unless they contained entrapped eutectic. Such globule fracture was ductile, and exposed the entrapped eutectic.

3:15 PM
Cast Aluminum Alloy for High Temperature Applications: *Jonathan A. Lee*[1]; [1]NASA/Marshall Space Flight Center, Matls. & Processes Dept., MC ED33, Huntsville, AL 35812 USA

Originally developed by NASA as high performance piston alloys to meet U.S. automotive legislation requiring low exhaust emission, the novel NASA alloys now offer dramatic increase in tensile strength for many other high temperature applications from 450F-750F. It is an ideal low cost material for cast automotive components such as pistons, cylinder heads, cylinder liners, turbo chargers, impellers, brake calipers and rotors. As a newly developed aluminum-silicon alloy, with silicon content ranging from 6% to 18%, it can be very economically produced from using conventional permanent mold or sand casting. At high silicon levels, the alloy also exhibits excellent thermal growth stability, surface hardness and wear resistant properties.

3:30 PM

Short Solution Heat Treatment of Semi-Solid-Formed Aluminum Alloy 357: *Vjekoslav Franetovic*[1]; Jon T. Carter[1]; [1]General Motors R&D, Matls. & Processes Lab., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

The effects of solution-treatment time on the tensile properties, hardness, fracture mechanism, and microstructure of subsequently age-hardened alloy 357 were studied empirically. Solution treatment times of a few minutes were found to be sufficient to change the mechanical properties. These results are explained in terms of diffusion-induced rounding of the eutectic silicon particles and the subsequent influence on fracture.

3:45 PM

Optimizing the Reuse of Light Metals from End-of-Life Vehicles: *Alexandre Cosquer*[1]; Randolph E. Kirchain[1]; [1]Massachusetts Institue of Technology, Matls. Sys. Lab., 77 Massachusetts Ave., Cambridge, MA 02139 USA

In 1994, Gorban, Ng and Tessieri demonstrated a feasible recycling system in 2010 in which almost all scrap from end-of-life vehicles could be consumed in new automotive production and suggested improvements to increase recyclability of aluminum. Nevertheless, they did not explore all the capabilities of linear optimization in their case study. This paper will focus on linear optimization and will show how it can help address the problems of achieving closed loop recycling for automotive aluminum. The use of linear optimization sensitivity parameters (shadow prices, opportunity costs) will shed light on the following issues: alloys choice in car design, compositional modifications of the alloys and the value of upgrading post-consumer scrap. This paper will highlight some strategies for increased recycling of light metals from current and future vehicles. Initial optimization results show that through strategic alloys choice, recycling rates and/or costs can be improved.

4:00 PM

The Effect of Short Thermal Excursions on the Precipitation Behaviour of AA6111: *Babak Raeisinia*[1]; David J. Lloyd[2]; Warren J. Poole[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Alcan International Ltd., Kingston R&D Ctr., Kingston, Ontario K7L 5L9 Canada

Natural ageing after solution treatment has been found to be deleterious to subsequent artificial ageing in Al-Mg-Si-(Cu) alloys. The emphasis of this work is to investigate the effect of short thermal excursions on the stability of naturally aged materials. To examine this phenomenon, electrical resistivity measurements at 77 K have been conducted for a series of short ageing treatments in the temperature range of 200-300°C. The starting structure was either i) a supersaturated solid solution, ii) a 24 hour naturally aged sample or iii) a 2 week naturally aged sample. The kinetics of the resistivity change have been interpreted in terms of the competition between the dissolution of clusters formed at room temperature and the formation of high temperature precipitates.

4:15 PM

The Effects of Heat Treatment on the Strength of Open-Cell Aluminum Foams: *Jikou Zhou*[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Eng., Princeton, NJ 08544 USA

This paper examines the effects of heat treatment on the strengths of open-cell 6101 aluminum foams. The stress-strain behaviors of struts extracted from the as-received and aged foams are determined using micro-tensile testing techniques. The measured strut tensile properties are then used to predict the foam strengths using unit cell models. Finally, the implications of the results are discussed for the microstructureal design of strong foams.

4:30 PM

Effect of Thermomechanical Processing on Texture Development in a Twin-Belt Cast Automotive Aluminum Alloy: *Hamid N. Azari*[1]; StÈphane X. Girard[1]; David S. Wilkinson[1]; [1]McMaster University, Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

The effect of thermomechanical processing on texture development in a twin belt cast AA5754 automotive aluminum alloy has been investigated. The as-cast alloy exhibits an equiaxed structure with clusters of intermetallic particles rich in iron and centreline porosity. The thermomechanical processing schedule consisted of cold or warm rolling, intermediate annealing, cold rolling and final annealing. Warm rolling induces a coarser and less homogeneous grain structure. The level of secondary rolling is a key parameter, with an increase in final cold work resulting in a finer and more homogeneous grain structure. Taylorís model successfully predicts rolling texture development along the · and , fibres. Such a texture coupled with high temperature annealing promotes the development of the CuND component at the expense of the cube component. Increased iron content in the range 0.09 to 0.28 wt.% leads to a finer grain structure but does not have any effect on the texture.

4:45 PM

Study of the Kinetics of Precipitation During Artificial Aging of Al-Mg-Si(-Cu) Alloys Using Calorimetry Methods: *Shahrzad Esmaeili*[1]; Warren J. Poole[2]; David J. Lloyd[1]; [1]Alcan International Ltd., Kingston R&D Ctr., PO Box 8400, Kingston, ON K7L 5L9 Canada; [2]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Isothermal calorimetry and differential scanning calorimetry were used to obtain the kinetics of precipitation during artificial aging of Cu bearing as well as Cu free Al-Mg-Si alloys. A variety of thermal histories prior to artificial aging of the Cu bearing alloy were chosen and the applicability of the individual calorimetry analysis for these histories was determined. The effect of thermal history on the precipitation kinetics was related to the evolution of microstructure during aging. It was found that pre-aging processes resulted in slower precipitation kinetics during artificial aging mainly due to lower precipitation rate for βî phase. For the solution treated as-quenched histories, slower precipitation kinetics was found for the Cu free alloy. Interesting information on the microstructural differences between the Cu bearing and Cu free alloys was also obtained.

5:00 PM

Roll Casting of Aluminum Alloys: *Burton R. Patterson*[1]; Hasso Weiland[2]; Ming Li[2]; [1]University of Alabama at Birmingham, Dept. of Matls. Sci. & Eng., 1530 3rd Ave. S., BEC 254, Birmingham, AL 35294 USA; [2]Alcoa Technical Center, 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

Roll casting production of low alloy aluminum sheet has shown considerable energy and cost efficiency and is currently a mainstream technology. This process has not yet been extended to more highly alloyed aluminum and little is known about the relationship of the roll casting process variables to the microstructure and properties of these alloys. This new program is currently investigating these processing/microstructure/property relationships in several high alloy aluminum systems with the goal of understanding the relationships to enable optimization off structure and properties to take advantage of the potential efficiencies in future production.

5:15 PM

Modeling of Anisotropic Plastic Flows of Automotive Sheet Metals: *Wei Tong*[1]; Nian Zhang[1]; Xiquan Jiang[1]; [1]Yale University, Mechl. Eng., 219 Becton Ctr., 15 Prospect St., New Haven, CT 0620-8284 USA

Rolled sheet metals that are used extensively in the automotive industry are typically orthotropic with symmetry axes defined in terms of the rolling, transverse, and normal directions. An effective and accurate description of anisotropic plastic flow behaviors of automotive sheet metals is critical for both improving existing and developing new sheet metal manufacturing technologies. In this presentation, a newly developed anisotropic plasticity theory is summarized and is applied to describe flow surfaces of orthotropic polycrystalline steel and aluminum sheet metals under plane stress. The theory is formulated by considering the anisotropic flow of a sheet metal in terms of a discrete set of planar macroscopic slips or shearing modes. An internal state variable other than the effective plastic strain or equivalent plastic work is introduced to define the isotropic hardening state of the sheet metal. Effects of planar anisotropy of a sheet metal on its isotropic hardening behavior are explicitly taken into account. The new anisotropic plastic flow theory requires a number of anisotropic material functions to describe accurately the flow surfaces of a sheet metal under plane stress. These anisotropic material functions can be specified based on the orthotropic symmetry of the sheet metals and the direct determination of their material constants by single balanced biaxial tension and multiple uniaxial tension tests at various offset angles from the rolling directions are elucidated. It is shown that the accuracy of the anisotropic flow model can be steadily improved using increasing number of the uniaxial tension tests. Anisotropic material functions and their parameters have been identified for a large number of automotive steel and aluminum sheet metals based on the experimental data reported in the open literature. Some preliminary experimental results on biaxial plastic strain ratio under balanced biaxial tension and plastic shear strain ratios under uniaxial tension are reported and it is shown that they can be incorporated into the new theory for further improving the accuracy of anisotropic plastic flow modeling.

Cast Shop Technology: Continuous and DC Casting

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; David H. DeYoung, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday PM — Room: 6C
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Wolfgang Schneider, VAW Aluminium Technology, R&D Cast Tech., Bonn 53177 Germany; Jake Niedling, Alcoa Inc., Ingot Tech., Knoxville, TN 37902-1810 USA

2:00 PM
Measurement of the Onset of Hot Cracking in DC Cast Billets: *BenoÓt Commet*[1]; Pascal Delaire[1]; Jan Rabenberg[2]; Joost Storm[2]; [1]PECHINEY, Ctr. de Recherches de Voreppe, 725, rue Aristide BergËs, BP 27, Voreppe, Cedex 38341 France; [2]CORUS, Rsrch., Tech. & Dvlp., PO Box 10000, (3G14), IJmuiden NL-1970 CA The Netherlands

Hot tearing is a major cause of DC casting production loss. Billet material is most vulnerable for centre cracks at start-up or at too high casting speed. The present work aims to understand the involved mechanisms and focuses on the steady state regime. Castings are performed to determine the speeds of crack at initiation and healing. The billets start with sound feet. Then the casting speed is gradually increased until a crack is detected by means of ultrasonic inspection. Then the speed is gradually decreased until the billet is again sound, all casting parameters being recorded. Numerous samples are cut in region of interest and are characterised metallographically. Experiments are performed varying the alloy class (3003 vs. 5182-01), chemical composition, grain refinement and hydrogen content. Some thermal numerical analyses are used to translate the casting parameters into more intrinsic hot tearing parameters, like the mushy zone thickness.

2:25 PM
The Influence of Caster Roll Diameter on the Microstructure of Twin Roll Cast Aluminum Strip: *Murat Dundar*[1]; A. Soner Akkurt[1]; Kemal Sarioglu[1]; Chris Romanowski[2]; [1]Assan Aluminium Works, Tuzla, Istanbul 81700 Turkey; [2]FATA Hunter, Inc., 1040 Iowa Ave., Ste. 100, Riverside, CA 92507 USA

In recent years the trend to wider casting widths and tighter sheet profile tolerances has resulted in increasing roll diameters on twin roll casters. There has been some speculation within the aluminum industry regarding the influence of caster roll diameter on the microstructure of the twin roll cast aluminum strip. Assan Aluminium is a unique position to investigate this effect as it operates a variety of FATA Hunter twin roll casters with roll diameters ranging between 660 mm and 1120 mm. Tests were conducted using the same alloy on each caster. Grain angle measurements were taken to ensure that each machine was operated with an equivalent amount of hot-work in the roll bite. The as-cast microstructure for each roll size was characterized in terms of cooling rate gradient, grain size distribution, segregation and any differences in response to thermo-mechanical processing.

2:50 PM
Improved VDC Air Pressurised Billet Casting Mould for Al-Sn Alloys: *Stephen Stuart Instone*[1]; Wolfgang Schneider[1]; Manfred Langen[1]; [1]Hydro Aluminium Deutschland GmbH, R&D, Georg-von-Boeselager-Str. 25, Bonn, NRW 53117 Germany

An improved air pressurised billet casting mould for aluminium alloys was developed to permit the casting of specialty aluminium alloys containing various amounts of tin. In these alloys tin is added to improve machinability and emergency running properties in bearing applications. Tin additions above about 1% significantly affect the castability of these alloys due to the large increase in the alloy solidification range. Consequently the alloys become extremely difficult to cast even when state of the art air pressurised casting technology with aluminium or copper moulds are employed. The new mould utilises both graphite and aluminium at the mould face which enables these types of alloys to be easily cast to the highest quality standards. This paper describes the design of the mould and also provides details of comprehensive temperature measurements that were made to asses the effect on heat flow within the mould when a second material is introduced into the critical area of mushy zone contact on the mould face. Micrographs and results of metallographical measurements of the important surface zone of the cast billets are also presented.

3:15 PM
Influence Parameters on Continuous Casting of Aluminium: *Hubert Sommerhofer*[1]; [1]University of Leoben, Dept. of Nonferrous Metall., Franz Josef Str. 18, Leoben 8700 Austria

Continuous Casting is a very effective process to produce semi products in an economic and productive way. Many different metals and alloys may be cast in that way, products are billets, rolling ingots, strips, plates, wires and in special cases thixoforming materials. The development of the strand is mainly influenced by the casting parameters and the mould design. A short time of sticking at the mould wall and the afterwards arising air gap are the reason for surface defects and inhomogen microstructure. Therefore many different solutions have been developed but have not really satisfied enough up to now. So further development work is still necessary to overcome the problems of surface defects. Experiments at the department of non ferrous metallurgy at the university of Leoben seem to be very promising and will open up new prospects for the future.

3:40 PM Break

3:50 PM
Casthouse Modifications for Improved Slab Quality: *Barbara Rinderer*[1]; Phillip Austen[2]; Andrew Tuff[2]; [1]Comalco Research & Technical Support, Metal Products Tech., 15 Edgars Rd., Thomastown, Victoria 3074 Australia; [2]Comalco Aluminium (Bell Bay) Limited, Metal Products, George Town, Tasmania 7253 Australia

The quality of rolling slab is critical for rolled sheet and foil. The Comalco Bell Bay slab casting facility was upgraded for full automation in 1995. The impact of more recent casthouse modifications on rolling slab quality are presented. The modifications include the introduction of low head composite (LHC) moulds to replace the conventional aluminium moulds used previously. This has resulted in improved surface condition and reduced mould chill zone thickness allowing reduced scalp depth prior to rolling. In addition, the introduction of LHC has enabled enhanced productivity. The hydrogen and alkali removal efficiencies were monitored during introduction of the Alcan Compact Degasser (ACD) to replace the previously used Alpur D5000 degasser. The ACD introduction was primarily for ease of alloy changes, greater equipment availability and reduced performance variation. A Staged CFF Filtration System completes the equipment upgrade.

4:15 PM
3D Simulation of Solidification Process Used in Twin Roll Casting: *Kemal Sarioglu*[1]; Philippe Thevoz[2]; [1]Assan Aluminium Works, R&D Dept., E5 Karayolu 32.KM, Istanbul 81700 Turkey; [2]Calcom SA, Parc Scientifique-EPFL CH-1015, Lausanne 1015 Switzerland

Simulation of solidification processes has become an important tool for continuous casting applications. Modeling approaches must be determined adequately to clarify the process. It is not easy to determine the boundary conditions needed to solve the problem. Commercial software named CalcoSOFT has been used to model the process. The package calcosoft enables to quickly check the influence of each parameter separately and to gain an in-depth understanding of the process. Fluid metal velocities, temperatures, fraction of solid regions has been calculated. The calculated values has been tested in-situ process. Modeling of the process reduces the experimental testing trials.

4:40 PM
Evaluation of Non-Mechanical and Mechanical Hot Tearing Criteria for DC Casting of Aluminum Alloys: *Mr. Suyitno*[1]; [1]Delft University of Technology, Lab. of Matls., Rotterdamseweg 137, Delft 2628 AL The Netherlands

One method to reduce the occurrence of hot tearing during DC casting is done by optimizing the process through simulation. However, the simulation approach needs criteria to determine whether hot tearing will occur. Several criteria are proposed in literature. In this paper, three non-mechanical [1-3] and two mechanical [4,5] criteria are evaluated for hot tearing prediction. In the simulation, a finite element model is used, which couples thermal and stress computations. The material flow model used in the computation is fitted to constitutive models which are valid for the whole temperature range in DC casting. In the solid state a modified Ludwik model, and in the semi-solid state an elasto-viscoplastic model is used. The output of the finite element model is used as input for the hot tearing criteria. The results are compared and the sensitivity of the criteria to the casting speed is reported.

5:05 PM
Permeability of the Mushy Zone in Aluminum Alloys: Evaluation of Different Approaches: Johan W.K. Boggelen[1]; *Dmitry G. Eskin*[1]; Laurens Katgerman[2]; [1]Netherlands Institute for Metals Research, Rotterdamseweg 137, Delft 2628AL The Netherlands; [2]Delft University of Technology, Appl. Matls. Sci., Rotterdamseweg 137, Delft 2628AL The Netherlands

Three different approaches to evaluate the permeability of the mushy zone were compared. Experimentally the permeability of several Al-Cu and Al-Si alloys was determined for volume fractions of liquid in the range from 0.2 to 0.35 in a permeameter based on the design by Nielsen. Then the microstructure data was used as an input for some analytical models, and the permeability was calculated. Hence, the experimentally determined permeability was compared with calculated one. Finally, numerical simulations were done using the Flow3D software package, both on simple model structures that consisted of periodic arrays of spheres and on complex structures that resembled the dendritic structures as in the permeability experiment mentioned above. These numerical results are compared with those obtained by analytical models for the same model structures. It is shown that permeabilities obtained by all three techniques agree well with each other. For the liquid fractions between 0.1 and 0.4% the Kozeny-Carman relationship is the simplest and accurate enough. However, its application to the liquid fractions approaching zero is yet to be investigated.

Cast Shop Technology: Melting and Alloying

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; David H. DeYoung, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Monday PM	Room: 6D
March 3, 2003	Location: San Diego Convention Center

Session Chairs: David Neff, Metaullics Systems Company, Solon, OH 44139 USA; Debi Van Dall, Air Products and Chemicals Inc., Allentown, PA 18195 USA

2:00 PM
Secondary Aluminum Melting Research in a Laboratory Scale Reverberatory Furnace: *John A. Clark*[1]; [1]US Department of Energy, Albany Rsrch. Ctr., 1450 SW Queen Ave., Albany, OR 97321 USA

The Albany Research Center (ARC), US Department of Energy, has developed a 175-pound capacity, natural gas, direct-fired reverberatory furnace. The high temperature reactions present during the aluminum remelt process are being investigated. Preventing dross formation has been the key aim to date. Reducing losses to dross by 25-50% will potentially lead to an annual energy savings of over 75 trillion BTUís by the year 2020. Schematics and operation characteristics of the ARC laboratory scale reverb will be presented. Potential gas-solid, gas-liquid, and liquid-solid interactions between the hot combustion gases, aluminum, and refractories will be discussed.

2:25 PM
The Effect of Solidification Defect on the Dross Formation during Re-melting of Aluminum 5182 Alloy RSI: *Qingyou Han*[1]; William A. Simposon[1]; John Zeh[2]; Vinod K. Sikka[1]; Edward A Kenik[1]; Peter Angelini[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6083 USA; [2]Logan Aluminum, Inc., PO Box 3000, Russellville, KY 42276 USA

This article addresses the effect of solidification defects, such as cracking and porosity, on dross formation during the remelting of aluminum 5182 alloy RSI. Remelting experiments were carried out to measure the dross formation. Optical and electron scanning microscopy were employed to characterize the solidification defects. It was found that a huge amount of dross was formed during the remelting of certain types of RSI which contained severe internal cavities such as interdendritic porosity and hot tears. These cavities provided continuous channels that exposed the internal interdendritic surfaces to the atmosphere outside of RSI. As a result, an oxide layer was formed on the surfaces of the interdendritic pores. Aluminum grains were entrained in the oxide shell, resulting in a large amount of dross formation during the re-melting of 5182 aluminum RSI.

2:50 PM
Refractory Requirements for Alcanís Aluminum Casthouses: *Andris Innus*[1]; Paul Rivard[2]; [1]Alcan International Ltd., Arvida R&D Ctr., 1955 Mellon Blvd., PO Box 1250, Jonquiere, Quebec G7S 3L5 Canada; [2]STAS, 1846 Outarde, Chicoutimi, Quebec G7K 1H1 Canada

Demands on maximizing value have spotlighted attention on molten aluminum confinement refractory used in Alcanís casthouses. Improvements in refractory life cycle reliability and duration are needed to increase furnace throughput and productivity, and to eliminate refractory as a potential inclusion source. Much effort has, and is, being directed by Alcan towards understanding aluminum-refractory interactions. From this effort it is becoming clear that product information provided by refractory suppliers is often insufficient and/or incomparable to facilitate intelligent refractory selection with respect to molten aluminum confinement. This paper will highlight some general needs and concerns with respect to molten aluminum confinement refractory, offer a menu of what information is needed from refractory suppliers and why, and point the way forward towards refractory better responding to the needs of Alcan.

3:15 PM
The Behavior of Selenium Impurities During the Alloying of Aluminum with Manganese Additions: *Roderick I.L. Guthrie*[1]; Mihaiela Isac[1]; Reza Aboutalabi[1]; [1]McGill University, McGill Metals Procg. Ctr., 3610 University St., Wong Bldg., Rm. 2M051, Montreal, Quebec H3A 2B2 Canada

Manganese is an important alloying element for both aluminum and steel melts. In the case of aluminum cast house practices, the manganese is either added as briquettes, or is injected pneumatically into the melt through submerged lances. Some of the electrolytic grade manganese alloys used by the Light Metals Industries can be heavily contaminated with selenium as a result of the electrolytic processing route adopted for their production. Laboratory tests have been carried out in order to simulate the briquette method of alloying and to determine how the selenium distributes itself between the melt, dross and atmosphere. Similarly, since the chemical form of selenium is important from an environmental point of view, being hazardous to humans when present in compound form, chemical analyses of the vapors exhausted from the melt were made.

3:40 PM Break

3:50 PM
DC CastingñMathematical Modeling to Optimize the Running Cost of a Casthouse: *Jan Migchielsen*[1]; [1]Thermcon Ovens B.V., Process Design Dept., PO Box 97, Geldermalsen 4190 CB The Netherlands

When designing a complete casthouse package, the total running cost of such a facility is of paramount importance, every effort is to be undertaken to reduce these cost to enhance the competiveness of the facility where as at the same time consistent quality is to be ensured. To be able to control quality, the parameters for the DC casting process are one of the key issues for the success of the facility. The complex physical process of DCC was captured in a mathematical model which in turn is a part of the model which addresses the running cost of a complete casthouse.

4:15 PM
Aluminum Melting and Dross FormationñA Historical and Modern Perspective of the Problem: *Wesley Stevens*[1]; Gaston Riverin[1]; Vincent Goutieres[1]; [1]Alcan International Inc., Arvida R&D Ctr., 1955 Mellon Blvd., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada

Dross formation during aluminium melting has always been a major concern. Cost reduction should make limiting dross formation a major goal for all smelter casting and remelt centers. Many prior investigations in this field have been conducted by most aluminium producers. A resumÈ of numerous publications is presented, along with the principal findings which have become ´generally accepted common knowledgeª. Field work, and lab scale experiments over the past decade have shown that the common knowledge does not always adequately explain observations being made. This led precise experimentation using a pilot scale melt furnace to study melting phenomena including dross formation. A summary of these experiments in Alcanís Arvida Research and Development Centre will be described. Historical and recent results will be discussed as to how this knowledge can be used to influence future furnace and process design to limit dross formation in aluminum melting.

4:40 PM
Large Capacity Melting System: *Chris T. Vild*[1]; *Alan M. Peel*[2]; [1]Metaullics Systems, 31935 Aurora Rd., Solon, OH 44139 USA; [2]EMP

Technologies, Ltd., Beedles House Easton Ave., Stratton Burton-on-Trent, Staffordshire VE13 0VB Great Britain

The secondary aluminum producer has available a variety of scrap types. While light gauge scrap is usually less expensive to purchase, often by a wide margin, it presents operational challenges which may offset the low purchase price. In 1996, a system was designed for use in aluminum reverberatory furnaces to aid the melting of turnings, borings, UBC and other light scrap while achieving high recovery rates, excellent production rates and low energy consumption. Now the system has been updated with a Electromagnetic Pumping System and a Larger Capacity Design, increasing the scrap submergence rate to 35,000 lbs. per hour. This paper will discuss operating principles, and field data from the new system.

Computational Methods in Materials Education: Insertion of Computational Methods II

Sponsored by: TMS-Education Committee
Program Organizers: Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, State College, PA 16082-5006 USA; Mark D. Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA

Monday PM — Room: 13
March 3, 2003 — Location: San Diego Convention Center

Session Chair: Herbert A. Chin, Pratt & Whitney, Structl. Alloys & Processes, E. Hartford, CT 06108 USA

2:00 PM Invited
Teaching Computational Materials Design: *G. B. Olson*[1]; [1]Northwestern University, Dept. Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

As part of a multiyear design curriculum, a junior-level, one-quarter Materials Design course integrates computational tools in a systems approach to the science-based design of dynamic multilevel-structured materials. In a studio environment, lectures cover systems design principles illustrated by design case studies, while laboratory sessions cover essential tools such as CES, Thermocalc and DICTRA which are employed in student team projects coached by graduate students actively involved in design research. The computational tools are also introduced in other courses, and projects typically include integration of results from applied mechanics (ABAQUS, DEFORM) and quantum physics (FLAPW, VASP) codes.

2:30 PM Invited
Advanced Computations When Teaching Phase Transformations - Experiences: *John A.L. ≈gren*[1]; [1]Royal Institute of Technology, Dept. of Matls. Sci. & Eng., Stockholm 100 44 Sweden

A realistic treatment of phase transformations during heat treatment and usage of alloys requires extensive thermodynamic and kinetic data and advanced software. If such tools are available most students become stimulated by the the fact that they can tackle realistic problems, sometimes even in collaboration with industrial researchers. A drawback is that there is usually not time to understand in detail what is behind the calculations. If too little is understood the students become frustrated because they feel they have no idea of what they have done. The use of computational methods in teaching is always a compromise between the realism of the problems to be tackled and the understanding of the methods to be used. The experience from more than 15 years of computational thermodynamics in physical metallurgy education at KTH will be discussed.

3:00 PM Invited
Tool Building for Computational Materials Science: *James A. Warren*[1]; [1]NIST, CTCMS, 100 Bureau Dr., Stop 8554, Gaithersburg, MD 20899-8554 USA

The Center for Theoretical and Computational Materials Science, part of NISTís Materials Science and Engineering Laboratory, has placed a strong focus on the development of computational tools. These tools allow researchers to quickly solve problems of scientific interest without being forced to ìreinvent the wheelî. The tools also form the core of the CTCMSís educational efforts. The educational benefits of these tools are at least twofold: (1) Students are involved in the development and implementation of these tools, thereby learning about theoretical and computational materials science; (2) dissemination of these tools acts as a powerful teaching device. As an added bonus, students often take there facility with these tools with them to industrial settings, where they can then be more effective, productive employees. In this presentation I will discuss some of our experiences in building software tools, promoting existing tools, and working with educators interested in harvesting some of the fruits of computational materials science.

3:30 PM Break

4:00 PM Invited
Educational Approach in Computational Materials Science: A Myopic View: *Duane D. Johnson*[1]; [1]University of Illinois Urbana-Champaign, Depts. of Matls. Sci. & Eng. & Physics, & the Frederick Seitz Matls. Rsrch. Lab., 1304 W. Green St., Urbana, IL 61801 USA

Two educational forums are discussed, an advanced-undergraduate and first-year-graduate course on ìAtomic-scale Simulationî and advanced topical Summer Schools in Computational Materials Science developed through the Materials Computation Center with integrated lectures from multiple disciplines/areas. Observations from these two local, but distinct, forums will be discussed with examples. The design and structure of each is highly dependent upon the courseís goals and studentís background. For large variation in material or backgrounds, we find that learning structures that include pre-developed tools/codes are successful by allowing students to focus on concepts and understanding via specific applications, rather than spending time writing and debugging codes. Educators can also highlight particular points and focus discussions. Such an approach facilitates ease of grading, avoiding as many codes, languages, and errors as students. Educational and research codes are available on the MCC Software Archive (www.mcc.uiuc.edu). Please contribute and use.

4:30 PM Invited
NSF Supported Computational Education Program at Penn State: *Zi-Kui Liu*[1]; Long-Qing Chen[1]; Karl E. Spear[1]; Carlee K. Pollard[1]; [1]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA

The three-year NSF supported computational education program at Penn State (July 2000-June 2003) aims to improve the student learning experience and educator teaching experience on two of the core components in the curriculum of materials science and engineering, i.e. Thermodynamics and Kinetics, emphasizing on graduate study. In this talk, our activities and experiences in the past two years and future plans will be presented and discussed.

5:00 PM Invited
Computer-Based Material and Process Selection: *Mike Ashby*[1]; [1]University of Cambridge, Eng. Dept., Trumpington St., Cambridge CB2 1PZ Great Britain

University courses on Engineering Materials develop the underlying knowledge of materials and their selection, but they do not always supply the tools that allow the knowledge to be used effectively. The CES4 EduPack is designed to meet this need. It provides a set of tools to enable Professors to develop successful courses and labs in Materials and Materials Processing. The software draws on three levels of data and methods, simple at the lowest level but progressing to a professional-level materials selection system for advanced students and post-graduate training. Level 1 contains limited data for 68 of the most commonly used materials drawn from all families (metals, polymers, ceramics, glasses, elastomers) and for 60 of the commonest processes; it allows students to explore materials without getting overwhelmed by detail. Level 2 retains this format, expanding the range of attributes for which data are listed and adding information on design, technical details and possible environmental concerns. The final 3rd level develops this further, providing a tool with which the student is already familiar, but now capable of accessing information for 3000 materials and 240 processes. The software is supported by a set of lectures in PowerPoint format, exercises and suggested projects. The talk will demonstrate the software and describe its use in teaching.

Computational Phase Transformations: Atomistic Modeling II - Diffusion, Elastic Property and Crystal Growth

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Monday PM Room: 11B
March 3, 2003 Location: San Diego Convention Center

Session Chairs: Mark D. Asta, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Chem. & Matls. Sci., Livermore, CA 94551 USA

2:00 PM Invited
Interstitial Diffusion in BCC Metals: *David J. Srolovitz*[1]; [1]Princeton University, Princeton Matls. Inst., 70 Prospect Ave., Princeton, NJ 08544-5211 USA

Self-interstitial migration in body centered cubic materials is central to understanding microstructure evolution under irradiation conditions. Since interatomic spacings around self-interstitials are uncommonly small, interatomic potentials fit to only crystalline data are not reliable for interstitials. We perform firt-principles calculations of interstitial structure, formation and migration energies in bcc Mo and V and use the results to fit new potentials. MD simulations of self-interstitials migration show that in V, the stable <111>-dumbell interstitial migrates in 1-d at low T. With increasing T, the dumbells rotate into other <111> directions but only show true 3-d diffusion at very high T. The apparent activation energy increases with temperature, even before the onset of significant rotation. This is attributed to self-interstitials reversing their trajectory at low T, uncorrelated jumps at intermediate temperature and multiple site hops at high T.

2:30 PM Invited
Vacancy and Self-Diffusion in Ordered Intermetallics: *Murray S. Daw*[1]; Michael J. Mills[2]; [1]Clemson University, Physics & Astron., Clemson, SC 29634-1911 USA; [2]Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Vacancy migration and self-diffusion influence many of the important high-temperature properties of intermetallics. Many of the fundamental features of such processes are yet to be determined. To investigate the fundamental mechanisms governing these processes, we have combined several recent theoretical advances. Using energetics from the Embedded Atom Method, saddlepoints are found using the Dimer Method. The energetic barriers are used in Kinetic Monte Carlo to simulate longer time scales than are attainable by ordinary Molecular Dynamics. Macroscopic diffusion rates are obtained using Relevant Rate Extraction. One major advantage of the techniques presented here is that a large range of possible mechanisms is explored automatically, including potentially quite complex sequences, thus requiring virtually no assumptions to be made as to mechanism. Applications include vacancy and self-diffusion in Ni3Al. Supported by NASA Grant No. NAG3-2675.

3:00 PM Invited
Solute-Vacancy Binding in Aluminum: *Christopher Wolverton*[1]; [1]Ford Motor Company, MD 3083/SRL, PO Box 2053, Dearborn, MI 48121-2053 USA

Solute-vacancy binding not only plays a crucial role in determining solute diffusion, but also can significantly affect the diffusion of a third species. The literature is replete with experimental reports of solute-vacancy binding in Al, taken from lattice parameter and density measurements, quenching experiments, and positron annihilation experiments. However, these experimental data do not provide a quantitatively consistent picture of binding energetics. Hence, we have performed a systematic study of solute-vacancy binding in Al for a wide range of solutes, using atomically-relaxed first-principles total energy calculations. We elucidate the crucial physical factors controlling an accurate description of binding energies. Using our results, we can explain the effects of small additions of Sn, Cd, In on Cu diffusion and GP zone suppression in Cu-containing Al alloys.

3:30 PM Break

3:50 PM Invited
Saddle-Point Structures and Elastic Instability: *John William Morris*[1]; David C. Clatterbuck[1]; [1]University of California, Matls. Sci., Evans Hall, Berkeley, CA 94720 USA

An elastically strained solid is always at least metastable. Given a kinetically plausible pathway, it will spontaneously transform into a sheared or broken replica of itself or into a new phase entirely. In that sense, plastic deformation is a structural phase transformation whose onset is governed by the usual criteria. It can be nucleated (and ordinarily is) but, failing that, must commence at the limit of stability of the elastic state. This thermodynamic instability sets the upper limit of strength. The present paper defines the limits of elastic stability (which are surprisingly subtle), shows how those limits reflect the symmetry of the strained lattice, reviews ab initio computations for a number of metals and compounds, and discusses the experimental situations in which they are known or expected to be important.

4:20 PM Invited
Simulations of the Growth of Aluminum Crystals Using Kinetic Monte Carlo Simulations Without Predefined Event Catalog: *Hannes Jonsson*[1]; Graeme Henkelman[1]; [1]University of Washington, Chem. Dept., 351700, Seattle, WA 98195-1700 USA

We have simulated the growth of the (100), (110) and (111) faces of aluminum crystals using a multiple time scale simulation algorithm that incorporates both the short time scale dynamics of deposition events and the intervening long time scale activated diffusion events. Processes involving concerted motion of multiple atoms are found to be important in determining the morphology of the growing surface. Not only is the activation energy of such multi-atom processes surprisingly low, but also the prefactor in the rate constant is found to be larger the more atoms are displaced. Result of simulations of copper crystal growth will be discussed for comparison.

4:50 PM
Influence of Dilute Alloying on the Vibrational Properties of Vanadium: *Olivier Delaire*[1]; Tabitha Swan-Wood[1]; Ryan Monson[1]; Brent Fultz[1]; [1]CALTECH, Matls. Sci., Keck Bldg., MC 138-78, Pasadena, CA 91125 USA

In this study, we investigate the effect of impurity alloying on the lattice dynamics and vibrational entropy of vanadium. Using inelastic neutron scattering, we have measured the phonon density of states (DOS) at room temperature for three random-substitutional alloys: V93.75Ni6.25, V93.75Pd6.25 and V93.75Pt6.25. Our data show an important stiffening of the phonon DOS upon alloying, resulting in a large negative entropy of alloying, comparable in magnitude to the configurational entropy in the alloy. In order to analyze these results, we have written a Born-von Karman lattice-dynamics simulation that allows us to invert the experimental phonon DOS and calculate atomic force-constants for the alloyed material. The phonon DOS curves we calculated are in very agreement with the experimental data. We analyze our results of force-constant calculations in terms of the difference in metallic radius between the vanadium and impurity atoms.

5:10 PM
Force Constants of Pd_3Fe Under High Pressure: *Alexander B. Papandrew*[1]; Alan F. Yue[1]; Peter D. Bogdanoff[1]; Itzhak Halevy[1]; Johnny Lin[1]; Brent T. Fultz[1]; Wolfgang Sturhahn[2]; Ercan E. Alp[2]; [1]Caltech, Eng. & Appl. Sci., MC 138-78, Pasadena, CA 91125 USA; [2]Argonne National Laboratory, Argonne, IL 60439 USA

The vibrational entropy of an alloy depends on interatomic forces, which depend on interatomic spacings. We studied the effect of pressure on the interatomic forces in $L1_2$ ordered Pd_3Fe. Inelastic nuclear resonant x-ray scattering was performed on samples in a diamond anvil cell at pressures up to 17 GPa. The phonon partial density of states (DOS) of Fe in the alloy were obtained from these spectra, and the interatomic force constants were obtained by finding the interatomic forces that best fit these results. With increasing pressure, the first nearest neighbor radial force constants increased, though the Pd-Fe force constant exhibited a proportionally greater increase than the Pd-Pd force constant. These results are consistent with a tighter atomic packing of Pd-Pd pairs.

Computational Phase Transformations: Microstructural Evolution and Interdiffusion

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)

Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Monday PM — Room: 4
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Perry Leo, University of Minnesota, Dept. of Aeros. Eng. & Mech., Minneapolis, MN 55455 USA; John A.L. ≈gren, Royal Institute of Technology, Dept. of Matls. Sci. & Eng., Stockholm 100 44 Sweden

2:00 PM Invited
Theory and Simulation of 3-D Crystal Growth: *John S. Lowengrub*[1]; [1]University of Minnesota, 206 Church St., VinH, Minneapolis, MN 55455 USA

In this talk, we consider the quasi-steady evolution of growing crystals in 3-D. A re-examination of this problem reveals that the Mullins-Sekerka instability may be suppressed by appropriately varying the undercooling (far-field temperature) in time. For example, in 3-D, by imposing the far-field temperature flux (rather than a temperature condition), a class of asymptotically self-similar, non-spherical growing crystals can be found. Simulations show that this class of solutions is robust with respect to perturbations and anisotropies and is well-predicted by solutions of the linearized equations. To simulate the problem numerically, we use a boundary element method with a fully adaptive surface triangulation. This enables us to simulate 3-D crystals stably and accurately well into the nonlinear regime. Simulations of both stable and unstable crystal growth will be presented. This work has important implications for shape control in processing applications. This work is joint with Dr. Vittorio Cristini (School of Math, Dept Chem. Eng. and Mat. Sci, U. Minn.)

2:30 PM
A Hybrid Monte Carlo/Elastic Continuum Model for Simulating Microstructural Evolution: *Pu Liu*[1]; Mark T. Lusk[1]; [1]Colorado School of Mines, Matls. Sci. Prog., Div. of Eng., Golden, CO 80401 USA

We present a method for coupling a Monte Carlo Ising model to a damped spring/mass assembly as a means of studying microstructural evolution in setting where localized lattice distortion influences the kinetics. Each of the two Ising states are given elastic constants and a stress free cell size, so that stresses develop at interfaces due to both transformation strain and a mismatch in elastic stiffness. A Metropolis algorithm is used in the Monte Carlo simulation, so that flip probability is based on the associated change in system energy. However, this energy change includes an elastic contribution that approximates the work required to distort the lattice in order to accommodate the flip. The spring/mass assembly is constructed on a square lattice, and a relation is given so that the spring constants and lattice spacing can be re-expressed as elastic constants in a continuum with at least tetragonal symmetry. The damped assembly is allowed to relax between Monte Carlo steps. Results are shown for two-dimensional domains with circular inclusions and also with planar boundaries. The rate of interface motion is compared with predictions that employ the results of a previous investigation which relates Monte Carlo kinetics to both phase-field and sharp-interface paradigms.

2:50 PM
Interaction and Stochastic Effects in Microstructural Evolution: *Kegang Wang*[1]; Martin Eden Glicksman[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng. Dept., 110 8th St., CII Rm. 4219, Troy, NY 12180 USA

Mesoscale simulation of phase coarsening remains as an active and important topic in materials science. Classical theory, as formulated by Lifshitz and Slyozov and by Wagner (LSW), excludes all interactions among coarsening particles and ignores local environmental information. LSW coarsening rate and particle size distribution, however, differ markedly from experimental results. To simulate the dynamics of phase coarsening we formulated and solved multiparticle diffusion equations, including both interactions among particles and the influence of local environments, or ìlocales.î Interactions lead to fluctuations in the growth rate of individual particles that can be measured in our simulations. These simulations also reveal that the growth rates of individual particles deviate nonlinearly from the LSW predictions with increasing volume fraction. Intermediate configurations of a coarsening microstructure can also be visualized in our simulation. Finally, stochastic effects will be described using ìmultiplicative noise,î and recent progress in simulating microstructure evolution will be discussed.

3:10 PM
Ferrite Growth Kinetics Model and Solute Drag Effect: *Fateh Fazeli*[1]; Matthias Militzer[1]; [1]University of British Columbia, The Ctr. for Metallurgl. Process Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

A mixed-mode (MM) model for the kinetics of ferrite growth has been developed in which both carbon diffusion and interface reaction are considered. Adopting the Purdy-Brechet approach, the effect of solute drag is characterized and then incorporated explicitly into the model by diminishing the amount of available driving force at the austenite/ferrite interface. Solute drag parameters are employed as adjustable quantities. However, due to the lack of information on the intrinsic interface mobility the solute drag terms depend on the choice for the mobility. Alternatively, the solute drag effect can also be implemented into a diffusional model (DM) for ferrite growth by means of changing the interfacial carbon concentration. This eliminates to assume an intrinsic interface mobility. The predictions of these two modeling approaches, i.e. MM and DM, have been validated with continuous cooling transformation data of various steel chemistries. Further, the physical relevance of the resulting solute drag parameters are discussed and compared.

3:30 PM Break

3:50 PM Invited
Computational Modeling of Internal Oxidation: Yali Li[1]; *J. E. Morral*[1]; Kaisheng Wu[2]; Yunzhi Wang[2]; [1]University of Connecticut, Metall. & Matls. Eng., 97 N. Eagleville Rd., U-136, Storrs, CT 06269-3136 USA; [2]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Internal oxidation is a well-known phenomenon. It occurs in alloys when an element from the gas phase diffuses into an alloy and oxidizes the solute to form oxide precipitates below the surface. In the current work internal oxidation is being modeled in three ways: with error functions, with DICTRA finite difference software, and with the phase field method. The results will be compared with experimental data and with predictions of classical theories of internal oxidation.

4:20 PM
A Computational Program for Interdiffusion Fluxes, Interdiffusion Coefficients and Diffusion Paths for Single Phase Multicomponent Diffusion Couples: *Mysore A. Dayananda*[1]; Ramdas Ram-Mohan[2]; J. Moussa[3]; J. Gagnon[3]; [1]Purdue University, Sch. of Matls. Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907 USA; [2]Worcester Polytechnic Institute, Dept. of Physics, 100 Institute Rd., Worcester, MA 01609 USA; [3]Quantum Semiconductor Algorithms, Inc., 5 Hawthorne Cir., Northborough, MA 01532 USA

A program identified as MultiDiflux has been developed for the analysis of experimental data on concentration profiles of single-phase diffusion couples in multicomponent systems for the determination of interdiffusion fluxes of all components over the entire diffusion zone and interdiffusion coefficients over selected ranges of compositions along the diffusion paths. The program is based on the integration procedures, developed by Dayananda and coworkers, for the calculation of interdiffusion fluxes directly from the concentration profiles and for the determination of average main and cross interdiffusion coefficients over selected composition ranges from the calculated interdiffusion fluxes. The details of the analysis are discussed and the use of the program is illustrated with experimental concentration profiles of diffusion couples in selected ternary systems.

4:40 PM Invited
Zero-Flux Plane Kinetics in Multicomponent Solids: *Martin E. Glicksman*[1]; A. Lupulescu[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng. Dept., 110 8th St. (CII-9111), Troy, NY 12180-3590 USA

Multicomponent diffusion can lead to zero flux planes (ZFPís), where an individual component flux vanishes. The conditions for ZFPís are well understood through the works of Morral, et al. We analyzed diffusion kinetics near ZFPís in single phase Cr-Al-Ni alloys near the composition 10 at.%-Cr, 10at.%-Al, 80 at.%-Ni. The presence of a stationary ZFP requires that mixing of the blocked component occurs via coupled diffusion waves. In thick couples these waves spread symmetrically away from the Matano plane. The wave pair consists of a ìdepletionî wave that reduces the blocked component in the component-rich alloy, and a conjugate ìrepletionî wave that increases it in the adjacent component-poor alloy. Each wave establishes equilibrium concentration unilaterally - a curious circumstance with practical implications for the design of stable alloy coatings. Specific multicomponent behaviors, such as reduced total atomic transport, are found to be associated with the localization of multiple ZFPís.

5:10 PM
Coupled Microscopic-Mesoscopic Simulation of Nitriding of Fe-Cr-C Alloy: Lars Hˆglund[1]; *Henrik Larsson*[1]; John A.L. ≈gren[1]; [1]KTH, Dept. Matls. Sci. & Eng., Stockholm SE-100 44 Sweden

A new model for simulation of internal nitriding in multicomponent steels has been developed and implemented on computer. The new model allows for parallel simulation of the evolution over the long range (i.e. on the order of hundreds of microns) concentration and phase fraction profiles and the short range (i.e. on the order of microns) profiles as well as the local dissolution and precipitation reactions. The secondary particles, i.e. the carbides and nitrides, act as sources or sinks for carbon, nitrogen and chromium. Nitriding of an Fe-Cr-C alloy was simulated by means of the new model and the results are compared with experimental data.

Defects and Deformation of Crystalline Solids - in Honor of Dr. Man H. Yoo - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
Program Organizers: Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Department, Houghton, MI 49931-1200 USA; Sean R. Agnew, University of Virginia, Materials Science and Engineering Department, Charlottesville, VA 22904-4745 USA; K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA

Monday PM — Room: 17A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: James Li, University of Rochester, Dept. of Mechl. Eng., Rochester, NY 14627 USA; William Nix, Stanford University, Matls. Sci. & Eng., Stanford, CA 94305-2205 USA

2:00 PM Invited
Dislocation Organisation in TiAl Alloys: *Patrick Veyssiere*[1]; [1]CNRS-ONERA, LEM, BP 72, Chatillon, Cedex 92322 France

This talk addresses two cases of dislocation spontaneous organization in TiAl alloys. One is the widespread occurrence or irregular dipolar and multipolar dislocation tangles and interspersed prismatic loops especially in single-phase gamma TiAl strained in single slip at room temperature by the so-called ordinary dislocations (b = 1/2<110]). Mechanisms will be presented to interpret the following properties: - when produced by conservative processes, prismatic loops are in general organized in staircase strings where the end of a given loop is aligned with the beginning of the next loop in the screw direction. Strings interact with mobile dislocations forming a variety of configurations including tangles and hairpin features. - in loose tangles, strings can be totally or partially annihilated by a single impacting dislocation. In dense walls, loop refinement should in addition take place by glide of loops of opposite kinds. The other case of dislocation organisation deals with planar intralamellar networks. These networks consist in rectangular cross-grids of screw dislocations with 1/2<110] and 1/2<112] Burgers vector. They are parallel to the lamellae and they are formed entirely by glide in samples strained in the soft load orientation while primary slip takes place by <011]{111} dislocations.

2:25 PM Invited
Dynamic Atomistic Simulations of Low Angle Dislocation Boundaries in FCC Metals: *Frank J. Cherne*[1]; Rodney J. McCabe[1]; Michael I. Baskes[1]; [1]Los Alamos National Laboratory, MST-8, Struct. & Prop. Relations, PO Box 1663, MS G755, Los Alamos, NM 87545 USA

Deformation microstructures of medium to high stacking fault FCC metals (Cu, Ni, Al) consist of cell blocks separated by low angle dislocation boundaries. These boundaries run roughly parallel to the plane(s) of maximum shear stress with boundary spacing decreasing and boundary misorientation increasing with strain. These boundaries play a significant although not well understood role in work hardening and other plastic behavior of these metals. The present atomistic study, utilizing both embedded atom method and modified embedded atom method potentials, deals with the interaction of individual dislocations with low angle dislocation tilt boundaries. The interaction is studied as a function of grain orientation and misorientation across the boundary. Boundaries were assumed to be made up of the lowest energy (lowest dislocation density) configuration of dislocations from the three most highly stressed slip systems. Comparisons are made to experimental studies.

2:50 PM
Investigation of the Deformation Behavior of Rocks by Neutron Diffraction: *Sven C. Vogel*[1]; Mark A.M. Bourke[1]; Bj□rn Clausen[2]; Timothy W. Darling[1]; James A. TenCate[1]; [1]Los Alamos National Laboratory, MST-8, MS H805, PO Box 1663, Los Alamos, NM 87545 USA; [2]California Institute of Technology, Matls. Sci., MC 138-78, 1200 E. California Blvd., Pasadena, CA 91125 USA

We present results of a neutron diffraction study of the deformation behavior of rocks, namely, sandstone (quartz, SiO_2) and marble (calcite, $CaCO_3$). Although rocks have been studied by macroscopic mechanical experiments for a very long time, the microscopic behavior associated with a particular macroscopic observation is in many cases not well understood. Specifically, we attempt to gain understanding in the microscopic origin of the non-linearity and hysteresis (end-point-memory) evident in macroscopic stress-strain curves of some rocks. Neutrons, with there deep penetration into most materials, are one of a few probes that allow to gain understanding of the mechanisms on the atomic or microscopic level that govern the macroscopic behavior and properties of such complex materials as rocks. With the new neutron time-of-flight powder diffractometer SMARTS we are able to apply uni-axial load to a specimen and measure the lattice strain response of specific grain orientations with respect to the loading axis.

3:10 PM Invited
Plasticity and Deformation Microstructure of Some Intermetallics: *Gernot Kostorz*[1]; Helge Heinrich[1]; Markus Wollgarten[1]; [1]ETH Zurich, Angewandte Physik, Zurich CH 8093 Switzerland

Results on the plastic deformation and the deformation microstructure (obtained from transmission electron microscopy) of three intermetallic materials will be presented and analyzed. (i) Lamellar two-phase $TiAl/Ti_3Al$ samples (polysynthetically twinned single crystals) were prepared and tested in uniaxial compression. For different angles between the lamellae and the stress axis, strain-rate changes and stress relaxation along the deformation path indicate a very large activation volume between 500 K and 800 K, while an almost constant yield stress and strong strain localization are found in the same temperature range. At higher temperatures, recovery processes induce an increasing strain-rate sensitivity. The activation volume does not depend significantly on the lamellar orientation and decreases to 0.12 nm^3 at 1300 K. (ii) Small single crystals of RuAl (CsCl structure) were prepared and deformed in uniaxial compression at temperatures ranging from 300 to 1200 K. The results of stress relaxation and strain-rate changes indicate strong variations of the activation parameters with increasing strain, especially at low and high temperatures. (iii) For RuSi near the stoichiometric composition, two modifications are reported; CsCl and FeSi. Like RuAl, RuSi in the CsCl modification is metallic. The plastic deformation shows less ductility for RuSi than for RuAl.

3:35 PM Break

3:55 PM Invited
Atomistic Modeling of Dislocation Motion and Twinning in TiAl: Effects of Directional Bonding: *V. Vitek*[1]; R. Porizek[1]; S. Znam[1]; D. Nguyen-Manh[2]; D. G. Pettifor[2]; [1]University of Pennsylvania, Dept.

of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA; [2]Oxford University, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

Recently we constructed Bond-Order Potentials for TiAl that are a real space description of atomic interactions based on the tight-binding method. These potentials reflect correctly that bonding in Ti-Al alloys is not purely metallic but the covalent component of bonding is significant. Using these potentials, we study the structure, glide and, in general, the effect of applied stresses on the core of the ordinary 1/2<110] dislocation, <110] superdislocation and 1/2<112] dislocation in L10 TiAl. For 1/2<110] and <110] dislocations we investigate the orientation dependence of the Peierls and thus possible break-down of the Schmid law. The 1/2<112] dislocation may serve as a nucleus for twinning and development of a macroscopic twin from such nucleus will be discussed. Earlier atomistic studies were made using central-force potentials and comparison with these studies allows us to identify effects related to the non-central bonding, which is one of the principal goals of this study.

4:20 PM

Experimental Characterization of Dislocation Core Structures with Transmission Electron Microscopy: *Kevin J. Hemker*[1]; Mingwei Chen[1]; [1]Johns Hopkins University, Dept. Mechl. Eng., Baltimore, MD 21218 USA

The development and use of theoretical and computationally intensive techniques provide unique opportunities for understanding and modeling atomic level processes, and holds great promise for enhancing our understanding of the processing-structure-property-performance relations that govern alloy design and materials behavior in general. Recent advances in theoretical modeling hold great promise, but they are currently inhibited by the limited amount of experimental data that is available to verify and test their predictions. This paper will describe efforts to provide quantitative measures of dislocation core structures in pure metals and intermetallic alloys. A combined weak-beam TEM and HREM study of fcc metals, that has resulted in the quantification of extremely small dislocation dissociation distances for single-crystalline gold and iridium, will be outlined. This presentation will also describe efforts to measure atomic column positions in the near core region of a/2<111> screw dislocations in bcc meta ls and to develop a TEM EELS based methodology that will allow us to measure the electronic structure of <001> oriented dislocation cores in NiAl.

4:40 PM Invited

Incomplete Kear-Wilsdorf Locks and Yield Stress Anomaly of Ni_3Al: *Daniel Caillard*[1]; [1]CEMES-CNRS, 29 rue Jeanne Marvig, BP 4347, Toulouse F-31055 France

The yield stress anomaly of Ni_3Al is due to the thermally activated locking of mobile dislocations by cross slip onto the cube plane. This process is enhanced by the difference in the antiphase boudary (APB) energies in cube and octahedral planes, and by the torque determined by Man Yoo. A comparison of the main experimental results of different groups obtained by either mechanical tests, post mortem or in situ electron microscopy, shows that the strength of the locks can be measured, and their exact geometry can be determined. They are ìincompleteî Kear-Wilsdorf locks formed by short-range cross slip over an interatomic distance. This behavior is similar to that proposed earlier by Pope Paidar and Vitek, except that cross-slip extends over the whole dislocation length, and unlocking takes place by the reverse cross-slip process. The roles of the different fault energies (APBs and complex staking fault) are discussed quantitatively.

5:05 PM

The Effects of Microstructure and Loading Orientation on the Evolution of Slip in Single Crystal Superalloys: *Fereshteh Ebrahimi*[1]; Hongqi Li[1]; Xavier Foltete[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, Gainesville, FL 32611 USA

In this study the evolution of slip bands as a function of strain has been characterized for various tensile loading orientations in two superalloy single crystals. Dog-bone shaped samples were loaded along the <100>, <110>, and <111> directions at room temperature. The development of slip bands was investigated on both the face and the side of each sample. The main difference between the two superalloys was the presence of interdendritic eutectic structure and porosity in the microstructure of one of them. The results of this study indicate that the presence of these structures significantly affects the evolution of the slip bands by creating triaxial stress states and inducing macroscopic shear bands between these structures. Cross-slip was enhanced in the superalloy with the interdentritic structure and resulted in a wavy slip behavior. In this paper the effect of microstructure on the propensity to strain localization in single crystal superalloys are discussed.

Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong: High Strain Rate Deformation and Shear Localization

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Marc Andre Meyers, University of California-San Diego, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; George T. Gray III, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA; Kenneth S. Vecchio, UC San Diego, Dept of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA

Monday PM Room: 16B
March 3, 2003 Location: San Diego Convention Center

Session Chair: G. T. Gray, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

2:00 PM

Constitutive Framework for Solution Hardened Metals: *U. Fred Kocks*[1]; [1]Retired, PO Box 89, Placerville, CO 81430 USA

Many solution hardened alloys exhibit a regime of dynamic strain aging (DSA), which has long been linked, in a qualitative way, with the phenomena of (at least partial) negative rate sensitivity and an inverse (ìanomalousî) temperature dependence. The former has been treated in a quantitative form, together with observations on static strain aging; the effects are proportional to the flow stress reached in previous strain hardening; jerky flow (and PLC bands) are observed only in the regime where the total rate sensitivity is negative. It is also well-known, in a qualitative way, that the anomalous temperature dependence is much stronger for strain-hardened than for annealed materials. Above the ìhumpî, the behavior is dominated by viscous drift. In the low-temperature limit, on the other hand, dislocation activity is controlled by bulge nucleation. ó This paper is an attempt to integrate these various physical mechanisms into a single phenomenological framework and specify experiments needed, for any partic ular material, to determine the quantitative relations. These are to complement the master curves for rate-dependent plasticity and evolution established for pure materials.

2:30 PM

Dynamic Deformation Analysis of the Cellular Solids and Structures with Consideration of a Unit-Cell Structure: *Tatsuhiko Aizawa*[1]; Hidetaka Kanahashi[2]; Shigeyuki Tamura[3]; Masahide Katayama[3]; [1]RCAST, The University of Tokyo, High Perf. Matl., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8904 Japan; [2]The University of Tokyo, Dept. of Metall., Grad. Sch. of Eng., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8904 Japan; [3]CRC Solutions Corporation, 2-7-5 Minamisuna, Koto-ku, Tokyo 136-8581 Japan

The cellular solid and structures have regularized or semi-regularized pattern in their inside, completely different from the foam materials. In the design of open or closed foams, since their static and dynamic response is dependent on the ductility of columns, the conventional or classic approach by Ashby et al. can be used with success. When the structural connectivity is regularized to obtain smooth stress transfer in materials, new theoretical model is necessary to describe the deformation process in relatively wide dynamic range. The present study is the first step where the two/three dimensional finite difference code, Autodyn, is applied to this dynamic response simulation of a unit cell. As a unit cell, polyhedral and quasi-polyhedral connectivity in the three dimensional case, is modeled by a structured assembly of beam elements. From the calculated results, the effect of regularization in spatial connectivity in the open cell structure on the dynamic stress-strain curve is discussed with comparison to the experimentally measured dynamic response.

2:45 PM

AFM Study of Sub-Lattice High Steps and Shear Band Melting in Impacted Gold and Shocked RDX: *Jagadish Sharma*[1]; Scott M. Hoover[1]; Charles S. Coffey[2]; Wayne L. Elban[3]; [1]NSWC, Carderock Lab., 9500 MacArthur Blvd., Code 645, W. Bethesda, MD 20817 USA; [2]Naval Surface Warfare Center, Indian Head Div., Indian Head, MD 20903 USA; [3]Loyola College, Dept. of Eng., Baltimore, MD 21210 USA

The plastic deformation of gold has been studied using solidified 2-mm globules sandwiched between either mica sheets or steel shim stock and subjected to 10 and 100 cm impacts in 10-kg drop-weight machine. The impacted surfaces were examined with AFM and STM. Comparison is made with RDX, a molecular explosive crystal. Hexagonal atom arrays were found in gold at low energy (10 cm) impacts and attributed to deformation occurring on well-known {111} slip planes: however, 100 cm impacts produced square arrays, indicating that slip is on {100} planes. Prominent surface bending and roughness reflected high number of slip systems and unrestricted slip step heights. Sub-atomic steps were observed in gold as were sub-molecular step heights in shocked RDX. Melting and exudation from shear bands were observed for both. Model considerations will be presented for the deformational heating/melting in the nanometric scale.

3:00 PM
Dislocation Dynamics and Length-Scale in Metal Plasticity: Physics-Based Modeling and Experimental Verification: *Sia Nemat-Nasser*[1]; [1]University of California-San Deigo, CEAM, 4207 EBU1, 9500 Gilman Dr., La Jolla, CA 92093-0416 USA

Plastic deformation of a broad class of metals occurs by the motion of dislocations. The structure of dislocations, their density and distribution, as well as their interaction with each other and with the solute atoms and other defects lie at the foundation of crystal plasticity. Collectively, all these affect the motion of dislocations and, hence, the resulting plastic flow. The collective resistance to the dislocation motion defines the flow stress of the material at the continuum scale. Based on the results of systematic experiments on numerous commercially pure metals, the rate- and temperature-dependence of the flow-stress has been modeled by the author. This modeling naturally involves length-scales that characterize the underpinning dislocation activities. The introduction of the density of dislocations and its evolution, into the constitutive relations appears to be sufficient to account for essentially all existing experimental results that otherwise cannot be modeled by the classical plasticity theories. Rate- and temperature-dependence of the material response are accounted for as an integral part of the kinematics and kinetics of the dislocation motion. The general approach applies to both single crystals and polycrystals. With slight modification, it also applies to the phenomenon of dynamic strain-aging (at high and low strain rates) that stems from the interaction between dislocations and solute atoms.

3:15 PM
Effect of High Strain Rates on the Yield Stress of 4340 Steel: *Nabil Bassim*[1]; [1]University of Manitoba, Fac. of Eng., Winnipeg, Manitoba R3T 5V6 Canada

The effect of high strain rates on the yielding behavior of 4340 steel was investigated experimentally using a torsional Split Hopkinson Bar. The strain rates varied from 100 to 1000 s-1. The yield stress was calculated from the response of the system to the insident and reflected waves. Significant variations are observed when the yield stress was plotted versus the strain rate, which can be defined into three distinct regions A, B and C. In region A, at low strain rates, a small yield drop is observed with both the upper and lower yield stress stress increase with the strain rate. In region B, corresponding to intermediate values of strain rates, the yield stress drops significantly, with the lower yield stress reaching a minimum value. In region C, corresponding to high strain rates, there is no yield drop and continuous yielding is observed. Also, It is observed that the upper yield stress is almost independent of strain rate in regions B and C. A model is proposed to explain these observations. The variation of the initial drag stress, dynamic drag stress and kinetic friction with strain rate describe the dynamic interaction between dislocations and atmospheres. The model explains the occurrence of the three regions observed experimentally.

3:30 PM
Shear Localization in Metals: Self Organization and Microstructural Evolution: *Marc Andre Meyers*[1]; Qing Xue[2]; V. F. Nesterenko[1]; Y. B. Xu[3]; [1]University of California-San Diego, Dept. of MAE, MC 0411, La Jolla, CA 92093-0411 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [3]Chinese Academy of Sciences, Inst. of Metals Rsrch., Shenyang 110016 China

The behavior of adiabatic shear bands generated by the collapse of a thick-walled cylinder exhibits a self-organization character. Experiments were carried out in Ti, Ti-6Al-4V, and AISI 304 SS at controlled and prescribed global strains. The spacing of shear bands varies with their evolution. At the initiation stage, the spacing is smaller. As the bands grow, their stress fields interact, leading to the selective growth of some and arrest of others. This behavior is not captured by the existing one-dimensional theories (Grady-Kipp, Wright-Ockendon-Molinari) and a two-dimensional description is proposed, starting with a distribution of sites with different potencies and evolving with the interaction of the stress fields. This description is successfully applied to the three materials above and the significant differences in response are explained. The microstructural evolution within the shear bands is characterized by transmission electron microscopy and a variety of microstructures is observed, evolving with the imposed plastic shear strain and with the temperature excursion. Recovery, recrystallization, and amorphization are observed. Support: US ARO-MURI Program.

3:45 PM Break

4:00 PM
Plastic Instability of Al-Mg Alloy 5052 During Stress Rate Change Test: *Chen-Ming Kuo*[1]; Chi-Ho Tso[2]; [1]I-Shou University, Dept. of Mechl. Eng., 1, Sec. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan; [2]I-Shou University, Dept. of Matls. Sci. & Eng., 1, Sec. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

Plastic instability is observed during stress rate change test of aluminum-magnesium alloy 5052 at room temperature. In the stress rate change experiments, strain retardation and plastic instability are observed, that is, although the applied stress rate changes, plastic strain is insignificant until the plastic instability occurs. If there is no unstable phenomenon, plastic strain rate would practically have no changes. The occurrence of plastic instability is related to the applied stress rate and retention time before the applied stress rate change. The bigger of applied stress rate, the more significant instability of plastic deformation is, also at the same stress rate, the longer of retention time, the higher of true stress value of initial instability is. By the argument of dynamic strain ageing effect, plastic instability could be justified as interactions between solid solution element, magnesium, and dislocations.

4:15 PM
Shear Bands Patterning and Ballistic Performance of Ti-6Al-4V Homogeneous and High-Gradient Targets: *Vitali F. Nesterenko*[1]; Yabei Gu[1]; Sastry S. Indrakanti[1]; Singh Brar[2]; [1]University of California-San Diego, MAE, La Jolla, CA 92037 USA; [2]University of Dayton Research Institute, Dayton, OH 45469 USA

This presentation is related to the seminal contributions of Ronald Armstrong toward shear instability, structure/property relations and ballistic performance of materials, particularly Ti-6Al-4V alloy. We analyze high strain rate properties, postrcritical behavior and ballistic performance of Hot Isostatically Pressed (HIPed) Ti-6Al-4V material in straight comparison with baseline samples of the same alloy (MIL-T-9047G). We will present data on constitutive equations (Hopkinson bar tests) and data characterizing postcritical behavior of materials - results of hat shape specimen test (forced shear flow in single shear band) and thick walled cylinder (TWC) test (patterning of multiple shear bands). The results of penetration test (tungsten rod, velocity 886-960 m/s, diameter D=4.98 mm, L/D=10) with solid and porous composite samples of Ti-6Al-4V alloy with different microstructures (Widmanstatten pattern and equiaxed) will be reported. Qualitative difference in ballistic performance of baseline alloy and HIPed materials is attributed to a difference in shear bands patterning on the post-critical stage of material flow during penetration process - bulk distributed complex shear bands pattern in HIPed material in comparison to a regular pattern of widely spaced shear bands in baseline material. This work is supported by Army Research Office under MURI program No. DAAH004-96-1-0376.

4:30 PM
Influence of Shock Prestraining on the Formation of Shear Localization in 304 Stainless Steel: *Qing Xue*[1]; George T. Gray[1]; Shuh-Rong Chen[1]; [1]Los Alamos National Laboratory, Matl. Sci. & Tech. Div., MST-8, MS G755, Los Alamos, NM 87545 USA

Adiabatic shear localization was investigated in both annealed and shock pre-strained 304 stainless steel. The forced shear technique with hat-shaped specimens was utilized under high-strain-rate loading using a compression Hopkinson bar. The shock pre-strained specimens were seen to exhibit narrower bands with much less shear in the matrix adjacent to the shear band, while those of the annealed steel displayed a gradient increase of shear deformation with increasing proximity to the band. The mechanical responses of the shock pre-strained steel at high strain rate shearing indicate that they arrived at the instable point at much smaller plastic strain and the shear stress continuously drops after the initiation of shear localization, although they have a higher yield stress. The experimental results demonstrate that loading path dependency exerts a strong influence on shear-band formation. The comparison of the microstructures evolved in these two materials shows that the shock pre-strained steel displays a large amount of deformation twins from prestraining and these accelerate shear localization, while localization in the annealed steel develops more slowly.

The mechanisms controlling strain localization for the shock prestrained 304 SS are discussed.

4:45 PM
Microstructural Features of the Localized Shear Bands in Ti-6Al-4V Alloy: *Y. B. Xu*[1]; [1]Chinese Academy of Sciences, Shenyang Natl. Lab. for Matls. Sci., Inst. of Metal Rsrch., Shenyang, Liaoning 110016 China

This paper presents the microstructural features observed in localized shear bands in Ti-6Al-4V alloy induced during collapse testing of the thick-walled cylinder speciments. Observations by TEM and HREM show that a number of microstructure features occur in the bands during explosion collapse, including phase transformation, recovery/recrystallization. These features are discussed and interpreted in terms of the mechanisms of deformation.

5:00 PM
Current Status of the Theory of Adiabatic Shear Bands: *T. W. Wright*[1]; [1]US Army Research Laboratory, WMRD, Aberdeen Proving Ground, MD 21005 USA

Over the last two decades the theory of adiabatic shear bands has developed from the continuum point of view so that today a great deal is known about timing or critical strain, structure, spacing, speed of propagation, etc. Scaling laws have also been developed to describe virtually all of these features so that an experimentalist can attempt to optimize the design of a particular experiment in a rational way. The paper reviews these developments and describes a recent application to a damage model for use in large scale impact calculations.

5:15 PM
Microstructural Evolution of Shear Bands Induced During Dynamic Deformation of TI-6AL-4V: *Y. B. Xu*[1]; Q. Xue[2]; Marc A. Meyers[3]; [1]Chinese Academy of Sciences, Shenyang Natl. Lab. for Matls. Sci., Inst. of Metal Rsrch., Shenyang 110016 China; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [3]University of California-San Diego, Dept. of MAE, La Jolla, CA 92093-0411 USA

The microstructural features of the localized shear bands generated under high-strain-rate in a Ti-6Al-4V alloy during the collapse of a thick-walled cylinder were observed by SEM, TEM and TREM. The main results observed in the band are described and given as follows: (1) Twinning is a major mode of deformation in this alloy under the imposed high-strain rate deformation; (2) Dynamic recrystallized grains with equiaxed shape and distortion-free appearance are present in the bands. (3) The phase (Ti3Al) precipitated from the Ti parent matrix during dynamic deformation, and there are a certain orientation relationship between the Ti3Al and parent phase. Research supported by US Army Research Office MURI.

General Abstracts: Nonferrous Alloys and Other Materials

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Monday PM — Room: 19
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Jim Foley, Iowa State University, Ames Lab., Ames, IA 50011-3020 USA; Patrick R. Taylor, Colorado School of Mines, Metallurgl. & Matls. Eng., Golden, CO 80401 USA

2:00 PM
The Influence of Microstructural Stability on Mechanical Properties in Nickel-Base Single Crystal Superalloys: *Laura J. Rowland*[1]; Q. Feng[1]; T. M. Pollock[1]; [1]University of Michigan, Dept. of Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA

The mechanical properties of single crystal nickel-base superalloys are affected by the microstructural stability during high temperature exposure. It is well known that the misfit between the γ-γí provides a driving force for rafting under an applied stress at high temperatures, thus an alloy with a near-zero misfit is expected to have a greater degree of microstructural stability. Specimens of two commercial alloys containing different levels of Re and cuboidal γí precipitates and specimens of an experimental high Ru alloy with near-zero misfit were crept in tension to 1% strain at 950°C in vacuum and then tensile tested at room temperature. While the post-crept commercial alloys show lower post-crept yield strengths and higher ductility, the experimental alloy exhibited higher yield strength relative to the material without prior creep deformation. Scanning electron microscopy revealed rafting in commercial alloys, with rafts oriented normal to the axis of the applied tension. Interestingly, no evidence of rafting was found in the experimental Ru-containing alloy. Possible reasons for this behavior will be discussed.

2:20 PM
The Effect of Stress Level and Grain Size on the Ambient Temperature Creep of Alpha Ti-1.6%V Alloy: Ameet K. Aiyangar[1]; Sreeramamurthy Ankem[1]; *Brett W. Neuberger*[1]; [1]University of Maryland, Dept. of Matls. & Nucl. Eng., College Park, MD 20742-2115 USA

The effect of stress level and grain size were studied on the ambient temperature creep deformation behavior of alpha Ti-1.6%V alloy. It was found that for a given grain size, the amount of creep strain decreases with a decrease in stress level, where the stress level was varied from 75 to 95% of yield stress. Similarly, it was found that for a given stress level, a decrease in grain size results in a decrease of the amount of creep strain, with grain sizes studied varying from 38 to 150 micrometers. In all the cases, the decrease in creep strain was associated with a decrease in the phenomenon of time dependent twinning. The time dependent twinning along with slip was found to be the major deformation mechanisms in this alloy. Details of these studies will be presented. This work is being supported by the National Science Foundation under Grant Number DMR-0102320.

2:40 PM
High-Temperature Corrosion of Iron Alumindes in O2/2%Cl2/Ar Environments: *Gilsoo Han*[1]; *W. D. Cho*[1]; [1]University of Utah, Dept. of Metallurgl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

The high-temperature corrosion of iron aluminide (Fe3Al) in environments containing 2% chlorine, various oxygen contents and argon at 700°C has been investigated using themogravimetric method. Depending on O2/Cl2 ratio, two kinetic modes were observed: weight loss and gain. The weight loss mode consists of two distinct stages: initial slow weight loss and the subsequent fast linear weight loss. The rate of weight loss is decreased with oxygen concentration in the mixtures. The trend for repassivation or defect-healing by alumina-forming active oxidation is increased as oxygen concentration in the gas mixture increases. The corrosion products were analyzed using various analytical tools including SEM, XRD and EDX. Based on kinetic behavior and microstructure, the corrosion mechanisms are discussed.

3:00 PM
The Effect of Static and Dynamic Precipitation of AlMn-Dispersoids on the Recrystallization Behavior in a Cold Rolled AA3103-Alloy: *Stian Tangen*[1]; Hans Bjerkaas[1]; Knut Sj□lstad[1]; Erik Nes[1]; [1]Norwegian University of Science and Technology, Alfred Getz vei 2b, Trondheim 7491 Norway

During thermomechanical processing or annealing of a deformed and supersaturated material, recovery as well as recrystallization may be influenced by static dispersoids resulting from precipitation during homogenisation heat treatment or by dynamic concurrent precipitation, which in turn is strongly affected by the heterogeneity of the deformed microstructure. The deformation and annealing behavior in a cold rolled AA3103-alloy has been studied. In order to separate between static and dynamic precipitation, the material has been processed in two different ways before the final cold rolling to a true strain of 3. One part of the material was homogenized related to industrial standards, which gave a supersaturation of Mn in solid solution. The second part of the material was homogenized, cold rolled to a prestrain of 0.5, and then annealed at medium temperature (300°C-350°C) in 106s in order to precipitate AlMn-dispersoids on the deformation microstructure. The material was then finally cold rolled again to a total strain of 3. Following cold deformation the materials were isothermally annealed in order to recrystallize at different temperatures. The softening reactions during annealing were followed by hardness and electrical conductivity measurements. Recrystallized grain sizes have been measured both by the use of the Optical Light Microscope and the EBSD orientation mapping technique in SEM (OIM). The EBSD technique has also been used to study the texture development during the deformation and annealing of the materials. The particle size distributions after deformation were investigated with a FEGSEM equipped with an image analysis system, and the dispersoids and deformation structure were studied by TEM.

3:20 PM
Precipitate Nucleation at Vacancy Condensation Loops in Al-Cu-Mg Alloys: *Graham B. Winkelman*[1]; Krishnamurthy Raviprasad[1]; Barry C. Muddle[1]; [1]Monash University, Sch. of Physics & Matls. Eng., PO Box 69M, Victoria 3800 Australia

It could be argued that the topic of heterogeneous nucleation in aluminium alloys has not received a degree of attention commensurate with its technological significance. Conventional processing of Al-Cu-Mg alloys leads to the formation of vacancy condensation (dislocation) loops, where the loop habit plane and Burgers vector display a correlation with alloy composition (and hence, stacking fault energy). Through the application of conventional and high-resolution TEM to a range of artificially aged Al-Cu-Mg alloys, it is demonstrated here that nucleation of S-phase and q¢ precipitates occurs at discrete sites on the various loops. The observations can be rationalised as a function of dislocation line direction. Furthermore, the loop Burgers vector and dislocation line direction is observed to play a significant role in S-phase variant selection and morphology. The observations will be discussed in the context of an analysis of mechanisms of strain accommodation in the environment of individual dislocation loops.

3:40 PM Break

3:50 PM
Effect of Ta Content on Phase Constitution and Heat Treatment Behavior of Ti-Ta Alloys Quenched from a Temperature Within Beta Single-Phase Region: *Masahiko Ikeda*[1]; Yuichiro Nakamura[2]; Shin-ya Komatsu[1]; [1]Kansai University, Fac. of Eng., Dept. of Matls. Sci. & Eng., 3-3-35, Yamate-cho, Suita, Osaka 564-8680 Japan; [2]Kansai University, 3-3-35, Yamate-cho, Suita, Osaka 564-8680 Japan

As fundamental study for development of a new titanium alloy for medical applications, phase constitution and heat treatment behavior of Ti-5 to 50 mass% Ta alloys after solution treatment were investigated by electrical resistivity and Vickers hardness measurements and X-ray diffractometry (XRD). In Ti-5mass% to 30mass% Ta alloys, hexagonal martensite was identified by XRD at room temperature, whereas orthorhombic martensite was identified in Ti-40 and 50mass% alloys. Electrical resistivity at room and liquid nitrogen temperatures (RT and LN) monotonously increased with Ta content and Vickers hardness also increased with Ta content. Negative temperature dependence of resistivity did not appear in the all alloys after solution treatment. On isochronal heat treatment, increase of resistivity at LN was observed. This increase is due to reverse transformation of orthorhombic martensite to metastable beta phase.

4:10 PM
Transient and Supersolidus Sintering of Premixed and Prealloyed Bronze: *A. Upadhyaya*[1]; [1]Indian Institute of Technology, Dept. of Matls. & Metallurgl. Eng., Kanpur 208016 India

The present study investigates the processing of premixed and prealloyed bronze through transient and supersolidus liquid phase sintering. The research closely looks at the microstructural evolution in these alloys sintered in different conditions. The bronze compacts pressed at 150 and 600 MPa were sintered over a range of temperature varying from 220 to 880°C. The specific focus of the present work was to relate the microstructure and dimensional change to the transient, solid-state and supersolidus sintering occurring in bronze compacts. This study shows that microstructural evolution and sintering trajectory in bronze strongly depends on the starting powder. The microstructure and macrostructure of Cu-12Sn alloys prepared by mixing the powders differs significantly that those prepared by using prealloyed powders. Premixed bronze exhibits swelling at processing temperatures which are sufficiently high for the Sn melt to diffuse into Cu. This causes large, irregular pore formation at the intergranular regions, corresponding to prior Sn sites, which leads to poor structural rigidity. Consequently, during supersolidus condition, for the same compositions, the premixed bronze compacts distort whereas the prealloyed compacts retain their structural rigidity.

4:30 PM
Interdiffusion in Ni/CrMo Composition Modulated Films: *Alan F. Jankowski*[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., PO Box 808, L-352, Livermore, CA 94551-9900 USA

The measurement of diffusivity at low temperatures in the Ni-Cr-Mo alloy system is accomplished through the use of composition-modulated structures, i.e. multiplayer materials. The behavior of the Ni-22wt.%Cr-13wt.%Mo alloy is known at high temperatures. The stability of this corrosion resistant alloy at low temperature could be made by an extrapolation from high temperature data. As an alternative, a direct assessment of stability at lower temperatures is possible through measurements of the interdiffusion between alternating layers of Ni and Cr-Mo. X ray diffraction provides measurement of the changes in the short-range order, i.e. the composition fluctuation along the growth direction of the film, through quantification of changes in the intensity of satellite peaks positioned about Bragg reflections. The decay rate of the artificial composition fluctuation of Ni with Cr-Mo is then analyzed using the microscopic theory of diffusion to quantify the generalized interdiffusivity.

4:50 PM
Microstructural and Superconducting Properties of V, Nb, and Ti doped MgB2 Bulk and Wires: *Ulf Peter Trociewitz*[1]; Sastry V.P. S. S. Pamidi[2]; Oscar E. Castillo[2]; Justin Schwartz[3]; [1]National High Magnetic Field Laboratory, MS&T, 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA; [2]Florida State University, Ctr. for Adv. Power Sys., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA; [3]Florida A&M University-Florida State University College of Engineering, Natl. High Magnetic Field Lab., Ctr. for Adv. Power Sys., Dept. of Mechl. Eng., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA

Investigations on magnetic and transport properties of chemically doped MgB2 superconductors have been carried out. Bulk samples and powder-in-tube manufactured sheathed wire segments have been produced using Mg and B precursor powders added with various amounts of V, Nb, Ti. The impact on the microstructural, magnetization, and transport properties of both bulk and wire samples are investigated and the results compared. Preliminary experiments applying SQUID magnetometry show distinct field and temperature dependence in the effects of dopants on magnetization.

5:10 PM
Nano-Compression Properties of Carbon Micro-Balloons: *M. C. Koopman*[1]; K. K. Chawla[1]; G. Gouadec[1]; G. M. Gladysz[2]; M. W. Lewis[3]; [1]University of Alabama at Birmingham, Dept. of Matls. Sci. & Eng., BEC 254, 1530 Third Ave. S., Birmingham, AL 35294-4461 USA; [2]Los Alamos National Laboratory, Eng. Scis. & Applic. Div., Weapon Matls. & Mfg., MS C930, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Weapon Response Grp., ESA-WR, MS P946, PO Box 1663, Los Alamos, NM 87545 USA

A novel technique has been developed to measure the compressive strength of individual carbon micro-balloons, CMB, which range in size from 5-80 micrometers. CMB are used to make carbon based syntactic foams. Mechanical properties of CMBs are of interest in relation to the mechanical properties of the syntactic foams. The technique employs a nano-indentation device equipped with a cylindrical tip. The compressive strength of CMBs have been measured between 5-50 mN. Details of the procedure, as well as associated characterization of this material will be presented.

Hot Deformation of Aluminum Alloys: Deformation, Recrystallization and Texture - II

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA; Armand J. Beaudoin, University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; Balasubramaniam Radhakrishnan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6359 USA

Monday PM — Room: 6E
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Anthony D. Rollett, Carnegie Mellon University, Matls. Sci. & Eng., Pittsburgh, PA 15213 USA; Bala Radhakrishnan, Oak Ridge National Laboratory, Compu. Sci. & Math. Div., Oak Ridge, TN 37831-6359 USA

2:00 PM Invited
Static Recrystallization in AL Alloys After Hot Working: *H. J. McQueen*[1]; E. Evangelista[2]; [1]Concordia University, Dept. of Mechl. & Industl. Eng., 1455 de Maisonneuve Blvd. W., Montreal, Quebec H3G 1M8 Canada; [2]University of Ancona, Mech. Dept., Ancona 1-60131 Italy

Static recrystallization (SRX) occurs after hot working when the metal remains uncooled (possibly another straining stage) or when it is reheated (possibly solution treatment). The objectives of hot processing are products with specified strength, ductility and texture either for service or for additional forming. The mechanisms of nucleation and growth of new grains and the kinetics are essentially similar to those after cold working. However, the highly recovered microstructure, dependent on temperature and strain rate as well strain, has reduced site concentration and driving force that slow the rate and increase grain size. After extrusion, inadvertent surface SRX to large grains with a soft texture is to be avoided yet controlled SRX softening during solution is counteracted by precipitation hardening. The development of microstructure and texture in multistage rolling is controlled by both pass strain conditions and interpass times. The effects of change in strain path are shown to be significant along with those of solutes, dispersoids, precipitates, and constituent particles.

2:25 PM Invited

Recrystallization Behavior in Two Al-Mn Alloys with Different Fe Content after Extrusion and Subsequent Cold Rolling: *Stian Tangen*[1]; Hans Bjerkaas[1]; Trond Furu[2]; Erik Nes[1]; [1]Norwegian University of Science and Technology, Matls. Tech., Alfred Getz vei 2b, Trondheim 7491 Norway; [2]Hydro Aluminium, R&D Matls. Tech., Sunndals□ra 6600 Norway

The deformation and annealing behavior in two Al-Mn alloys after extrusion and subsequent cold rolling has been studied. An industrial Al-Mn alloy with relatively high concentration of iron (0.57wt%) and one Al-Mn alloy with lower Fe content (0.2wt%) were DC-cast into extrusion billets, and extruded to flat profiles. The profiles were subsequently cold rolled to true strains of 0.5, 1.5 and 2.5. Following cold deformation the materials were isothermally annealed in order to recrystallize at different temperatures. The softening reactions during annealing were followed by hardness and electrical conductivity measurements, and Time-Temperature-Transformation-diagrams have been constructed for both alloys. Recrystallized grain sizes have been measured both by the use of the Optical Light Microscope and the EBSD orientation mapping technique in SEM (OIM). The EBSD technique has also been used to study the texture development during the deformation and annealing of the materials. The particle size distributions after deformation were investigated with a FEGSEM equipped with an image analysis system.

2:50 PM

Formation of Surface Recrystallization of Al-Mg-Si Alloy: *Paul T. Wang*[1]; [1]Alcoa Inc., Process Tech. Div., Alcoa Techl. Ctr., Alcoa Ctr., PA 15069 USA

Surface coarse grain formation during extrusion has become one of the critical issues in the control of microstructure uniformity in extrusion products of Al-Mg-Si alloy. A lab-scale indirect extrusion approach is used to observe the surface behavior of an Al-Mg-Si alloy. Various thermomechanical histories under extremely large strains, strain rates, and elevated temperatures are imposed on extrudates with relative ease of handling. The lab deformed sample is then characterized by traditional metallography and electron backscatter diffraction (EBSD) techniques to understand the influence of processing conditions on surface coarse grain formation. Since the transition between coarse grain and as-extruded grain regions is very distinct by the abrupt changes of grain appearance, we propose an energetic approach through Gibbs free energy to represent a recrystallization front criterion. To track heterogeneous deformation history from surface to sub-surface of extrudates, we employ a finite element flow analysis to track the front criterion. The proposed recrystallization front criterion is sensitive to drag forces imposed by dispersoid, and thermal and deformation histories. Validations and applications of this approach are discussed.

3:10 PM

Die Shape and Surface Recrystallisation in Hot Extrusion of Aluminium Alloys: Xavier Velay[1]; *Xinjian Duan*[1]; Terry Sheppard[1]; [1]Bournemouth University, Sch. of Design, Eng. & Compu., 12 Christchurch Rd., Bournemouth, BH1 3NA England

For hard aluminium alloys, such as 2xxx series and 7xxx series, a typical structure is observed in the extrudate after press quench: a fibrous core surrounded by a shell of recrystallised grains. Previous work has shown that the die configuration has a substantial influence on the recrystallisation behaviour. In this communication, the influence of die design on the control of surface recrystallisation is detailed. The fraction recrystallised is predicted by coupling the physical models (based on dislocation density, subgrain size and misorientation) with the FEM. The die shapes considered include: flat-faced nonlubricated die, choked non-lubricated die and stream line die. The distributions of equivalent strain and velocity in the cross section under different extrusion conditions are also presented.

3:30 PM Invited

A Mathematical Model of the Microstructural Evolution During Hot Rolling of Aluminum Alloy AA5083: *H. Ahmed*[1]; M. A. Wells[1]; D. M. Maijer[1]; M. R. van der Winden[2]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]IJmuiden Technical Centre, Corus RD&T, PO Box 10.000, IJmuiden 1970 CA The Netherlands

Microstructure engineering is increasingly gaining attention with the goal of developing predictive mathematical models that link the operational parameters of an industrial process with the final properties of the product. Towards this goal, models have been developed over the past decade to predict the final microstructure and subsequent evolved texture during hot rolling of aluminum sheet products. In this research, a 2-D coupled thermomechanical model was developed and used to simulate multi-pass hot rolling of an AA5083 aluminum alloy using the commercial finite element software package, ABAQUS. The model was used to predict through thickness strain, strain rate, and temperature distributions of the strip at any position in the roll bite, and the ensuing microstructure evolution in the inter-strand region. The model predictions, including microstructure evolution, were validated through comparison with detailed experimental measurements conducted using the CORUS Research Multi-mill, a pilot scale experimental rolling facility in IJmuiden, Netherlands. A sensitivity analysis was carried out to study the effect of boundary conditions used in the model and processing conditions in the mill on the predicted results.

3:55 PM Invited

Development of Through-Thickness Texture Gradient During Continuous Cast Processing of AA 5052 Aluminum Alloy: *Wenchang Liu*[1]; Zhong Li[2]; Tongguang Zhai[1]; Chi-Sing Man[3]; James G. Morris[1]; [1]University of Kentucky, Dept. of Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA; [2]Comonwealth Aluminum Corporation, 1505 Bull Lea Rd., Lexington, KY 40511 USA; [3]University of Kentucky, Dept. of Math., 715 Patterson Office Tower, Lexington, KY 40506 USA

Because the texture that develops during hot rolling has been shown to directly influence the texture of the annealed materials entering the cold-rolling mill, understanding the texture development in the hot rolling process is especially important. In the present work, the texture evolution and the development of through-thickness texture gradient during continuous cast processing of AA 5052 aluminum alloy was investigated by X-ray diffraction. The continuous cast slab and the samples in each pass during hot rolling were obtained from the industrial continuous cast process. The microstructure change through the thickness in the continuous cast slab of AA 5052 aluminum alloy was determined. The effect of the microstructure change through-thickness in the continuous cast slab as well as the roll-gap geometry and the friction between the roll and the sheet contact surface on the through-thickness texture gradient in the hot bands is discussed.

4:20 PM Invited

Analysis and Comparison of Dislocation Structures in Cold and Hot Deformation: *Grethe Winther*[1]; Niels Hansen[1]; [1]Risoe National Laboratory, Matls. Rsrch. Dept., Ctr. for Fundl. Rsrch., Metal Struct. in 4D, Roskilde DK-4000 Denmark

Studies of dislocation structures are relevant for both cold and hot deformation as the dislocation structure is an important factor controlling mechanical properties and also the environment in which recovery and recrystallisation take place. Characterisation of structures in cold deformed aluminium has now reached a state where understanding of the physical processes leading to these structures is well under way. Specifically, strain and crystallographic slip have been identified as controlling factors. A number of structural parameters, e.g. boundary spacing and misorientation and boundary planes, can be linked to the processing conditions. Structural characterisation along these lines is more elaborate than that normally carried out for hot deformation. The framework established for cold deformation will be applied to hot deformation. Analysis of cold and hot deformation microstructures in the same framework facilitates assessment of temperature-induced similarities and differences in the physical mechanisms behind structure evolution.

4:45 PM

Microstructural Evolution in Al-3%Cu Alloy: *Inna Mazurina*[1]; Rustam Kaibyshev[1]; Oleg Sitdikov[2]; Irina Denisova[1]; [1]Institute for Metals Superplasticity Problems, Khalturina, 39, Ufa 450001 Russia; [2]University of Electro-Communications, Dept. of Mechl. & Control Eng., Chofu, Tokyo 182-8585 Japan

The microstructure evolution during plastic deformation was examined in an Al-3%Cu alloy by metallographic technique, TEM and electron back-scattered diffraction (EBSD) technique. Samples were subjected to intense plastic straining via equal channel angular extrusion up to true strain ranging from 1 to 8 at a temperature of 150°C. It was shown that dense dislocation walls are evolved after e=1. Following strain results in the formation of elongated subgrains owing to transformation of dense dislocation walls into subgrain boundaries. Low-angle boundaries convert into high-angle boundaries with further strain and crystallite become essentially equiaxed. As a result, recrystallized grains were found at e≥4. At e=8, volume fraction of recrystallized attends about 50 pct. Mechanism of fine grain formation is discussed.

5:05 PM
Geometric Dynamic Recrystallization During Intense Plastic Straining in an 2219 Aluminum Alloy: *Rustam Kaibyshev*[1]; Oleg Sitdikov[2]; Inna Mazurina[1]; [1]Institute for Metals Superplasticity Problems, Khalturina, 39, Ufa 450001 Russia; [2]University of Electro-Communications, Dept. of Mechl. & Control Eng., Chofu, Tokyo 182-8585 Japan

Microstructural evolution was examined during equal channel angular extrusion (ECAE) has been performed in an 2219 aluminum alloy with initial grain size of about 120 mm at a temperature of 475°C up to true strain e=12 The deformed structure was examined by optical microscopy, TEM method, electron back-scattered diffraction (EBSD) technique. It was found that severe plastic deformation leads to subdivision of initial grains into coarse new grains. It was shown that geometric dynamic recrystallization (GRX) takes place. Mechanism of GRX is discussed. The role of deformation induced low-angle boundaries in GDR is considered.

International Symposium on Gamma Titanium Aluminides: Fundamentals

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee

Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

Monday PM	Room: 6F
March 3, 2003	Location: San Diego Convention Center

Session Chairs: Michael H. Loretto, University of Birmingham, IRC in Matls., Birmingham B15 2TT UK; Vijay K. Vasudevan, University of Cincinnati, Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA

2:00 PM Invited
Equilibrium of Multi-Phase Gamma Alloys: A Quantitative Approach Based on Site Occupancies of Alloying Additions: *Rui Yang*[1]; Yulin Hao[1]; Yan Song[2]; Yuyou Cui[1]; [1]Chinese Academy of Sciences, Inst. of Metal Rsrch., Titanium Alloy Lab., 72 Wenhua Rd., Shenyang 110016 China; [2]Queen Mary, University of London, Dept. of Matls., Mile End Rd., London E1 4NS England

The sophisticated alloying strategy that accompanies the development of high performance gamma alloys does not always produce predictable effects on the balance of constituent phases. A mean-field type model that estimates the phase fractions from site occupancy data is therefore useful for alloy design in the absence of systematic phase equilibrium information. This paper will summarize our recent studies aimed at accurate determination and electronic structure interpretation of the site occupancy information of more than a dozen elements in gamma and related phases. In particular, it will be shown that the structure of point defects has significant influence on the accuracy of site preference measurement. The effectiveness of this approach in predicting the amounts of constituent phases of gamma alloys will be demonstrated using examples of ternary and quaternary two-phase alloys and ternary three-phase alloys.

2:30 PM Invited
Suppression of the Massive Transformation in a Ti-48 At. % Al Alloy: A Direct Consequence of the Oxygen Induced Chemical Ordering of the Alpha Phase: *Alain Menand*[1]; Williams Lefebvre[1]; Annick Loiseau[2]; [1]University of Rouen, Inst. of Matl. Rsrch., Grp. on Physl. Metall., UFR Sci., BP 12, Saint Etienne du Rouvray 76800 France; [2]ONERA, LEM-UMR CNRS-ONERA, 29 ave. de la Division Leclerc, BP 72, Chatillon 92322 France

We have investigated, for years, the oxygen behaviour in Ti-Al alloys. Recently we focussed on the influence of oxygen on the development of microstructures in a Ti-48 at. % Al model alloy. We observed that, for oxygen content higher than 1.2 at. %, the massive transformation is suppressed. Instead of a massive γ structure, the alloy displays a fully ultrafine lamellar structure. Then we established that the starting temperature of the chemical ordering reaction of the α-phase increases with the oxygen content. This means that oxygen induces the chemical ordering of the α-phase above the eutectoid temperature. From this result we deduce that, above a critical oxygen content, the starting temperature of the chemical ordering reaction of the α-phase becomes superior to the starting temperature of the massive transformation. The direct consequence is the suppression of the massive transformation above a 1.2 at. % nominal oxygen content.

3:00 PM
Phase Transformations and Microstructure Evolution in Multi-component Gamma Titanium Aluminides: *Krishna C. Cherukuri*[1]; Dennis M. Dimiduk[2]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Air Force Research Laboratory, WPAFB, Bldg. 655, Dayton, OH 45433 USA

Phase transformations and microstructure evolution involving the high-temperature beta, alpha or both phases during continuous cooling, as well as during step-quenching + isothermal holding in multi-component gamma alloys (viz., Ti-(45-47)Al-2Cr/Mn-2Nb-(0-1)Mo-(0-0.3)B) were studied, and the competition in temperature and time space between the various transformation modes, namely, lamellar, cellular, feathery and massive gamma were ascertained. The structure and chemistry of phases present in alloys were determined, and these results were useful for understanding the change in decomposition processes with alloying and temperature. Continuous cooling transformation diagrams were established and detailed microscopy was performed to unravel transformation mechanisms associated with the various non-lamellar transformation modes (i.e. massive, feathery and cellular) and the influence of chemistry. Decomposition of the beta phase is found to take two forms: precipitation of coarse gamma platelets within grain boun dary films and as a cellular reaction leading to alternate gamma and beta/B2 lamellae. This reaction is driven by the reduced solubility of Al and increased Cr and Mo solubilities in the beta phase with decreasing temperature. Both the Al level and the concentration of transition elements can significantly alter both the kinetic windows for the various constituents and the ability to form perfect lamellar structures. Reducing the levels of both and additions of boron are beneficial for minimizing unfavorable cellular reactions and promoting the lamellar transformation. The authors are grateful for support of this research by the National Science Foundation (Grant # DMR-9731349, Dr. Bruce MacDonald, Program Monitor) and AFOSR (Grant # F49620-95-1-0116, Dr. Craig. S. Hartley, Program Monitor).

3:20 PM Invited
Evaluation of the Jogged-Screw Model for Creep of TiAl: Karthik Subramanian[1]; G. Babu Viswanathan[1]; *Michael John Mills*[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

A modified version of the jogged-screw model has been proposed recently to describe creep of equiaxed Ti-48Al. Previous TEM studies have shown that these dislocations are indeed prominent and typically have numerous, tall jogs along their length which limit their movement. This presentation will describe our attempts to verify and validate the parameters and functional dependencies that have been assumed thus far in the model. The original dislocation velocity law has been reformulated to take into account the finite length of the moving jogs. The critical substructural parameters of average jog height, jog spacing and dislocation density (and their dependence on stress) have also been more completely evaluated based on a combination of experimental measurements and computer simulation. Combining all of these parameters and dependencies leads to a new model that provides an excellent prediction of creep rates and stress exponents. The further extension of this model to the problem of creep in fully lamellar structures will also be discussed.

3:50 PM
Channeled Flow During Deformation of PST TiAl Single Crystals, an AFM Study: *David P. Pope*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

PST TiAl crystals have been deformed in compression such that the deformation axis lies in the (111) interfacial planes, producing so-called ìchanneled flowî. The sample simply shortens axially and spreads laterally in the channels defined by the (111) interfacial planes with zero strain perpendicular to the lamellae. We have found, after observation of many slip bands in the AFM, that the character of the bands and the orientations of the lamellae can be ascertained simply by observing how straight and continuous the slip bands are. The bands can be identified by the fact that those containing a combination of slip and twinning are very straight and uniform, while those that contain just slip are much less so (there are no bands that contain just twinning for this orientation). By the same token, knowing the nature of the bands, tells us the orientation of the lamellae through which the bands pass† without using TEM to orient the bands. Also, once the deformation process is well developed, an individual band tends to propagate entirely across the crystal, even though pairs of bands do not necessarily meet at a given lamellar interface.

4:10 PM Invited
Deformation TwinsñTheir Nucleation and Development in TiAl: *Franz Dieter Fischer*[1]; Henryk Petryk[2]; Fritz Appel[3]; Helmut Clemens[4]; [1]Montanuniversit‰t Leoben, Inst. of Mech., Franz-Josef-Strasse 18, Leoben 8700 Austria; [2]Polish Academy of Sciences, Inst. of Fundaml. Technologl. Rsrch., Swietokrzyska 21, Warszawa 00-049 Poland; [3]GKSS Forschungszentrum, Inst. for Matls. Rsrch., Max-Planck-Strasse, Geesthacht 21502 Germany; [4]GKSS Forschungszentrum, Inst. f¸r Werkstoffforschung, Max-Planck-Strasse, Geesthacht 21502 Germany

Twinning is an important deformation mechanism in TiAl at room and high temperature. Due to the reduced number of slip systems twinning is activated even at small strains. A micromechanical study explains that twin nuclei of a certain length and distance are generated only due to the mechanical driving force from geometrically necessary dislocations along grain boundaries. Twin bands can suddenly develop from the twin nuclei if the energy of an externally loaded specimen is globally lowered. The thickness and spacing of twin bands can be estimated from the stability analysis. Finally twinning is macroscopically interpreted as an additional slip system to the dislocation slip systems with only unidirectional shearing. Acoustic emission tests signal the onset of twins. A good correlation between experiments and modelling has been found. This contribution delivers an understanding of twinning starting from atomistic investigations to micromechanical modelling.

4:40 PM
Grain Coarsening and Subsequent Lamellar Structure Formation in a Ti-45Al-2Nb-0.4Mn Alloy at Super-Transus Temperatures: *Uttara Prasad*[1]; Mahesh C. Chaturvedi[1]; [1]University of Manitoba, Dept. of Mechl. & Industl. Eng., Winnipeg, Manitoba R3T 5V6 Canada

The present research aims at addressing some of the fundamental aspects of lamellar structure formation in gamma titanium aluminides. The phenomenon of grain growth in the alpha phase field, and the effect of annealing time/prior alpha grain size on the formation of lamellar microstructure upon subsequent furnace cooling were studied in Ti-45Al-2Nb-0.4Mn alloy. The grain size increased with an increase in annealing time in the alpha-phase field and the grain growth exponent of 0.52 was obtained at 1350°C. Interlamellar spacing in the lamellar microstructure formed upon furnace cooling from the alpha-phase field was also observed to increase with an increase in annealing time, i.e, interlamellar spacing increased with increase in the grain size. In addition, it was also observed that the lamellar grain boundaries became increasingly planar with annealing time. The role of the prior alpha grain boundaries on lamellar structure formation and mechanical properties will be presented and discussed.

5:00 PM Invited
Microstructural Modification for Enhanced Creep Resistance in XD TiAl Alloys: *Dong Yi Seo*[1]; Linruo Zhao[1]; Jonathan Beddoes[2]; [1]National Research Council of Canada, Struct., Matls. & Propulsion Lab., Inst. for Aeros. Rsrch., Montreal Rd., Bldg. M-13, Ottawa, Ontario K1A 0R6 Canada; [2]Carleton University, Mechl. & Aeros. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada

The effects of novel heat treatment and directional solidification (DS) process on the microstructure and creep properties of XD TiAl alloys are reviewed in this paper. Fine grain fully lamellar (FGFL) microstructure is desirable to achieve a combination of high strength and good ductility in TiAl intermetallics. Solution heat treatments with different cooling rates were applied to investment cast XD TiAl alloys to obtain the FGFL microstructure. Both grain size and lamellar spacing are function of the cooling rate. For microstructural stabilization, the solution treated samples were subsequently subjected to an aging treatment in the a□2□g□ field. Although the aged FGFL microstructure exhibits relatively low steady state creep rate, the unstable grain boundary region is responsible for the early onset of tertiary creep. To alleviate the negative influence of transverse grain boundaries on creep, DS process was applied to XD TiAl alloys using a pilot-scale Bridgeman furnace, followed by solution and aging treatments. The resulting microstructure contains short elongated columnar grains, with a fully lamellar morphology, aligned with the withdraw direction. Preliminary creep tests have shown that the XD TiAl alloys processed by DS technique yield drastic improvement in creep resistance over the FGFL XD TiAl alloys. This study indicates that directional solidification could be the process of choice for fabricating TiAl components for which creep resistance is the primary concern in application.

5:30 PM
Atomic Scale Chemistry of Lamellar Interfaces in a Complex TiAl Alloy: *Stephan S.A. Gerstl*[1]; Young-Won Kim[2]; David N. Seidman[1]; [1]Northwestern University, Matls. Sci., 2225 N. Campus Dr., Evanston, IL 60208 USA; [2]UES, Inc., Matls. & Procg. Div., 4401 Dayton-Xenia Rd., WPAFB, OH 45432 USA

Microalloying of titanium aluminide alloys provides precipitation strengthening, refines lamellar spacing, and improves creep and oxidation resistance. Little is known, however, where the alloying elements ultimately reside within the microstructure. We report on a K5-series alloy, which achieves carbide strengthening above 840∞C, which is approximately a 100∞C improvement over conventional TiAl alloys. A three-dimensional atom-probe microscope (3DAP) is utilized for analyzing lamellar interfaces, in an alloy containing 3 Nb, 1.5 Cr, and 0.2 (at.%) of B, C, Hf, Mn, W, and Zr. Carbon, Mn, and Cr partition to the a2 phase, whereas Nb and Zr prefer the g phase. Boron was found primarily in borides. Both W and Hf exhibit concentration gradients within 6 nm of the interface. Their near interfacial excesses are determined and it is suggested that Hf, or the combination of W and Hf, provide an additional stabilizing effect for the lamellae at higher temperatures.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C.T. Liu: Intermetallics II–Nickel Aluminide

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Seetharama C. Deevi, Philip Morris USA, Research Center, Richmond, VA 23234 USA; Fritz Appel, GKSS Research Centre, Geesthacht D-21502 Germany; Robert W. Cahn, University of Cambridge, Materials Science and Metallurgy, Cambridge CB2 3QZ UK; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Guo Liang Chen, University of Science and Technology-Beijing, State Key Laboratory for Advanced Metals and Materials, Beijing 100083 China; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208-3108 USA; Shuji Hanada, Tohoku University, Sendai 980-8577 Japan; Linda Horton, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6132 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Dong-Liang Lin, Shangai Jiao-Tong University, Shangai 200030 China; T. G. Nieh, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; Masaharu Yamaguchi, Kyoto University, Department of Materials Science & Engineering, Kyoto 606-8501 Japan

Monday PM — Room: 8
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: T. G. Nieh, Lawrence Livermore National Laboratory, L350, Livermore, CA 95014 USA; Masaharu Yamaguchi, Kyoto University, Dept. of Matls. Sci. & Eng., Kyoto 606-8501 Japan

2:00 PM Invited
Intermetallic Compounds as Contacts to IIIV Compound Semiconductors Including Gallium Nitrides: *Y. A. Chang*[1]; Chris M. Pelto[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Scis. & Eng., 1509 Univ. Ave., Madison, WI 53706-1595 USA

In this presentation, we will first review a combined thermodynamic/kinetic model to successfully use intermetallics such as NiAl, CoAl, and PdIn as contacts to IIIV compound semiconductors for Schottky and ohmic enhancement and then our recent results on a thermally stable, oxidation-resistant capping technology utilizing TiAl3, also an intermetallic, for the state-of-art ohmic contact to n-GaN. In the first part of the presentation, we will give examples to show the power of this combined model in identifying intermetallic compounds either as potential ohmic or Schottky contacts to IIIV compound semiconductors and their alloys. In the second part we will show the capping technology developed in our laboratory can be readily adapted for industrial practice in order to significantly improve the contact technology currently used in fabricating GaN-based lasers!

2:20 PM

Vacancies in Al-Rich Ni-Al: *P. M. Hazzledine*[1]; Y. Q. Sun[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]University of Illinois, Dept. of Matls. Sci. & Eng., Champaign, IL 61801 USA

On the Al-rich side of stoichiometry, Ni-Al responds to the shortage of Ni atoms largely by forming Ni vacancies rather than anti-site defects. The concentrations of these Ni vacancies may be very large, in the % range. In addition to the constitutional Ni vacancies, quenching and aging experiments show that the concentrations of thermal vacancies (equal numbers of Ni and Al vacancies) may also be exceptionally high, as high as 1% at the melting point of 1911K. Concentrations as large as this indicate an effective formation energy for a vacancy pair which is much smaller than the calculated value. A possible explanation for the discrepancy between experiment and theory is that there is a strong interaction between vacancies by which a high concentration of vacancies promotes the creation of further vacancies. The nature of this interaction and an estimate of its value is made in the paper.

2:35 PM Invited

Magnetism-Induced Solid Solution Softening in Intermetallic NiAl: *C. L. Fu*[1]; C. T. Liu[1]; Maja Krcmar[1]; Xun-Li Wang[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA

The effect of iron and cobalt solutes on hardening in NiAl has been investigated. Ternary additions of iron and cobalt with similar atomic sizes were added to replace nickel in Ni-40Al. Cobalt solutes did not affect the lattice parameter or the hardening behavior of NiAl alloys. Iron solutes, on the other hand, substantially expanded the lattice, resulting in unusual solid solution softening. From the contrasting behavior of the iron and cobalt solutes, magnetic interactions induced by iron atoms located on the aluminum sublattice have been identified as the physical source responsible for the unusual solid solution softening. Neutron diffraction experiments are underway to determine the magnetic structure in Ni50(Al40Fe10). Our study will be extended to include Mn and Cr solutes. Work sponsored by the Division of Materials Sciences and Engineering, ORNL is operated by UT-Battelle, LLC, for the USDOE under contract DE-AC05-00OR22725.

2:55 PM

Mechanisms of Grain Boundary Diffusion in B2 NiAl: *Diana Farkas*[1]; Benjamin Soule de Bas[1]; [1]Virginia Tech, Matls. Sci. & Eng., Blacksburg, VA 24061 USA

We present the results of large scale molecular dynamics simulations of grain boundary diffusion in B2 NiAl. The studies use EAM interatomic potentials to simulate the diffusion process in a direct manner, starting with vacancies located in different sites in the grain boundary region. The simulations show a variety of diffusion jumps, all introducing disorder in the grain boundary. This is in contrast to diffusion in bulk NiAl, where cyclic mechanisms, restore order. Several series of correlated jumps were identified in the grain boundary region. The most common of these result in the vacancy remaining in the same structural unit. The rate controlling jump sequences that result in the vacancy diffusing to a neighboring structural unit were also observed and are reported in detail.

3:10 PM Invited

Large-Scale Manufacturing of Nickel-Aluminide Transfer Rolls for Steel Austenitizing Furnaces: *Vinod Kumar Sikka*[1]; Michael L. Santella[1]; Peter Angelini[1]; John Mengel[2]; Robert Petrusha[2]; Anthony P. Martocci[3]; Roman Ivan Pankiw[4]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA; [2]Bethlehem Steel Corporation, Burns Harbor Div., Box 248, Chesterton, IN 46304 USA; [3]Bethlehem Steel Corporation, Corporate Office, 1170 Eighth Ave., Martin Tower, Rm. 467, Bethlehem, PA 18016-7699 USA; [4]Duraloy Technologies, Inc., 120 Bridge St., Scottdale, PA 15683-0081 USA

Transfer rolls are used in steel heat-treating furnaces for the transfer of steel plates from one end to the other end of the furnaces such that the steel plates are exposed to the correct austenitizing temperature prior to cooling by water quenching or air cooling. Typical furnace temperatures can range from 900 to 1000∞C. The rolls fabricated from conventional alloys undergo a phenomenon of blister formation on the roll surface. The surface areas of the blisters are 2 to 3 by 4 to 2 in. that is raised up from the smooth surface by as much as 0.25 to 0.75 in. The edges of the blister are sharp and they cause surface scratching of the steel plates moving over them. In order to minimize surface damage, the edges of the blisters are ground smooth on a very frequent basis. In early furnace trials ranging up to five years, no blisters were observed on rolls fabricated from Ni_3Al-based nickel aluminide alloy IC-221M. Based on the trial experience, a full furnace lead of 115 nickel aluminide rolls are being manufactured. This paper will describe the details of melting, casting, machining, welding, and operating experience for large-scale manufacturing of Ni_3Al-based alloy rolls. Research sponsored by the US Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Industrial Materials for the Future Program under Contract De-AC05-00OR22725 with UT-Battelle, LLC.

3:30 PM Break

3:45 PM Invited

The Role of Hydrogen Diffusion and Desorption in Moisture-Induced Embrittlement in Intermetallics Doped with Alloying Elements: Yanfeng Chen[1]; *Yip-Wah Chung*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

A statistical model was developed to explain effects of alloying elements in moisture-induced embrittlement in intermetallics. This model shows that if alloying elements have strong binding to atomic hydrogen, they may suppress moisture-induced embrittlement by slowing hydrogen diffusion even at low concentrations. The concentration needed to effectively slow down the diffusion process decreases with temperature. Grain boundary segregation is not a necessary condition to suppress embrittlement as long as the alloying element binds to hydrogen sufficiently strong and its concentration is above a certain level. In the other extreme, when the binding of hydrogen is so weak that hydrogen desorption occurs during tensile deformation, the model demonstrates that above a certain desorption rate, there may be not enough hydrogen diffusing to the crack tip to cause embrittlement.

4:05 PM Invited

Mechanical Properties of Cold-Rolled Ni_3Al Thin Foils: *Toshiyuki Hirano*[1]; Masahiko Demura[1]; Kyosuke Kishida[1]; Yozo Suga[2]; [1]National Institutes for Materials Science, Mechl. Eng. Lab., 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047 Japan; [2]Nippon Cross Rolling Company, 697 Mobara, Mobara, Chiba 297-0026 Japan

Thin foils of intermetallic compounds are attractive as lightweight high-temperature materials in the form of honeycomb structure because of their excellent high-temperature properties. The key issue is how to fabricate of such thin foils of brittle intermetallic compounds. Recently we have successfully fabricated binary Ni_3Al thin foils, about 20 micrometer in thickness, by cold rolling of the nearly single crystals. The total reduction in thickness exceeds 99%. We will present that these foils have good mechanical properties for application in honeycomb structure. Because of heavy cold reduction, the foils have very high tensile fracture strength at room temperature, 1.3~1.8 GPa, having weak anisotropy about the rolling direction. Though no tensile elongation is discernable, interestingly they have a good bending ductility parallel to the rolling direction. The tensile and bending properties can be related to the deformation textures and microstructures developed in the foils.

4:25 PM Invited

Essentials in Diffusion Behavior of Nickel- and Titanium-Aluminides: *Christian Herzig*[1]; Sergiy V. Divinski[1]; [1]University of Muenster, Inst. fuer Materialphysik, Wilhelm-Klemm-Str. 10, Muenster D-48149 Germany

Recently we have extensively investigated bulk and grain boundary diffusion in technologically important Ni- and Ti-aluminides. These compounds exhibit different lattice structures and different types and concentrations of lattice defects on their sublattices (vacancies, antistructure atoms of constitutional and/or thermal origin) allowing for a restricted number of atomic jump possibilities and diffusion mechanisms. This study therefore provides fundamental insight into the interdependence of diffusion behavior and diffusion mechanisms on structure and ordering. The overview includes the direct tracer diffusion measurements of the transition metal component and the determination of the Al diffusivity through interdiffusion data and by using Al-substituting solutes. The interpretation is strongly supported by EAM-potential calculations of defect properties and Monte-Carlo simulations of possible diffusion mechanisms. Among other features, the experimentally established curvature of Ti diffusion in TiAl, the surprising effect of the missing diffusion enhancement by structural vacancies in Al-rich NiAl, and the paradoxically appearing deep minimum of the chemical diffusion coefficient in stoichiometric NiAl are explained.

4:45 PM

Alloying Effect on Stability of Multi-Variant Structure of Ni_3V at Elevated Temperatures: *Akane Suzuki*[1]; Hisayasu Kojima[1]; Masao Takeyama[1]; Takashi Matsuo[1]; [1]Tokyo Institute of Technology, Dept. of Metall. & Ceram. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Stoichiometric tetragonal compound of $Ni_3V(D0_{22})$ quenched from disordered fcc(A1) state exhibits a unique morphology of ìmulti-variant structure (MVS)î in which three variants are periodically arrayed with coherent $(102)_{D022}$, $(10\text{-}2)_{D022}$ and $(010)_{D022}$ interfaces. The major variant exhibits quadrangular-prism shape and two minor variants fill in the $\{102\}_{D022}$ and $\{010\}_{D022}$ channels, respectively. This MVS is formed to minimize the transformation strain due to tetragonality of the $D0_{22}$ phase ($a_{D022} < a_{A1} < c_{D022}/2$). However, because of the rigidity the MVS collapses rapidly accompanied with coherency loss of these interfaces during aging at elevated temperatures. Introduction of A1 to the channels by excess additions of Ni and/or Co makes the MVS stable without coherency loss of the interfaces. The resulting morphology becomes either ìpyramidal-maze structureî or ìprismatic chess-boar d structureî, depending on magnitude and sign of the lattice misfit at $A1/D0_{22}$ interfaces. The details on the formation and stability of these structures will be presented.

5:00 PM Invited
Microstructures and Mechanical Properties in Ni_3Al-Ni_3Ti-Ni_3Nb-Based Multi-Intermetallic Alloys: *Takayuki Takasugi*[1]; Yasuyuki Kaneno[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

The phase relation, microstructures, high-temperature deformation and oxidation behavior of intermetallic alloys based on Ni_3Al-Ni_3Ti-Ni_3Nb pseudo-ternary alloy system were investigated. As the constituent intermetallic phases, $L1_2(Ni_3Al)$, $D0_{24}(Ni_3Ti)$, $D0_a(Ni_3Nb)$ and $D0_{19}(Ni_3Ti_{0.7}Nb_{0.3})$ were identified and then their phase fields were discussed based on the electrical and geometrical factors of constituent atoms. Among four intermetallic phases, five kinds of two-phase relations and two kinds of three-phase relations were found to exist. Also, $D0_{24}(Ni_3Ti)$ phase extended up to concentration field in which a majority of constituent Ti elements were replaced by Al and Nb elements. The prepared alloys exhibited widely different microstructures, depending on the number and kinds of the constituent intermetallic phases. Three-phase micros tructures composed of $L1_2(Ni_3Al)$, $D0_{24}(Ni_3Ti)$ and $D0_a(Ni_3Nb)$ showed extremely superior high-temperature strength and ductility, and also corrosion and oxidation properties to their constituent intermetallic phases.

5:20 PM Invited
Sintering Study for Ni3Al and Ni3Al/NiAl P/M Products: Wen-Chich Chiou[2]; *Sanboh Lee*[1]; Chen-Ti Hu[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 101 Kuang Fu Rd., Sec. 2, Hsinchu 300 Taiwan; [2]National Tsing Hua University, 101 Kuang Fu Rd., Sec. 2, Hsinchu 300 Taiwan

A multistage sintering process for nickel aluminide intermetallic compound (IC) has been employed to manufacture the Ni3Al and the Ni3Al plus 25 vol% NiAl powder metallurgical (P/M) products. It comprises at least two stages of sintering and an inter-stage cold deformation to collapse and eliminate the sintering induced pores. A useful transient phase Ni2Al3 has been developed during the preliminary heating stage. It plays an important role to prevent the growth of significant cracks in the pore-eliminating process. The second-stage of sintering at an elevated temperature (1200°C) is employed to transform a transient liquid phase from the Ni2Al3 to heal any microcracks and collapse pores as well as to transform the material to the final Ni3Al or the Ni3Al plus 25 vol% NiAl structure. The tensile ductilities of Ni3Al and Ni3Al plus 25 vol% NiAl specimens at room temperature are 2.9% and 0.3%, respectively.

International Symposium on Structures and Properties of Nanocrystalline Materials: Mechanical Properties I

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Monday PM	Room: 14B
March 3, 2003	Location: San Diego Convention Center

Session Chairs: Amiya K. Mukherjee, University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616 USA; Sung H. Whang, Polytechnic University, Dept. of Mechl. Eng., Brooklyn, NY 11201 USA

2:00 PM
Bulk Nanostructured SPD Materials with Unique Properties: *Ruslan Z. Valiev*[1]; [1]Ufa State Aviation Technical University, Inst. of Physics of Adv. Matls., 12 K. Marx St., Ufa 450000 Russia

When severe plastic deformation (SPD), i.e. intense plastic straining under high imposed pressure is applied to crystalline solids, the processed bulk materials can posses nanostructures and exhibit novel properties. In this paper we show that an advanced combination of mechanical and physical properties can be observed in SPD-processed materials as well. The selected materials were a shape memory alloy NiTi, a Cu-0.5%Al2O3 metal-matrix composite and a ferromagnetic Nd-Fe- B alloy. SPD processing allowed these materials to be highly cold worked, in spite of its intrinsic brittleness. The processed nanostructures and their evolution during heating were studied by TEM/HREM, X-ray, and DSC methods. The formation of nanostructures in NiTi by means of SPD enabled to observe an enhanced strength and improved inelasticity and shape-memory effect. The bulk Cu-0.5%Al2O3 nanocomposite could exhibit very high mechanical properties and electric conductivity. The nanostructured Nd-Fe-B demonstrated attractive magnetic properties. The origin and promising applications of nanostructured SPD-processed materials with multifunction properties are considered and discussed.

2:30 PM
Viscoelastic Behavior in Nanostructured Nickel: *Sung H. Whang*[1]; Weimin Yin[1]; [1]Polytechnic University, Dept. of Mechl. Eng., Six MetroTech Ctr., Brooklyn, NY 11201 USA

Viscoelastic behavior is one of the distinct deformation characteristics in nanostructured metals and alloys. Such viscoelastic behavior appears to be associated with grain boundary sliding based on recent findings by many researchers. In this presentation, we will report creep and damping in nanostructured nickel at/above room temperatures. It is remarkable that the anelastic relaxation and deformation characteristics are very sensitive to the segregation of interstitial elements at the grain boundaries. The results show that the creep in nickel appears to be a Coble type, but a significant departure from the Coble creep is seen above room temperatures, which will be discussed. The results on creep and internal friction experiments will be presented. Discussion will be made concerning viscoelastisc behavior and probable grain-boundary sliding in this material.

2:50 PM
Anelasticity and Mechanical Behaviour: Size Effects in Nanocrystalline Metals: *Ennio Bonetti*[1]; [1]University of Bologna, Dept. of Physics & INFM, v.le Berti Pichat 6/2, Bologna 40127 Italy

Experimental evidence has been accumulated in recent years, demonstrating that material size reduction below critical values significantly modifies physical properties with respect to those commonly observed in conventional materials. The mechanical behaviour in the whole range encompassing elastic to plastic regime via anelasticity, does not constitute exception to the general rule for which dimensionality effects deriving from structural constraints on physical mechanisms, can set up when structural dimensions enter the scale lenght of a specific property. Anelastic relaxation processes have been recently reported which are properly described taking into account dimensionality. The talk will review experimental results obtained by mechanical spectroscopy techniques on nanocrystalline metals and alloys prepared by different synthesis procedures. The results will be critically compared to those obtained on conventional materials aiming at understanding some specific aspects of the mechanical behaviour of nanocrystalline metals.

3:10 PM
The Study on Nanocrystalline (Al+12.5at.%Cu)3Zr Intermetallic Compounds Sintered by SPS: *Seung Hyun Lee*[1]; Seung Chul Kim[1]; Kyung Il Moon[1]; *Kyung Sub Lee*[1]; [1]Hanyang University, Dept. of Matls. Sci. & Eng., #404, New Matl. Eng. Ctr., 17, Haengdang-dong, Seongdong-ku, Seoul 133-791 S. Korea

We report spark plasma sintering(SPS) process as one of the most effective consolidation process of trialuminide intermetallic compound. L12 phase (Al+12.5at.%Cu)3Zr intermetallic powder was prepared by planetary ball mlling(PBM). Its grain size was 5-7nm. The powder could be consolidated to full density by SPS at 565°. The temperature was 300-400° lower than the conventional consolidation temperature of zirconium trialuminide. The L12 phase was maintained during SPS process. The average grain size of the compact was 8.8 nm and all the grains had sizes between 6-13 nm. This is one of the smallest grain size ever reported in the nanocrystalline compact prepared by various consolidation methods. Accordingly, the specimens had the micro-hardness values over 1000 kg/mm2.

3:30 PM Break

3:50 PM
Nanocrystalline Materials from Devitrification of Fe-Based Metallic Glasses: *Alla V. Sergueeva*[1]; Nathan A. Mara[1]; Amiya K. Mukherjee[1]; [1]University of California, Cheml. Eng. & Matl. Sci., 1220 Bainer Hall, Davis, CA 95616 USA

Crystallization by annealing of Fe-based metallic glasses was used to produce fully dense nanocrystalline materials. It was shown that the size, morphology of crystallites, mechanism of crystallization, and crystallization products themselves depend on the temperature of devitrification of a metallic glass. Therefore, completely crystallized nanostructures with different morphological characteristics can be obtained through differences in processing. Strong influence of the microstructural characteristics other than grain size were revealed by tensile tests of such materials. The main emphasis of the current investigation was on the deformation behavior of equiaxed nanocrystalline materials with homogeneous structure. Results were analyzed in terms of a rate equation describing deformation behavior with respect to evolution of microstructure to nanometer scale.

4:10 PM
Mechanical Properties of Co-Based Nanocomposites: *Glenn David Hibbard*[1]; Doug Lee[2]; Uwe Erb[3]; Gino Palumbo[1]; [1]Integran Technologies, Inc., 1 Meridian Rd., Toronto, Ontario M9W 4Z6 Canada; [2]Babcock and Wilcox Canada, Cambridge, Ontario N1R 5V3 Canada; [3]University of Toronto, Matls. Sci. & Eng., 184 College St., Toronto, Ontario M5S 3E4 Canada

A series of Co-based nanocomposites (Co-SiC, Co-TiO2, Co-BN, and Co-B4C) were produced by pulse current electrodeposition. The electrodeposits were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The nanocrystalline matrix structure was not adversely affected by the incorporation of micron size particulate. By controlling the plating parameters and bath chemistry, significant volume fractions of particulate could be incorporated into the nanocrystalline matrix. Mechanical properties of the nanocomposites were evaluated by microhardness testing, pin-on-disk wear tests, Taber wear tests, and bend tests. Structure-property correlations as a function of particle concentration are presented.

4:30 PM
Mechanical Properties of Nb/Zr and Nb/Ti Multilayers: *Gregory B. Thompson*[1]; Rajarshi Banerjee[1]; Arda Genc[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The mechanical properties of the sputter-deposited Nb/Zr and Nb/Ti multilayers with varying layer thicknesses have been investigated by nanoindentation. These multilayers exhibit different combinations of phases in the constituent layers, bcc Nb/hcp Zr, bcc Nb/bcc Zr, and hcp Nb/hcp Zr depending on the individual layer thicknesses. Additionally, in case of bcc Nb/bcc Zr, either a semi-coherent or a coherent interface can form. Similar combination of phases is observed in the Nb/Ti multilayers. Phase stability in these multilayers has been modeled using a classical thermodynamic approach which will be briefly discussed in this paper. Nanoindentation experiments on the Nb/Ti multilayers suggest that for similar bilayer thickness values, the hcp Nb/hcp Ti multilayers exhibit a significantly higher modulus value as compared with the bcc Nb/bcc Ti multilayers. Also for the same volume fraction of Nb in bcc Nb/bcc Ti multilayers, as the bilayer thickness reduces below a critical value, the modulus decreases substantially. The influence of layer thickness and phase stability on the mechanical properties will be presented in this paper.

4:50 PM
Deformation Mechanisms of Cryomilled Nanostructured Al Alloys: *B. Q. Han*[1]; E. J. Lavernia[1]; F. A. Mohamed[1]; [1]University of California-Irvine, Dept. of Cheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

Most recently, bulk nanostructured aluminum alloys with grain sizes of approximately 100 nm have been successfully manufactured by consolidation of cryomilled aluminum powders. In the present study, uni-axial deformation behavior was used to investigate deformation mechanisms. Characteristics of high strength and low work hardening were observed in deformation of cryomilled nanostructured aluminum alloys. To investigate deformation mechanisms, microstructure characteristics and X-ray diffraction patterns were also used in the present study. The correlation of microstructural characterization, strengthening mechanisms, plastic deformation mechanisms was discussed in terms of dislocation activity.

Magnesium Technology 2003: Magnesium Casting Properties

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, LMD-Magnesium Committee, International Magnesium Association, MPMD-Solidification Committee
Program Organizers: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

Monday PM — Room: 2
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Neal Neelameggham, US Magnesium LLC, Salt Lake City, UT 84116 USA; Allen Schultz, Hatch, Mississauga, Ontario L5K 2R7 Canada

2:00 PM
Mechanical Properties and Microstructure of Magnesium High Pressure Die-Castings: *Brandon J. Coultes*[1]; Gerry Wang[2]; J. T. Wood[1]; Richard Berkmortel[2]; [1]The University of Western Ontario, Mechl. & Matls. Eng., London, Ontario N6A 5B9 Canada; [2]Meridian Technologies, Inc., Global Tech. Ctr., 25 McNab Ave., Strathroy, Ontario N7G 4H6 Canada

Magnesium die-cast components are seeing a wider range of application in the automotive industry due to magnesium alloysí excellent castability and low density. With the increased application of magnesium high-pressure die-castings, especially for thin-wall automotive structural components, it is necessary to better understand the relationship between mechanical properties and microstructure features. In the present paper, coupon specimens with a range of characteristic microstructures were tested under tension and 4-point bending loads. The experiments described in this paper correlate the mechanical properties with microstructural and geometric features. These results provide a better understanding of how to predict the mechanical performance of die-cast components.

2:25 PM
Factors Affecting the Corrosion Behavior of Cast Magnesium Alloys: *Pei Yong Li*[1]; Hai Jun Yu[1]; Shen Chuan Chen[1]; Ying Mei Yu[1]; [1]Beijing Institute of Aeronautical Materials, Al & Mg Alloys Lab., PO Box 81-2, Beijing 100095 China

Poor corrosion resistance is one of the main causes to prevent magnesium alloys from widely applications. In this paper, the corrosion behavior of Mg-8.2Al-0.5Zn-0.3Mn (ZM-5), Mg-4.5Zn-0.7Zr (ZM-1), and Mg-2.4Nd-0.7Zr-0.4Zn (ZM-6) cast magnesium alloys were studied by salt spray corrosion tests for times up to 7 days and scanning electron microscope observations. The pure magnesium ingots for preparing these alloys were produced by Pidgeon process or electrolytic process. The results showed that, the alloys prepared using the pure magnesium ingots produced by Pidgeon process showed better corrosion resistance than those prepared using the pure magnesium ingots produced by electrolytic process. It was also confirmed that impurities, such iron etc., could accelerate the corrosion of these magnesium alloys, while alloying elements Zr and/or Nd could increase their corrosion resistance.

2:50 PM
Corrosion Behaviour of Die Cast Magnesium in ASTM B117 Salt Spray and GM9540P Cyclic Corrosion Test: *Jan Ivar Skar*[1]; Darryl L. Albright[2]; [1]Norsk Hydro ASA, Corporate Rsrch. Ctr., PO Box 2560, Porsgrunn N-3907 Norway; [2]Hydro Magnesium, Magnesium Market Dvlp., 39209 Six Mile Rd., Ste. 200, Livonia, MI 48152 USA

ASTM B117 salt spray is commonly used to test corrosion performance of die cast magnesium. The criteria for acceptable levels of impurity levels are based on salt spray test results. In the present study a two-cavity die was used to produce two side-by-side equivalent corrosion test plates. One of the plates was exposed to salt spray and the other to GM9540P. Plates of AZ91 and AM60 with a range of iron content were prepared. In salt spray, an exponential relationship between iron content and corrosion rates was found. In the GM9540P test the relationship was linear. The corrosion mechanism of magnesium is attributed to micro-galvanic corrosion between matrix and noble intermetallic particles/secondary phases. The corrosion is strongly promoted in salt spray due to a very conductive electrolyte and continuous wetting of the surface. The cyclic corrosion test uses a less conductive electrolyte and combines drying/wetting of the surface and the test becomes less aggressive. Salt spray testing leads therefore to very conservative evaluation of die cast magnesium compared to real life exposure.

3:15 PM
Process and Property Relationships in AM60B Die Castings: *Gerry G. Wang*[1]; Bryan Froese[1]; Per Bakke[2]; [1]Meridian Technologies, Inc., Global Tech. Ctr., 25 MacNab Ave., Strathroy, Ontario N7G 4H6 Canada; [2]Norsk Hydro, Magnesium Matls. Tech., Porsgrunn N-3910 Norway

As more and more magnesium metal is used, particularly for structural components, the maintenance of metal cleanliness remains of critical importance. A number of previous studies have addressed aspects of the relationship between tensile properties and the level of inclusions measured in magnesium alloys. This paper investigates the role of the size of inclusions on tensile properties on casting coupons. It is found that small size oxide inclusions play a minor role in influencing tensile properties of castings. Specifically, the results indicate that oxide contents at up to 1000 ppm levels have apparently no effect on tensile properties when most oxide particles are less than 15 microns and oxide films are less than 50 microns in size. In such cases, the tensile properties are at typical levels for AM60B die castings and might be controlled by other casting defects.

3:40 PM Break

3:55 PM
Fatigue Properties of Cast Magnesium Alloy AM60: T. A. Koch[1]; *Michael A. Gharghouri*[1]; [1]Dalhousie University, Dept. of Mining & Metallurgl. Eng., 1360 Barrington St., Rm. G212, Halifax, Nova Scotia B3J 2X4 Canada

Cast magnesium alloys are increasingly being used to manufacture structural components for the aerospace and automotive industries due to their high specific stiffness and excellent castability. Fatigue tests (R = 0.1) have been performed on as-cast magnesium alloy samples in order to study the influence of the as-cast surface on the fatigue limit. Optical and scanning electron microscopy have been used to characterize the as-cast surface as well as the grain size and precipitate distribution along the cross-section. In-situ optical microscopy on samples under load has been used to observe surface crack initiation and propagation. In addition, post-mortem SEM analyses of fracture surfaces have been performed in order to identify crack initiation sites and to characterize crack propagation paths.

4:20 PM
Enhancement of the Properties of Mg-Li Alloys by Small Alloying Additions: Guang Sheng Song[1]; Mark Staiger[1]; *Milo V. Kral*[1]; [1]University of Canterbury, Matls. Eng. Grp., Dept. of Mechl. Eng., Christchurch New Zealand

The addition of lithium to magnesium, with a relative density of 0.53:1.0, can reduce the alloy density significantly. Mg-Li alloys thus have potential to be the lightest structural metallic materials available. Furthermore, the addition of at least 11 wt% Li can convert the hexagonal structure of pure Mg to a BCC structure, markedly improving formability. However, Mg-Li alloys are well known to present difficulties in conventional casting due to serious oxidation or burning during melting process. Also, improving the specific strength would have obvious advantages. It has been shown that the addition of certain other elements to the alloy can improve both oxidation resistance and mechanical properties. This paper presents a comparison of the oxidation behavior and mechanical properties of Mg-12Li alloys with additional elements, such as Be and Ca. The improvements noted will be related to surface chemistry and microstructural differences through observations by Auger Electron Spectroscopy, optical microscopy and transmission electron microscopy.

4:55 PM
Parametric Study of Laser Cladding of AS 21 Magnesium Alloy with Aluminium Silicon/Tungsten Carbide Powder: *Meity Carolina Mandagie*[1]; Milan Brandt[1]; Yvonne Claire Durandet[1]; Mahnaz Jahedi[2]; [1]Swinburne University of Technology, IRIS, PO Box 218 Hawthorn, Melbourne, Victoria 3122 Australia; [2]CSIRO, Mfg. Sci. & Tech., Cnr. Raglan & Albert St., Preston, Melbourne, Victoria 3072 Australia

Magnesium alloys are of growing interest for a number of applications in the automotive industry. An impediment to their increased usage is the low resistance to both wear and corrosion compared to steel or aluminium due to its low hardness and chemical affinity for numerous elements respectively. This paper describes the results of experiments investigating Nd:YAG laser cladding of AS 21 magnesium alloy with a mixture of Aluminium Silicon (40% wt) and Tungsten Carbide (60% wt) powder to improve its wear properties. The effects of laser power, scan speed, powder feed rate and shielding gas on the clad layer thickness and its hardness were examined. The result indicates that for the processing speed of 200 mm/min and powder feed rate of 7.5 g/min, the thickness of the clad layer decreased from 1.7 mm to 0.3 mm when the laser power was reduced from 1.6 kW to 1.4 kW. That changing of the parameter also produced less porosity in the clad layer, while the quantities of microcracks remained the same. When the proce ssing speed was decreased from 300 mm/min to 200 mm/min at 7.5 g/min and 1.4 kW, the layer thickness increased from 0.8 mm to 1.7 mm. The clad layer produced has few microcracks. The hardness of the clad layer was up to 145 HV which was at least a factor of two greater than that of the AS 21 substrate (54 HV).

5:20 PM
Surface Finishing of Aluminium and Magnesium Alloys Using Plasma Electrolytic Oxidation (PEO): *Frank C. Walsh*[1]; Keith T. Stevens[2]; Colin G. John[2]; [1]University of Bath, Cheml. Eng., Claverton Down, Bath BA2 7AY UK; [2]Poeton Industries, Ltd., Eastern Ave., Gloucester GL4 3DN UK

Plasma Electrolytic Oxidation (PEO) is a recently introduced surface finishing technology which is capable of producing a range of hard, dense oxide coatings on magnesium, aluminium, titanium and other light alloys. This review highlights the development of practical PEO coatings for a wide range of industrial sectors using data from our laboratories and industrial production facilities. The following aspects of PEO coating are considered: (a) the technology used to produce PEO coatings (electrochemical aspects); (b) the thickness and morphology of the surface coating (optical and scanning electron microscopy imaging); (c) the physical properties of the oxide film (e.g., hardness and tribology); (d) the chemical properties of the coating (composition and corrosion resistance). The importance of controlled process conditions, selected to suit a particular alloy composition and metallurgy, is highlighted. Present industrial applications are considered together with their coating requirements. While PEO coating is a specialised process, it is seen to be capable of meeting the need for controlled appearance, hardness, wear resistance, frictional properties and corrosion resistance for an expanding range of industrial sectors. The paper concludes with a forward look at the exciting possibilities offered by modifications to the PEO process to produce next generation coatings on magnesium alloys and other light metals.

Martensitic Transformations in Low Symmetry Materials - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS), MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Robert D. Field, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Richard W. Fonda, Naval Research Laboratory, Washington, DC 20375 USA

Monday PM — Room: 7B
March 3, 2003 — Location: San Diego Convention Center

Session Chair: Richard W. Fonda, Naval Research Laboratory, Washington, DC 20375 USA

2:00 PM Invited
Shock-Induced Martensitic Phase Transformations in Low-Symmetry Materials: *G. T. Gray*[1]; P. A. Rigg[1]; R. S. Hixson[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

The study of the physical properties of ductile solids subjected to shock wave loading strives to understand how the thermodynamic conditions and strain rate affect material response.†The severe loading path conditions imposed during a shock induce a high density of defects in most materials, i.e., dislocations, point defects, and/or deformation twins.† During the shock process some materials may also undergo a pressure-induced-phase martensitic transition that will affect the material response.† In this talk details of the shock-loading response of Ti, Zr, U-6Nb, and NiTi will be presented.†Work conducted under the auspices of the US Department of Energy.

2:35 PM
Modeling and In Situ Observations of Stress Induced Transformation in Shape Memory Alloys: *Cate Brinson*[1]; Debbie Burton[1]; Xiujie Gao[1]; [1]Northwestern University, Mechl. Eng. Dept., 2145 Sheridan Rd., Evanston, IL 60208 USA

This talk will discuss both micromechanical experiments and associated modeling techniques for shape memory alloys. In the experiments, in situ optical microscopy allowed observation of the microstructural evolution during loading. Features of geometrical banding as well as stress-strain response are explained through the behavior of the variants seen. A micromechanics based Multivariant Model is developed for both single and polycrystalline SMA response. This model provides both macroscopic stress-strain curve prediction and identification of the active variants under any thermomechanical loading. The model is based on the habit plane and transformation directions for the variants of martensite in a given material. The single crystal behavior to temperature and mechanical loads is derived using the concepts of a thermodynamic driving force. The polycrystalline model uses a self-consistent approach to account for the transformation of a material with grains of parent phase in random orientation. Relation to plasticity-based mechanics models will be discussed.

3:10 PM
The Crystallography of Transformation and Deformation Twins in U-Nb: *Robert D. Field*[1]; Dan J. Thoma[1]; Paul S. Dunn[1]; Don W. Brown[2]; Carl M. Cady[3]; [1]Los Alamos National Laboratory, MST-6, G770, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-8, H805, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, MST-8, G755, Los Alamos, NM 87545 USA

The presentation will summarize an investigation of the martensitic transformation and subsequent deformation of a U-Nb shape memory alloy, with an emphasis on the crystallography of twinning in this system, as revealed through TEM studies. First, the transformation will be presented. The self-accommodating twin structures will be considered in terms of the lattice parameters of the martensitic phases, including previously proposed intermediate transformations. Second, preliminary results on the active deformation twinning systems identified for the U-5.5wt.%Nb shape memory alloy will be presented. The discussion will center on the effect of the small monoclinic distortion associated with the alphaî structure on the choice of twinning systems, as well as the mechanisms of deformation twin propagation through the pre-existing transformation twins in the microstructure.

3:30 PM Break

3:45 PM
Diffraction Measurements of Phase Fraction, Texture and Strain Evolution During Stress-Induced Martensitic Transformations: *Raj Vaidyanathan*[1]; C. R. Rathod[1]; Mark A.M. Bourke[2]; D. C. Dunand[3]; [1]University of Central Florida, AMPAC/MMAE, Engr.-I Rm. 381, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA; [2]Los Alamos National Laboratory, MST-8, MS H805, Los Alamos, NM 87545 USA; [3]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

NiTi can exhibit superelastic behavior by undergoing a reversible stress-induced martensitic transformation from a cubic austenite phase to a monoclinic martensite phase. In this work we report on in situ neutron (at Los Alamos Neutron Science Center) and synchrotron X-ray (at the Advanced Photon Source) diffraction measurements during loading in superelastic NiTi. The experiments relate macroscopic strain (from an extensometer placed on the sample) with the texture, phase volume fraction and strain evolution (from diffraction spectra). Emphasis is placed on comparing this evolution in textured, wire samples used in the synchrotron X-ray diffraction work with random, hot isostatically pressed (HIP) samples used in the neutron diffraction work.

4:05 PM
The Role of Precipitates and Defects at Parent-Martensite Boundaries in the Phase Transformation Behavior of NiTi: *Amy Jaye Wagoner Johnson*[1]; Huseyin Sehitoglu[1]; Hans J. Maier[2]; Kenneth A. Gall[3]; [1]University of Illinois at Urbana-Champaign, Dept. of Mechl. & Industl. Eng., 140 Mechl. Eng. Bldg., MC-244, 1206 W. Green St., Urbana, IL 61801 USA; [2]University of Paderborn, Lehrstuhl f. Werkstoffkunde, Paderborn D-33095 Germany; [3]University of Colorado, Dept. of Mechl. Eng., 427 UCB, Boulder, CO 80309-0427 USA

NiTi Shape Memory Alloys are used in applications ranging from actuators to biomedical devices. Their shape memory and pseudoelastic properties vary significantly over a short range of compositions, with texture/orientation, and with precipitate size. While theory predicts fairly accurately the behavior of solutionized NiTi, it is less accurate in the presence of precipitates and defects. In this work, we examine the role of precipitates and defects generated at the parent-martensite interface in the phase transformation behavior of a single crystal NiTi alloy using in-situ straining and traditional TEM. Preliminary results show that martensite preferentially nucleates in the strain field of the coherent precipitate. The observation is consistent with theoretical predictions and observations by other research groups. In addition, emissary dislocations are observed at parent-martensite boundaries. Whether the boundary is pinned resulting in dislocation emission or whether the dislocations are generated during the twinning/detwinning process is not yet understood.

4:25 PM
Influence of Local Solute Ordering and Plasticity on the Martensitic Transformation Kinetics of Plutonium Alloys: *Christopher R. Krenn*[1]; Babak Sadigh[1]; Adam J. Schwartz[1]; Wilhelm G. Wolfer[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci. Direct., PO Box 808, L-353, Livermore, CA 94551 USA

On cooling, Ga-stabilized plutonium alloys transform from an fcc phase (δ) to a monoclinic phase (α) with a 20% smaller atomic volume. The large volume change is believed to induce significant plastic deformation. Using ab-initio techniques, we have identified both a kinetically stable strained and disordered alloy of Ga in α and a more stable ordered alloy with a smaller atomic volume. Since Ga is randomly distributed in the δ-phase, rapid quenching will produce the disordered a phase with significant elastic strain energy. Slow cooling may produce the ordered a phase. We discuss the impact of this ordering and of the volume-change induced plasticity on experimental observations of ìtemperedî two-phase material and of an unusual double-c transformation kinetics. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

Materials and Processes for Submicron Technologies - III: Characterization, Measurement, Modeling of Micro- and Nano-Scale Thin Films and Devices

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee, TMS
Program Organizers: Seung H. Kang, Agere Systems, Device and Module R&D, Allentown, PA 18109 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ USA; Mahesh Sanganeria, Novellus Systems, Inc., San Jose, CA 95134 USA

Monday PM — Room: 15A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Seung H. Kang, Agere Systems, Device & Module R&D, Allentown, PA USA; Choong-Un Kim, University of Texas, Dept. of Matls. Sci. & Eng., Arlington, TX 76019 USA

2:00 PM Invited
Revealing Deformation Mechanisms Through In Situ Nanoindentation in a Transmission Electron Microscope: *Andrew M. Minor*[1]; Erica T. Lilleodden[2]; Eric A. Stach[3]; John W. Morris[1]; [1]University of California at Berkeley, Dept. of Matls. Sci. & Eng., Matls. Sci. Div., Lawrence Berkeley Natl. Lab., 1 Cyclotron Rd., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Matls. Sci. Div., 1 Cyclotron Rd., Berkeley, CA 94720 USA; [3]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, 1 Cyclotron Rd., Berkeley, CA 94720 USA

The deformation mechanisms of Al and Si have been investigated using the novel experimental technique of in situ nanoindentation in a transmission electron microscope. Direct evidence from quantitative in situ nanoindentation will show that anomalous load-displacement measurements during the nanoindentation of Al are due to the nucleation of dislocations. The deformation mechanisms in silicon will be discussed in terms of the competition between dislocation nucleation and phase transformation during the initial stages of indentation. Additionally, it will be shown that the residual tensile fields of a plastically deformed volume in silicon can lead to non-crystallographic fracture during unloading.

2:30 PM
Mechanical Characterization of Low-K Materials Using Nanoindentation: Richard J. Nay[1]; Tony M. Anderson[1]; [1]Hysitron, Nanomech. Rsrch. Lab., 5251 W. 73rd St., Minneapolis, MN 55439 USA

Improvements in the dielectric constant of low-k materials play an important role in reducing gate delays in modern microelectronics. However, these improvements are often achieved through increases in porosity or usage of materials with low polarizability, which can reduce the overall mechanical strength of the material. This creates difficulties with further fabrication processes such as CMP. Therefore, the mechanical properties of low-k materials must be considered when evaluating the materials overall performance. Measuring the mechanical properties of these SOD and CVD films is made challenging by the fact that they have sub-micron thickness. The nanoindentation testing technique is uniquely suited for performing these measurements. Presented here is the mechanical analysis of three such low-k films using nanoindentation. All of these films are on a silicon substrate. Details of the testing methods and equipment are also discussed.

2:50 PM
Thermo-Mechanical Response of Passivated Copper Thin Films: *Y.-L. Shen*[1]; [1]University of New Mexico, Dept. of Mechl. Eng., Albuquerque, NM 87131 USA

The highly anisotropic nature and unique plastic yielding behavior of copper warrant a detailed thermo-mechanical study of copper thin films used as an interconnect material. In this work we focus on the thermal mismatch induced deformation of passivated copper films. Stresses in copper films of thicknesses 400 nm, 250 nm and 125 nm, passivated with silicon oxide on a quartz substrate, were measured using the curvature method. The thermal cycling spans a temperature range from -196 to 450C. It is seen that the strong relaxation at high temperatures normally found in unpassivated films is nonexistent for passivated films. Further analyses showed that significant strain hardening exists during the course of thermal loading. In particular, the measured stress-temperature response can only be fitted with a kinematically hardening model, if a simple constitutive law within the continuum plasticity framework is to be used. This is drastically different from the unpassivated and passivated aluminum films and unpassivated copper films. The possible micromechanisms responsible for this unique feature will be discussed. Implications to stress modeling of copper interconnects in actual devices, along with numerical modeling using the finite element method, will be presented.

3:10 PM Invited
Modeling Interface Instability of IC Interconnects Under Temperature and Current Stress: *Jun-Ho Choy*[1]; K. L. Kavanagh[1]; [1]Simon Fraser University, Dept. of Physics, Burnaby, BC V5A 1S6 Canada

Electromigration studies on IC copper metallization reveal that the failure mechanism is controlled by the copper/capping dielectric interface due to its relatively poor quality. The objective of the present study is to model the interface stability of copper wire when both electromigration and surface diffusion are active in atomic transport, under the condition that the adhesion between copper and dielectric is poor. Based on the 3 dimensional finite difference numerical scheme, the model provides the stability criteria in the non-linear regime where the transport driven by electromigraion may compete with surface diffusion.

3:40 PM Break

4:00 PM Invited
Electromigration Reliabilty in Ultra-Fine Cu Interconnects: *Nancy L. Michael*[1]; Choong-Un Kim[1]; Paul Gillespie[2]; Rod Augur[2]; [1]The University of Texas at Arlington, Matls. Sci. & Eng., Arlington, TX 76019 USA; [2]International SEMATECH, Austin, TX 78741 USA

Several studies on electromigration induced failure in Cu interconnects have suggested that interface electromigration takes place. Less well understood is how interface electromigration participates in the failure mechanism in narrow lines with near-bamboo microstructures. In this study, electromigration failure of damascene Cu interconnects has been studied using single level lines with widths between 80nm and 700nm. Lifetime shows little dependence on barrier material (Ta, TaN, or Ta/TaN) or linewidth to grain size ratio. Lifetime shows a stronger dependence on the ratio of perimeter to area, indicating the importance of the interface in the failure mechanism. Failure analysis indicates that significant interface electromigration is taking place but that localized voids, rather than those that spread rapidly along the interface, lead to fatal damage. Further study using electro-thermal fatigue indicates that the fatal damage may begin at microstructural defects or impurities resulting from standard manufacturing processes.

4:30 PM Invited
A Model for the Evolution of Annealing Textures in Interconnects: *Dong Nyung Lee*[1]; [1]Seoul National University, Sch. of Matls. Sci. & Eng., Seoul 151-744 Korea

The annealing textures of aluminum and copper interconnects depend on their deposition textures and geometries. Attempts have been made to explain the phenomena based on the surface, grain boundary, and interface energies. In this article, a new model is advanced to account for the annealing textures of aluminum or copper interconnects based on their thermal strain energies. The interconnects, whether they are conventionally fabricated or damascene process fabricated, are subjected to thermal stresses during annealing, which in turn give rise to strain energies. The strain energy of a deposit is influenced by its texture and geometry. The annealing texture of an interconnect line is determined such that its thermal strain energy is minimal. The minimum strain energy of the deposit can be achieved when the absolute maximum thermal stress direction is parallel to the minimum Youngís modulus direction of the material, whereby the strain energy release can be maximized.

5:00 PM Invited
Surface Roughness and Spectroscopic Ellipsometry Measurements on TaSi2/Si: *Steve Bromberg*[1]; N. M. Ravindra[2]; [1]Accurion, 935 Hamilton Ave., Menlo Park, CA 94025 USA; [2]New Jersey Institute of Technology, Dept. of Physics, Newark, NJ 07102 USA

Abstract not available.

Materials Processing Under the Influence of Electrical and Magnetic Fields - II

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee

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Monday PM — Room: 14A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Charalambos Doumanidis, National Science Foundation USA; Antonios Zavaliangos, Drexel University, Dept. of Matls. Eng., Philadelphia, PA 19104 USA

2:00 PM Invited
Temperature and Electric Current Distribution in FAST: *A. Zavaliangos*[1]; [1]Drexel University, Dept. of Matls. Eng., 3141 Chestnut St., Philadelphia, PA 19104 USA

In this paper we present macroscopic coupled thermoelectric models that provide insight into the temperature and current distribution in a punch/die/specimen system.†We focus our attention on: (a) the relative importance of joule heating versus heat conduction in the various parts of the assembly, (b) the importance of the dimensions of the die and punches, (c) the ratio of electrical conductivities of the sintering materials versus graphite and (d) the routes of heating losses namely the heat conduction.

2:30 PM Invited
The Diffusion-Oriented Processes Enhanced by Spark Plasma Sintering: *Mats Nygren*[1]; Zhijian Shen[1]; [1]Stockholm University, Dept. of Inorganic Chem., Arrhenius Lab., Stockholm SE-106 91 Sweden

Spark plasma sintering (SPS) and the related techniques have been successfully applied to synthesis, consolidate, and join materials covering a wide range of materials, e.g. ceramics, composites, cermets, metals and alloys. A common feature of the SPS process is that it uses a pulsed DC-current to heat the sample and that it enables reaction and consolidation to occur at comparatively low temperatures in a rapid manner. The enhanced kinetics of compaction and reaction have been ascribed to the use of rapid heating, an efficient heat transfer, and the application of comparatively high uniaxial pressures while the contribution of the external electrical field generated by the pulsed DC-current to the enhanced kinetics has not been discussed in any greater detail. In this presentation we will shown experimental evidences that disclose the impact of an electrical field on the diffusion-oriented processes, e.g. diffusion-controlled reactions in the synthesis of a series of novel complex oxides and oxynitrides, den sification of ceramics that strongly relies on the grain-boundary diffusion, diffusion and reaction controlled grain growth processes, etc.

3:00 PM

Temperature Evolution During Field Activated Sintering: *Martin Kraemer*[1]; Jing Zhang[2]; Groza R. Joanna[1]; Antonios Zavaliangos[2]; [1]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616 USA; [2]Drexel University, Matls. Eng. Dept., 3141 Chestnut St., Philadelphia, PA 19104 USA

Field activated sintering (FAST), or Spark Plasma Sintering (SPS), has gained growing attention over the last decade as an effective tool for enhancing sintering kinetics and reducing sintering temperatures. The technique combines hot-pressing with passing a pulsed electric current through a graphite die set. In order to gain a fundamental understanding of the underlying factors critical to FAST the temperature evolution and Joule heat generation within the graphite die set and specimen materials has been studied by thermocouple/pyrometer measurements and finite element modeling. The results show that significant differences develop between the temperatures the specimen experiences and the temperatures typically measured and quoted. A bipolar temperature field exists within the specimen volume. It depends on the overall geometry, temperature, and sample electric conductivity. The field can be described by a set of 5 characteristic temperatures and the averaged gradient between maximum and minimum temperature. The results are discussed in terms of Joule heat generation and heat transfer by thermal conductivity and radiation, allowing to establish a concept for improving experimental conditions in order to achieve better temperature uniformity during FAST sintering. Acknowledgement: This work was sponsored by NSF and a Penn State grant.

3:20 PM Invited

Effects of an Electric Field or Current on the Flow Stress of Metals and Ceramics: *Hans Conrad*[1]; [1]North Carolina State University, Matls. Sci. & Eng., Raleigh, NC 27695-7907 USA

The influence of an electric field or current on the plastic deformation of metals and ceramics is reviewed. Regarding ceramics, an electric field enhanced dislocation mobility and promoted cross slip in halides. Further, a field reduced appreciably, the flow stress of fine-grained oxides at high temperatures. Regarding metals, an electrostatic field either reduced or increased the flow stress, depending on polarity and specific metal. Further, high density electropulsing enhanced the mobility of dislocations and reduced the flow stress. The mechanisms by which the field or current produced the observed effects are discussed.

3:40 PM Break

3:55 PM

Plasma Pressure Compaction of Tungsten Powders: *K. C. Cho*[1]; R. J. Dowding[1]; B. R. Klotz[1]; R. H. Woodman[1]; [1]US Army Research Laboratory, Aberdeen Proving Ground, MD 21005-5069 USA

Compacts of tungsten powders were consolidated by Plasma Pressure Compaction (P^2C), an electric discharge technique. The powders were a variety of commercially available grades ranging in particle size from submicron to 12 microns. Following consolidation, the density of the compacts was measured, and the microstructure examined. Results revealed the effect of powder size, pulsing treatment, final hold temperature, and applied pressure on final part density and microstructure development. Most important to the purpose of the study, it was found that the short cycle time of P^2C did not suppress grain growth in the compacts of submicron powder. Thus, grain growth remained a consequence of full densification. Implications of these results for the development of nanograined microstructures using P^2C are discussed.

4:25 PM

Field Assisted Sintering for Ceramic Processing: *Lia A. Stanciu*[1]; Vladimir Y. Kodash[1]; Maria Zaharescu[3]; Adrian Jianu[2]; Joanna R. Groza[1]; Andrei Jitianu[3]; [1]University of California-Davis, Eng. Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Rostok University, Physics Dept., August-Bebel-Str-55, Rostok D-18051 Germany; [3]Institute of Physical Chemistry, 7 Splaiul Independetei, Bucharest 77208 Romania; [3]Institute of Physical Chemistry, 7 Splaiul Independentei, Bucharest 77208 Romania

An electrical field assisted sintering technique (FAST) has been applied on alumina-titania systems. The precursor powders have been obtained by a sol-gel method. In the alumina-titania system, the reaction to form aluminum titanate started at 1050°C. The structural evolution of the oxides with temperature has been monitored from the initial amorphous state of the precursors, up to 1300°C. The investigation techniques used were: FTIR, XRD, Transmission Electron Microscopy, High Resolution Electron Microscopy.

4:45 PM

Enhancement of Texture and Critical Current Density of Bi2212 Superconducting Tapes Heat Treated in High Magnetic Fields: *Sastry V. Pamidi*[1]; Ulf P. Trociewitz[1]; Hiroshi Maeda[1]; Justin Schwartz[1]; [1]Florida State University Center for Advanced Power Systems, Natl. High Magnetic Field Lab., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA

Improvements in texture and critical current density have been reported in high-Tc superconductors by heat-treating in magnetic fields. Magnetic field creates a torque on Bi2212 crystallites because of the anisotropy in magnetic moment, causing the grains to rotate with respect to the magnetic field. We have designed, built, and tested a furnace for the 20 T resistive magnet at the National High Magnetic Field Laboratory. Our experiments are focused on understanding the effect of magnetic field strength and the duration of the magnetic field during heat treatment on the microstructure and critical current density of Bi2212 tapes. Heat treatments were conducted with magnetic field of up to 15 T applied for 1 to 8 hours. Similar experiments were also conducted on Hg1212 and Hg1223 superconducting films. Some of the Bi2212 tape samples with dimensions of 0.5 mm X 3 mm showed critical current values of more than 1200 A when heat treated in a field of 10 T. Results of microstructure and superconducting properties are presented.

5:05 PM

Economic Comparison of Ferrite and NdFeB Magnets for Automotive Applications: *Darth E.M. May*[1]; Jacqueline A. Isaacs[1]; [1]Northeastern University, Mechl., Industl. & Mfg. Eng., 334 SN, 360 Huntington Ave., Boston, MA 02115 USA

With the increasing use of magnetic materials in automobiles - incorporating as many as 100 electric motors, the search for economically superior alternatives to ferrites is becoming vital. Size reductions in these motors will result in curbweight reductions that lead to improved fuel economy. NdFeB magnets are attractive as potential replacement materials, due to their higher flux density and thus smaller required size, however high material costs have limited their implementation. To investigate and compare magnetic materials, technical cost models (TCMs) were developed and used to examine the manufacturing economics related to the production of both NdFeB and ferrite magnets. Results from the TCMs are used to identify the cost drivers and opportunities for cost reductions. Further investigations include effects on vehicle fuel efficiency, and the environmental impact of magnet manufacture and disposal.

Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction: Role of Probabilistics in Prognosis

Sponsored by: Structural Materials Division,

Program Organizers: James M. Larsen, US Air Force, Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA; Leo Christodoulou, Defense Advanced Research Agency, Arlington, VA 22203-1714 USA; William J. Hardman, Naval Air Systems Command, Propulsion and Power Directorate (AIR 4.4.2), Patuxent River, MD 20670-1534 USA; Andrew J. Hess, Naval Air Systems Command, Propulsion and Power Directorate, Patuxent River, MD 20670-1534 USA; J. Wayne Jones, University of Michigan, College of Engineering, Department of Materials, Science and Engineering, Ann Arbor, MI 48109 USA; Stephan M. Russ, Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA

Monday PM — Room: 16A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Theodore G. Fecke, Air Force Research Laboratory, AFRL/PRTC, WPAFB, OH 45433-7251 USA; Michael J. Caton, US Air Force, AFRL/MLLMN, WPAFB, OH 45433-7817 USA

2:00 PM Invited

Materials Aging, Prognostics, and Life Cycle Engineering and Management: *Robert P. Wei*[1]; D. Gary Harlow[1]; [1]Lehigh University, Mechl. Eng. & Mech., 327 Sinclair Lab., 7 Asa Dr., Bethlehem, PA 18015 USA

In this paper, a simplified framework for prognostics is presented and is considered in terms of materials aging and in relation to the overall processes for the life cycle engineering and management (LCEM) of engineered systems. The need for a science-based probability approach for modeling the evolution and distribution of damage, and for a transformation and holistic integration of the processes for LCEM is discussed. The approach is illustrated through the development of mechanistic understanding and modeling of corrosion and corrosion fatigue of aluminum alloys. Its efficacy is demonstrated through comparisons between model predictions and observations on commercial and military aircraft that have experienced long-term service.

2:30 PM Invited
Fatigue Prognostics Using Microstructurally-Based Total Life Models: *Robert G. Tryon*[1]; Amimesh Dey[1]; [1]VEXTEC, 116 Wilson Pike, Ste. 230, Brentwood, TN 37027 USA

Technology for extending the life of fielded gas turbine engines offers the potential for signification savings in total ownership costs. On-board monitoring and analysis offers the potential to instill increased confidence in prediction of fatigue or remaining useful life on an individual system basis. This presentation focuses on quantitative models to relate information from on-board sensors to fatigue damage accumulation. Real time speed and temperature data is used with fluid dynamic, heat transfer and structural models to predict stresses and strains at remote locations. Dislocation theory is used to predict damage accumulations at the microstructural level. Energy based models are used to predict nucleation of cracks and their progression thought the short and long crack growth phase. System reliability methods are used throughout to account for multiple failure modes and uncertainty in the inputs to the various models. The methodology is used to predict of the probability of exceeding a certain crack size for an individual aircraft.

2:55 PM Invited
Physically-Based Life-Prediction Models for Applications in Engine Integrity Prognostics: *Kwai S. Chan*[1]; [1]Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA

This paper focuses on issues encountered during the process of extending a set of microstructure-based life-prediction models for possible applications in a prognostic setting that requires making real-time forecast based on on-broad sensor measurements. A brief summary of current microstructure-based fatigue models for treating crack initiation and propagation is presented first. Modifications required for extending the fatigue models to incorporate material variability are identified. A framework for evolving the microstructure-based fatigue models into physically-based prognostic tools for assessing engine integrity is discussed. Model simulation will be presented to illustrate the concept and the underlying physical processes. The feasibility of the proposed approach will be evaluated based on model simulations for simple, idealized loading conditions. Work supported by AFOSR through Contract No. F49620-01-1-0547, Dr. Craig S. Hartley, Program Manager.

3:20 PM Break

3:50 PM Invited
The Role of Statistical Variability in Fatigue Crack Growth Rates in Materials Prognosis: *Michael J. Caton*[1]; Andrew H. Rosenberger[1]; James M. Larsen[1]; [1]US Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLMN, WPAFB, OH 45433-7817 USA

A key element of a material prognosis system is the ability to predict the variability in fatigue crack growth rates for given material systems and loading conditions. Perhaps the most important factor influencing the variability in crack growth behavior is the inherent variability in material microstructure. This study examines the influence of grain size variations on the elevated temperature fatigue crack growth rates in Waspaloy. High-precision experimental methods were used to monitor crack growth rates for several temperatures, loading frequencies, and dwell periods. Thorough microstructural characterization of the test specimens reveals the role of microstructure on the variability in crack growth behavior and provides valuable insights into the mechanisms controlling fatigue crack growth. The results of this study are presented within the context of the historical database available in the literature, and the implications for enhancing the physically-based models incorporated into materials prognosis systems are discussed.

4:15 PM Invited
The Use of Ultrasonic Fatigue in the Modeling of Very Long Fatigue Life: *J. Wayne Jones*[1]; Michael J. Caton[2]; [1]University of Michigan, Matls. Sci. & Eng., 2018 H. H. Dow, 2300 Hayward Ave., Ann Arbor, MI 48109 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLMN, WPAFB, OH 45433-7817 USA

In recent years considerable progress has been made in modeling fatigue crack initiation and growth as a means to better predict the accumulation of fatigue damage and residual fatigue lifetime in advanced structural materials. However, at very long lifetimes where crack initiation and the growth of short cracks dominate fatigue life and where microstructure effects are most pronounced progress has been limited. Little statistically meaningful data on fatigue behavior and fatigue life exists in this regime because the fatigue experiments necessary to acquire this data are especially difficult, time-consuming and expensive to conduct. Recent advances in ultrasonic fatigue methodologies offer the potential for this technique to be used as an effective tool for rapid assessment of fatigue behavior in structural alloys. Ultrasonic fatigue is used to examine the very long life fatigue behavior of a cast Al-Si-Cu alloy and extension of the approach to assess the role of microstructural variability in probabilistic fatigue life prediction is presented.

4:40 PM
Fatigue Bahavior of Al-Si-Cu Aluminum Castings: *Qigui Wang*[1]; Peggy Jones[1]; [1]General Motors Corporation, CDVC-Powertrain, 1629 N. Washington Ave., Saginaw, MI 48605 USA

The influence of casting discontinuities and microstructure on the fatigue behavior of lost foam cast 319 aluminum alloys has been investigated under the over aged heat treatment condition. Fatigue performance of the 319-T7 castings is dominated mainly by porosity and slightly by intermetallic particles. Eutectic silicon modification decreases fatigue strength in the studied alloys due to the increased shrinkage porosity and segregation of Cu-rich intermetallic particles. Addition of the Al dendrite grain refiner is not beneficial to fatigue. Fatigue behavior of the Al-Si-Cu cast alloys can be predicted using fracture mechanics models together with the estimated extreme discontinuity size in castings by Extreme-Value Statistics (EVS).

Measurement and Interpretation of Internal/ Residual Stresses: Deformation Modes Part II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Shaping and Forming Committee
Program Organizers: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Bimal K. Kad, University of California, Ames Laboratory, La Jolla, CA 92093-0085 USA

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March 3, 2003　　Location: San Diego Convention Center

Session Chairs: Bimal Kad, University of California, Ames Lab., La Jolla, CA 92093-0085 USA; Sean R. Agnew, University of Virginia, Matls. Sci. & Eng., Charlottesville, VA 22904-4745 USA

2:00 PM Invited
Polycrystal Modeling of Residual Strains at Large Plastic Strains: *Carlos N. Tome*[1]; Ricardo A. Lebensohn[2]; Mark R. Daymond[3]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-8 MS G755, Los Alamos, NM 87545 USA; [2]Universidad Nacional de Rosario, Physics Dept., Pellegrini 250, Rosario, Santa Fe 2000 Argentina; [3]Rutherford Appleton Laboratory, ISIS, Chilton, Didcot, Oxon OX11 0QX UK

Large strain polycrystal models (such as the Visco-Plastic Self-Consistent model, VPSC) are traditionally used for describing texture evolution and hardening during plastic forming. A limitation of these models is that they only address the plastic component while neglecting the elastic deformation component. Elasto-Plastic Self-Consistent (EPSC) polycrystal models, on the other hand, account for the elastic and the plastic strains in each grain. They permit us to simulate the evolution of internal strains during loading and unloading, but are only applicable to small strains (up to about 4%). In this work we present an extension of the VPSC model which permits us to calculate, in an approximate way, the full Cauchy stress tensor in the grains. We apply this formalism to study an austenitic stainless steel cold rolled to 70% reduction. Tensile tests to 1% strain are simulated in the as-received and the cold-rolled material, and the evolution of internal strain is predicted. A comparison is made with internal strains measured by neutron diffraction, and the validity of the approach is assessed.

2:30 PM Invited
Internal Stress Measurements in Shape-Memory Alloys: *Raj Vaidyanathan*[1]; [1]University of Central Florida, AMPAC/MMAE, Engr.-I Rm. 381, 4000 Central Florida Blvd., Orlando, FL 32816-2455 USA

Several investigations have reported internal stress measurements in materials where initial elastic deformation is followed by slip associated with dislocation plasticity. However, this is not the case with alternative deformation mechanisms such as twinning and stress-induced transformations. In NiTi, a cubic phase can undergo a reversible stress-induced transformation to a monoclinic phase, i.e., exhibit superelasticity. The monoclinic phase can deform by twinning and subsequently undergo a thermally-induced transformation to the cubic phase, i.e., exhibit the shape-memory effect. In this work, an overview is provided of in situ neutron and synchrotron X-ray diffraction measurements during loading in both, shape-memory and superelastic NiTi, with the objective of following deformation twinning and stress-induced transformations. The experiments were performed at the Los Alamos Neutron Science Center and the Advanced Photon Source. A methodology to analyze such diffraction spectra is established and quantit ative measurements of the texture, phase volume fraction and strain evolution are reported.

3:00 PM

Yield Strength and Strain Hardening Behaviour of Ferrite and Austenite in TRIP-Assisted Multiphase Steels: Neutron Diffraction Measurements Coupled with Secant Mean Field Modelling: Pascal J. Jacques[1]; Quentin Furnemont[1]; FrÈdÈric Lani[1]; Kevin T. Konlon[2]; Gauthier Lacroix[1]; *Francis Delannay*[1]; [1]UniversitÈ Catholique de Louvain, PCIM, Place Sainte Barbe 2, Louvain-la-Neuve B-1348 Belgium; [2]CNRC, Chalk River Canada

TRIP-assisted multiphase steels are a new generation of low carbon ferritic steels exhibiting enhanced combination of strength and ductility thanks to the presence of retained austenite able to transform into martensite under straining. The microstructure may associate up to 4 different phases: intercritical ferrite, retained austenite, bainite, and martensite. Neutron diffraction experiments during tensile tests were used for measuring the stress partition between ferrite and austenite in several steels processed in such a way as to provide different stabilities of retained austenite. Conversely, the strain partition between the phases was measured by image analysis of SEM micrographs recorded during in situ tensile tests. The plastic flow laws of the two phases derived from these measurements were used as input parameters for modelling the overall plastic behaviour of the steels using the equivalent inclusion mean field method.

3:20 PM

Lattice Plane Response During Uniaxial Loading of ECAP Materials: *Sven C. Vogel*[1]; David J. Alexander[1]; Irene J. Beyerlein[1]; Mark A.M. Bourke[1]; Bjorn Clausen[2]; Cheng Xu[3]; Terence G. Langdon[3]; [1]Los Alamos National Laboratory, MST-8, MS H805, PO Box 1663, Los Alamos, NM 87545 USA; [2]California Institute of Technology, 313 Keck Eng. Lab., MC 138-78, 1200 E. California Blvd., Pasadena, CA 91125 USA; [3]University of Southern California, Dept. of Aeros. & Mechl. Eng., Los Angeles, CA 90089-1453 USA

The results of a neutron diffraction study of equal-channel angular pressed pure metals (nickel, aluminum, beryllium) are presented. The lattice strain response was investigated by performing uniaxial loading measurements up to 10% plastic deformation in-situ on the neutron time-of-flight powder diffractometer SMARTS. This technique yields the strain response of several lattice planes oriented parallel and perpendicular to the loading direction simultaneously. The results are compared to those from equivalent in-situ loading experiments with unprocessed materials. Additionally, the bulk-averaged residual strains introduced by the ECAP processing were measured using neutron diffraction. Bulk sample texture was also investigated using neutron diffraction. The use of neutrons provides in such experiments an average over a sample volume of about 0.5 cm3 and therefore the results are representative for the bulk sample volume. The elastic behavior of unprocessed materials and materials processed by ECAP was modeled using an elastic-plastic self-consistent (EPSC) scheme taking into account the measured sample texture.

3:40 PM Break

3:55 PM Invited

Investigation of Composite Deformation Using Diffraction: *Ersan Ustundag*[1]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA

The deformation mechanisms in composites are determined by a complex interaction of reinforcements and matrix. The deformation of various metal matrix composites was investigated systematically using an integrated approach that involves modeling and experiment. Employing X-ray and neutron diffraction techniques, in-situ deformation under load was studied in several Al/Al2O3, Ti/SiC and bulk metallic glass matrix composites at length scales ranging from sub-micrometer to cm. The diffraction data was then used in developing and validating micromechanics models. The results of this investigation will be presented in parallel with an exhibition of the state-of-the-art in diffraction techniques for the study of mechanical behavior of composites.

4:25 PM

Evolution of Grain-Orientation-Dependent Stresses in bcc Metals: *Y. D. Wang*[1]; X. L. Wang[1]; A. D. Stoica[1]; J. D. Almer[2]; D. R. Haeffner[2]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, Oak Ridge, TN 37830 USA; [2]Argonne National Laboratory, Adv. Photon Source, Argonne, IL 60439 USA

Recently, micro-beam synchrotron x-ray diffraction techniques using monochromatic or white beams have been developed to trace the orientation evolution and also resolve the sub-surface stress state of individual grains. However, in order to describe the properties of polycrystalline aggregates, collecting the stress distribution for a large number of individual grains with a reliable statistics is necessary. An alternative experimental method is to obtain information on the stress distribution from the measurement of lattice strains with respect to the sample directions using high-energy x-ray or neutron. A statistical distribution of strain/stress as a function of grain orientation (stress orientation distribution function, SODF) can be constructed from the measured strain pole figures with different hkl-planes. A systematic investigation on the SODF evolution during in-situ loading for a bcc metal with different primary microstructures and textures has been carried out at the APS. The material was commercial Ti-stabilized interstitial-free (IF) steel. Diffraction measurements were preformed with a monochromatic high-energy x-ray beam (E=80 keV). The strain pole figures covering the whole pole space can be easily measured during in-situ loading. The experimental results show that the micro-stress for some texture components is significantly deviated from the macro-stress after entering the plastic zone. The micro-stress along the transverse direction is not zero, which indicates that the grain-to-grain interaction occurs not only along the loading direction, but along the transverse direction as well. The direct experimental evidence on grain interaction in bcc metals during in-situ loading provides quantitative information for simulation of crystal plastic deformation.

4:45 PM

Comparison of Different X-Ray Approaches to Determination of Residual Macrostresses Using Experimental Data on the Microstress Distribution: Yu. Perlovich[1]; *M. Isaenkova*[1]; [1]Moscow Engineering Physics Institute, Kashirskoe Shosse 31, Moscow 115409 Russia

According to the widespread idea, residual macrostresses, equilibrated within the whole sample, determine an average level of interplanar spacings, whereas microstresses, equilibrated within a volume of several neighboring grains, are responsible for fluctuations of interplanar spacings about this level. Standard X-ray methods to measure micro- and macrostresses correspond to the above idea not entirely, since a number of essential aspects are missed or neglected and the mutual relation of found micro- and macrostresses does not follow from adopted experimental procedures. In particular, when determining microstrains Dd/d by the X-ray line profile analysis, the sharp inhomogeneity of microstrains depending on grain orientations is not taken into account, and when determining macrostresses by the sin2y-method, values of peak position 2qy, measured by tilt angles yi, are not interpreted in terms of microstrain distribution. The method of Generalized Pole Figures (GPF) allows to lay a new logical and methodical bridge between X-ray measurements of micro- and macrostresses, modifying both experimental procedures and data interpretation. The term GPF is used to denote distributions of diffraction or substructure parameters in the stereographic projection of the sample depending on the orientation of reflecting planes. The technique of GPF measurement involves registration of X-ray line profile by each successive position of the sample in the course of texture recording. After recalculation of measured values of the peak position 2qhkl(y,j), where y and j are coordinate angles of reflecting planes, into values of the interplanar spacing dhkl(y,j), its weighted average value dav and relative deviations [d(y,j)-dav]/dav from this value with signs ?+? or ?-? can be found. Then GPF Dd/dav describes the distribution of elastic strains in grains of the studied volume along equivalent crystallographic axes <hkl> depending on their orientation. Analysis of GPF Dd/dav for various rolled metal materials shows that regions of positive and negative Dd, corresponding to elastic extension and elastic compression, are situated in a well-ordered manner, providing a mutual equilibrium of tensile and contractile stresses about symmetry planes of the rolling scheme with usual alternation of the predominant sign by passing from one quadrant of PF to another. In order to ascertain the texture dependence of the strain distribution, correlation diagrams between GPF Dd/dav and GPF Ihkl were so constructed that each point (y,j) in GPF corresponds to some point in the diagram with the abscissa Ihkl(y,j) and the ordinate Dd/dav(y,j). In most cases these correlation diagrams prove to be symmetrical about the centre line, coinciding with the level, where Dd = 0. Such a character of the correlation diagram testifies that elastic stresses within the studied portion of the sample are equilibrated, i.e. these stresses quite satisfy the definition of microstresses, whereas dav corresponds to the lattice condition, connected with macrostresses. The sin2y-method gives macrostresses, acting in the surface plane on the assumption that the strain along the normal to the surface is equal to zero, whereas the GPF method results in the macrostress effect, irrespective of its direction. The interplanar spacing (dhkl)av, corresponding to this effect, is observed by all orientations (y,j) of axes <hkl>, having their points at the centre line of the correlation diagram. At the same time, GPF can be used for determination of the polar macrostress distribution by the sin2y-method. It is worthwhile by stress analysis as applied to textured polycrystals to combine methods of GPF and sin2y for the sake of the more unbiassed characterization of macrostresses.

Microstructural Processes in Irradiated Materials: Advanced Experimental Techniques II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

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Session Chairs: Michael Jenkins, University of Oxford, Dept. of Matls., Oxford OX1 3PH UK; Abderrahim Almazouzi, SCK-Mol Belgium

2:00 PM
State-of-the-Art TEM Characterization of Irradiated Microstructures: *Michael L. Jenkins*[1]; Mark A. Kirk[2]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK; [2]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA

This paper will discuss some recent advances in the characterisation of fine-scale microstructures by transmission electron microscopy, including: (1) The use of weak-beam microscopy to image and characterise defect clusters in the size range 1-5 nm by systematic control of the diffraction conditions. (2) Progress in image simulations under weak-beam diffraction conditions of nanoclusters of complex morphology. (3) The benefits of the use of energy-filtering to remove inelastically scattered electrons with energy losses greater than about 10 eV from weak-beam images and diffraction patterns. Preliminary experiments suggest that the elastic Huang scattering near weak Bragg spots in energy-filtered diffraction patterns obtained from regions containing single defects may contain rich information on the defect nature and strain field. This work was partially supported by DoE.

2:45 PM
Electrical Resistivity and Thermal Conductivity of Molybdenum in the Pre- and Post-Irradiated Condition: *Brian V. Cockeram*[1]; James L. Hollenbeck[1]; Lance L. Snead[2]; [1]Bechtel-Bettis Atomic Power Laboratory, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122-0079 USA; [2]Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6138 USA

Electrical resistivity is a fundamental measure of the defect density of a metal. Electrical resistivity, thermal conductivity, and baseline tensile data were measured for wire, sheet, and plate forms of Low Carbon Arc Cast (LCAC), Oxide Dispersion Strengthen (ODS), and TZM molybdenum to provide a basic assessment of the influence of the amount of work, grain orientation, and recrystallization on the relative defect densities. Slightly higher electrical resistivity values were observed for the wrought product forms that had a higher amount of work. Since the electrical resistivity and thermal conductivity of a metal are both controlled by the same mechanism of electronic conduction, these properties are related by a constant known as the Lorenz number (Wiedemann-Franz relationship). Out-of-pile electrical resistivity and thermal conductivity measurements were made for comparison with post-irradiated data and to experimentally determine the Lorenz number so that an indirect measurement of post-irradiated thermal conductivity could be obtained. Irradiation of molybdenum in the High Flux Isotope Reactor (HFIR) at temperatures ranging from 300C to 1200C and to neutron fluence levels ranging from 10.5 to 64.4 X 10^20 n/cm2 (E > 0.1 MeV) was shown to result in a small decrease (0% to 10%) in the thermal conductivity of molybdenum.

3:05 PM
Post-Irradiation Annealing of Small Defect Clusters: *Jeremy T. Busby*[1]; Matt M. Sowa[1]; Mark C. Hash[1]; Gary S. Was[1]; Edward P. Simonen[2]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; [2]Batelle Pacific Northwest National Laboratories, Richland, WA 99352 USA

Post-irradiation annealing studies indicate small defect clusters may be a potential contributor to IASCC. In this study, small defect clusters and their behavior during annealing are examined. A CP-304 SS alloy was irradiated with 3.2 MeV protons to 1.0 dpa at 360∞C or up to 0.5 dpa at <100∞C. For samples irradiated at 360∞C, the smallest dislocation loops and clusters were removed preferentially during annealing at 500∞C. The increase in hardness after 0.3 dpa at <100∞C was greater than that after 1.0 dpa at 360∞C, but dropped significantly after annealing at 350∞C for short times. The small defect clusters in samples irradiated at both temperatures are analyzed using both transmission electron microscopy (TEM) and small angle x-ray scattering (SAXS). Simulation results are compared to the experimental results to gain further insight into the annealing behavior and nature of small clusters. Research at the University of Michigan was supported under US Department of Energy grant DE-FG07-991D13768.

3:25 PM Break

3:55 PM
Analytical Electron Microscopy of Oxide Dispersion Strengthened Molybdenum: Effects of Irradiation on Material Microstructure: *Rita Baranwal*[1]; Mary Grace Burke[1]; [1]Bechtel Bettis, Inc., Bettis Atomic Power Lab., 08D/MT, PO Box 79, W. Mifflin, PA 15122-0079 USA

Oxide Dispersion Strengthened (ODS) molybdenum is attractive for high-temperature applications due to its superior creep performance relative to other molybdenum alloys. It is believed that the dispersed oxide phase lends itself to improved creep resistance by impeding grain boundary sliding and dislocation motion. To optimize these materials for use in nuclear applications, it is necessary to understand how neutron irradiation affects the microstructure of ODS molybdenum in terms of dimensional changes, defect substructure, stability of second phase particles, and the interaction between defects and microstructure. In turn, this understanding will enable the development of better, more radiation-resistant molybdenum alloys. To determine the effects of irradiation on material microstructure, ODS molybdenum has been characterized using analytical electron microscopy (AEM). This work describes the results-to-date from AEM characterization of unirradiated and irradiated ODS molybdenum. The general microstructure of the unirradiated material consists of fine molybdenum grains with numerous low angle boundaries and dislocation networks. ìRibbon-îlike lanthanum oxides are aligned along the working direction of the product form and are frequently associated with grain boundaries, serving to inhibit grain boundary movement. In addition to the ìribbons,î discrete lanthanum oxide particles have also been detected. After irradiation, the material was characterized by the presence of non-uniformly distributed large (~20 to 100 nm in diameter), multi-faceted cavities.

4:15 PM
Quantitative Measurement of Electron Diffuse Scattering by Single Nanometer-Sized Defects in Au and Mo: *Marquis A. Kirk*[1]; Michael L. Jenkins[2]; Ray D. Twesten[3]; [1]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK; [3]University of Illinois, Seitz Matls. Rsrch. Lab., 104 S. Goodwin Ave., Urbana, IL 61801 USA

Elastic diffuse scattering of electrons by single nanometer-sized defects in ion irradiated Au and Mo has been measured quantitatively. Huang scattering from isolated single defects is separated from Bragg scattering at a weakly excited diffraction peak (usually 2g, with near 5g excited). The asymmetry in the Huang scattering immediately reveals the interstitial or vacancy nature of the defect. Results will be compared among various dislocation loop geometries and with calculations. Comparison to similar measurements on necessarily broad distributions of defect types and sizes by diffuse x-ray scattering will be made. Contributions of inelastic electron scattering to defect images and diffraction data are also measured and will be discussed. Work is supported by DoE.

4:35 PM

On the Composition of Copper Rich Precipitates in Irradiated RPV SteelsñTemperature Dependent Magnetic Small Angle Neutron Scattering: *G. Robert Odette*[1]; [1]University of California-Santa Barbara, Dept. of Mechl. & Environl. Eng., Santa Barbara, CA 93106 USA

Controversy exists regarding the composition of copper rich precipitates (CRPs) in irradiated pressure vessel steels. Atom probe (AP) data is interpreted to indicate large amounts of iron in the CRPs. However, both SANS and positron annihilation data show the CRPs contain little or no iron. All techniques rely on assumptions. For example, the AP technique assumes sub-atomic scale position resolution while SANS results assume that CRPs are non-magnetic. The latter assumption can be probed by SANS measurements over a range of temperatures. If the CRPs are partially magnetic the magnetic scattering increases with increasing temperature, as a result of the effect of alloying on the CRP Curie temperature. On the other hand if the CRPs are non-magnetic, then the magnetic scattering decreases with increasing temperature. Temperature dependent SANS data is presented for both simple model alloys and complex steels that clearly show the CRP contain little iron.

4:55 PM

The Effects of Dose Rate, Temperature and Composition on Irradiation Hardening of RPV Steels: *G. Robert Odette*[1]; [1]University of California-Santa Barbara, Dept. of Mechl. & Environml. Eng., Santa Barbara, CA 93106 USA

Dose rate, temperature and composition effects on irradiation hardening (Dsy) of RPV steels are assessed. A wide range of alloy compositions were irradiated at fluxes (f) from = 5x1010 to 1012 n/cm2-s at 270, 290 and 310C to overlapping fluences (ft) up to about 3x1018 (lowest f) to 3x1019 (highest f) n/cm2. The Dsy versus ft curves systematically shift to lower ft with decreasing f, Ni and Mn and increasing Cu. Flux effects decrease with increasing irradiation temperature. The database is analyzed using a Cu-rich precipitate (CRP) diffusion controlled growth hardening model. Precipitation is accelerated by radiation enhanced diffusion; and the effect of dose rate can be described by a flux-dependent acceleration factor. The Ni-Mn-f dependent acceleration factor is consistent with Ni-Mn trapping enhanced recombination. The Dsy-based analysis is consistent with SANS data, but the efficiency of RED is higher that predicted by simple rate theory models.

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Alloy Phases Committee

Program Organizers: Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Michael R. Notis, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18015 USA; Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA

Monday PM — Room: 12
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Douglas J. Swenson, Michigan Technological University, Dept. of Matls. Sci. & Eng., Houghton, MI 49931 USA; C. Robert Kao, National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 32054 Taiwan

2:00 PM Invited

Phase Transitions in Rare-Earth Metal/Si Systems: *L. J. Chen*[1]; [1]National Tsing Hua University, Matls. Sci. & Eng., 101 Sec., 2 Kuang-Fu Rd., Hsinchu 300 Taiwan

Rare-earth silicides are unique in that they possess the lowest known Schottky barrier heights (~0.3-0.4 eV) on n-type silicon with potential applications in infrared detectors and as ohmic contacts or rectifying contacts. The compositions previously reported for the stable silicon-rich phases of rare-earth silicide films have ranged from RESi1.6 to RESi2, with the former representing a vacancy distribution in the Si sublattice. The AlB2 type RE silicides with hexagonal structure have excellent lattice match with Si so that high quality epitaxial thin films can be grown on Si. Recently, RESi2-x nanowires were grown on (001)Si. In this presentation, an overview of phase transitions in rare-earth metal/Si systems will be provided. Formation of amorphous interlayers and crystalline silicides, evolution of vacancy ordering, formation of double epitaxial heterostructure and growth of pinhole-free silicide film will be highlighted.

2:20 PM Invited

Phase Stability and Processing of the MgB2 Thin Film: *Zi-Kui Liu*[1]; Xiao Xing Xi[2]; [1]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA; [2]The Pennsylvania State University, Dept. of Physics, University Park, PA 16802 USA

MgB2 was discovered to be a superconductor with a critical temperature of 39K, the highest among known nonoxide compounds[1]. This presentation will review recent research activities at Penn State on phase stability and processing of the MgB2 thin film[2-5]. Following issues will be discussed: (1) Thermodynamic modeling of the Mg-B binary system, (2) First-principle calculation of the MgB2 phase, (3) Implications for the deposition of MgB2 thin films, (4) In situ epitaxial MgB2 thin films for superconducting electronics. [1]J. Nagamatsu et al., Nature, 410 (2001) 63-64. [2]Z. K. Liu, D. G. Schlom, Q. Li and X. X. Xi, Appl. Phys. Lett., 78 (2001) 3678-3680. [3]Z. K. Liu, Y. Zhong, D. G. Schlom, X. X. Xi and Q. Li, CALPHAD, 25 (2001) 299-303. [4]X. H. Zeng et al., Appl. Phys. Lett., 79 (2001) 1840-1842. [5]X. H. Zeng et al., Nature Materials, (2002).

2:40 PM Invited

Predicting Interdiffusion in Electronic Materials: *J. E. Morral*[1]; [1]University of Connecticut, Metall. & Matls. Eng., 97 N. Eagleville Rd., U-136, Storrs, CT 06269-3136 USA

Interdiffusion of layered microstructures in electronic materials can be a complex phenomenon to model. For example high diffusivity paths, internal stresses, plastic strain, and the nucleation of phases and voids can lead to behavior that is difficult to model without including details of the microstructural evolution. However when such complications are absent, interdiffusion microstructures can be predicted with finite difference software like DICTRA. Such software treats precipitates as point sources and sinks of solute and assumes local equilibrium. With the proper database it can predict concentration profiles and diffusion paths even in multicomponent, multiphase systems. In this presentation the principles needed to interpret the results of DICTRA type predictions will be given.

3:00 PM

High-Resolution Transmission Electron Microscopy of Silicide Formation and Stability of Ni/Si and Ni/SiGe: *Xiao Chen*[1]; Zhonghai Shi[1]; Jiping Zhou[2]; Llewellyn Rabenberg[2]; Sanjay K. Banerjee[1]; [1]The University of Texas at Austin, Microelect. Rsrch. Ctr., PRC/MER R9950, Austin, TX 78712 USA; [2]The University of Texas at Austin, TX Matls. Inst., Austin, TX 78712 USA

Nickel silicide phase formation and thermal stability in Ni/Si and Ni/SiGe systems have been studied using high-resolution transmission electron microscopy (HRTEM) in conjunction with energy dispersive spectrometry (EDS) and nano-beam diffraction (NBD) techniques. Correlated with sheet resistance data, both low- and high-resistivity nickel silicide samples in Si and strained Si1-xGex (x=0.20) were examined. With low temperature anneal, low-resistivity phase NiSi and Ni(Si1-xGex) (x<0.12) crystalline films were formed, and were relatively uniform and smooth on underlying layers. As the annealing temperature was increased, high-resistivity phase NiSi2 (>850¢™C) and heavily Ge-deficient Ni(Si1-xGex)2 (>600¢™C) were observed in the form of pyramids or trapezoidal islands in each system. EDS revealed that for SiGe, more Ge was segregated at the silicide growth front at higher temperature, especially in the lateral direction. It was therefore assumed that the discontinuous silicides films and high-resistivity phases are the main causes for high sheet resistance.

3:15 PM

Grain Growth in Nanoscale PdIn Thin Films: *Mianliang Huang*[1]; Y. Austin Chang[1]; [1]University of Wisconsin-Madison, MS&E, 1509 Univ. Ave., Madison, WI 53706 USA

Using TEM, the grain growth in 30 nm thick sputter-deposited PdIn thin films was investigated by annealing at a series of temperatures for various periods of time. The sequence of growth in this study was observed to begin with very rapid normal grain growth with monomodal and lognormal grain size distributions. The subsequent slower growth period occurred when the mean grain size was in the same range as the film thickness. The surface energy anisotropy led to abnormal grain growth with bimodal grain size distribution and textured fiber structure. A grain growth exponent of 2.3 was obtained by fitting the measured grain sizes to the grain growth law. A grain growth activation energy of 54 kJ/mol was estimated by plot lnα versus 1/T. The grain growth rate was dependent on heating rate.

3:30 PM Break

3:50 PM Invited
Reactions at the Liquid Silicon/Silica Glass Interface: *Rainer Schmid-Fetzer*[1]; Sophie M. Schnurre[1]; [1]Technical University of Clausthal, Inst. of Metall., Robert-Koch-Str. Nr. 42, Clausthal-Zellerfeld 38678 Germany

Experiments have been carried out to determine the nature and origin of the spots growing on silica glass surfaces in contact with liquid silicon during CZ-Si crystal growth. Silica glass ampoules were filled with silicon and tempered between 5\~min and 40\~h at a temperature (1693\~K) slightly above the melting point of silicon. Cross sections of the ampoules with solidified silicon have been examined by scanning electron microscopy and optical polarization microscopy. In addition cross sections from commercial silica glass crucibles used in the Czochralski process or dipped into the silicon melt were investigated with the same methods. At the silicon\~/\~silica glass interface different reaction zone morphologies were detected. A solution-precipitation mechanism is suggested for the fast lateral growth of the reaction zone, which is proposed to consist of small cristobalite crystals embedded in a silica glass matrix.

4:10 PM Invited
The Effect of Pd and Cu in the Intermetallic Growth of Au Alloy Wire-Bond: Hen-So Chang[1]; *Ker-Chang Hsieh*[1]; Theo Martens[2]; Albert Yang[2]; [1]National Sun Yat-Sen University, Inst. of Matls. Sci. & Eng., Kaohsiung 80424 Taiwan; [2]Philips Electronic Building Elements Industries (Taiwan), Ltd., Tech. Dev. Div., 10 Chin 5th Rd., N.E.P.Z., PO Box 35-48, Kaohsiung Taiwan

This is the new wire evaluation work for the reliability of wire bonding process. There is a trend for the plastic IC package to function at higher junction temperature with thinner wire. New alloy Au wires have been developed to meet the reliability requirements. Two types of alloy Au wires as Au-Pd and Au-Cu wire were evaluated in this study. These samples were aged between 155~205°C under air from 0 hour to 2000 hours. According to this study, the phase formation sequence of Au2Al, Au5Al2 and Au4Al intermetallic is similar to the pure Au wire. There is a Pd rich layer working as diffusion barrier to slow down the growth rate of intermetallic phases in Au-Pd wire. Au-Cu wire also slowed down the growth rate with different mechanism. Both wires have better reliability based on the microstructure examination. The contact resistance measurements also show longer working life at higher temperatures in comparison with the pure Au wire.

4:30 PM Invited
Li-Intercalation Cathode Material for Rechargeable Battery: Yong Jeong Kim[1]; Tae-Joon Kim[1]; Byoungsoo Kim[1]; *Byungwoo Park*[1]; [1]Seoul National University, Sch. of Matls. Sci. & Eng., Seoul 151-744 Korea

The market for Li-ion batteries is undergoing rapid expansion, as portable electronic devices demand a higher energy density and a better cycle life. Even though $Li_{1-x}CoO_2$ cathode has been widely used in commercial Li-ion batteries, electrochemical charge (Li deintercalation) and discharge (Li intercalation) cause capacity fading. The possibility of producing more-efficient rechargeable Li batteries is offered by nanoscale metal-oxide coating, resulting in the suppression of changes in the lattice constants during electrochemical cycling. The order of capacity retention correlates well with the fracture toughness of nanoscale oxides. The same method is applied to thin-film batteries, showing enhanced cycle-life performance. However, unlike the powder geometry, both bare and metal-oxide-coated $Li_{1-x}CoO_2$ thin films show negligible c-axis expansion. The potential mechanisms for the enhanced electrochemical properties will be discussed in this talk. J. Cho, Y. J. Kim, T.-J. Kim, and B. Park, Angew. Chem. Int. Ed. 40, 3367 (2001). Y. J. Kim, T.-J. Kim, J. W. Shin, B. Park, and J. Cho, J. Electrochem. Soc. (2002).

4:50 PM
Phase Stability of Co-Fe and Cu Multilayered Thin Films: *Peter F. Ladwig*[1]; Thomas F. Kelly[2]; David J. Larson[3]; Y. Austin Chang[1]; [1]University of Wisconsin-Madison, Matls. Sci. Prog., 1509 Univ. Ave., Madison, WI 53706 USA; [2]Imago Scientific Instruments Corporation, 6300 Enterprise Ln., Madison, WI 53719 USA; [3]Seagate Technology, 7801 Computer Ave., Bloomington, MN 55435 USA

Interdiffusion between Co-Fe and Cu thin films is a suspected long-term failure mechanism in giant magnetoresistive (GMR) devices used in the magnetic storage industry. The bulk thermodynamic phase equilibria suggest that the interface is stable. However, in GMR devices the thin film layer thicknesses are less than 3 nm, giving rise to strain/interfacial energy contributions that affect the integrity of the Co-Fe and Cu layers. Difficulty arises when trying to characterize interdiffusion on the sub-nm scale. Differential scanning calorimetry (DSC), high resolution TEM, electron energy loss spectroscopy (EELS), and X-ray diffraction give inconclusive information on the degree of intermixing. However, atom probe tomography was successfully employed to directly measure the intermixing and phase separation in annealed samples. This data proves that in thin-films, interfacial and strain contributions to the free energy can cause reactions that cannot be predicted from conventional bulk phase equilibria.

5:05 PM
Electrochemical Behavior of Zincate Pretreatment Prior to Electroless Nickel Deposition for UBM Applications: *Jae-Ho Lee*[1]; Ingun Lee[2]; Tak Kang[2]; Namseog Kim[3]; Seyong Oh[3]; [1]Hong Ik University, Dept. of Metallurgl. Eng. & Matls. Sci., Chungnam-do 339-800 S. Korea; [2]Seoul National University, Dept of Matls. Sci., Seoul 151-742 S. Korea; [3]Samsung Electronics Company, Ltd., Package Dvlp. Team, Youngin City, Kyunggi Do 449-711 S. Korea

The importance of under bump metallurgy (UBM) has been emphasized in flip chip packaging. The adhesion and uniformity of nickel/gold deposits on aluminum pad are a very important factor in the reliability of solder. Aluminum pad was pretreated in zincate solution prior to electroless nickel plating. Zinc on aluminum gave the good adherent nickel layer and then the adhesion and uniformity of zinc on aluminum is the key factor in UBM. The relationship between aluminum dissolution and the ratio of zinc and NaOH was investigated. When ZnO was presented in solution, the electrode potential was changed very rapidly in 20 sec, during this period seeding of zinc were occurred. Zincating duration and surface morphology were also investigated. The uniform and nano sized zinc nuclei gave better adhesion to electroless nickel deposition.

Residue Handling in Metals Processing - I

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizer: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880 USA

Monday PM	Room: 1B
March 3, 2003	Location: San Diego Convention Center

Session Chairs: Thomas P. Battle, DuPont Titanium Technologies, Wilmington, DE 19880 USA; Larry A. Lien, Osmonics, Industl. Processes & Wastewater, Vista, CA 92083-7986 USA

2:00 PM
Use of Cement-Bonded Agglomerates as Burden Material for Blast Furnaces: *Maneesh Singh*[1]; Bo Bjˆrkman[1]; [1]LuleÂ University of Technology, Div. of Process Metall., LuleÂ 971 87 Sweden

During the various stages of iron & steel production a number of iron-rich by-products are generated. The traditional way to recycle them back to the blast furnace has been the balling-sintering process. These by-products can also be made into cement-bonded agglomerates for use as burden material for blast furnaces. In order to improve the quality of agglomerates the effect of various processing parameters on the properties of cement bonded briquettes have to be controlled. This paper looks into the various aspects of producing cement-bonded agglomerates and using them as burden material for blast furnaces.

2:20 PM
Smorgasbord Recovery Solutions for Ferrous and Non-Ferrous Producers: *Gary E. Metius*[1]; James M. McClelland[1]; [1]Midrex Technologies, Inc., Business Dvlp., 2725 Water Ridge Pkwy., Ste. 100, Charlotte, NC 28217 USA

In metals processing, residue streams are routinely generated containing recoverable metallic compounds. These metallics represent both valuable materials and potential disposal problems to the producer. Midrex, primarily involved in ferrous conversion for many years, has developed a variety of new processing techniques for ferrous and non-ferrous recovery. The processing technologies involve either shaft or rotary hearth furnaces, and can be both hydrocarbon or coal based. Recent developments have included conversion studies for ferrous and non-ferrous residual streams that are energy efficient and environmentally friendly. The technologies to be presented, predominantly coal based, include FASTMET, FASTMELT and ITmk3, can be offered over a wide range of site specific capacity options.

2:40 PM
Thermal Decomposition of Basic Zinc Carbonate Obtained After Leaching of EAF Dust: *Bo Lindblom*[1]; ≈ke Sandstrˆm[1]; Nourreddine Menad[1]; [1]Lulea University of Technology, Process Metall., Lulea SE-971 87 Sweden

An EAF dust generated after smelting zinc-coated scrap has been leached in hydrochloric acid at pH=3. The solution obtained has been subjected to purification by cementation followed by precipitation of zinc with sodium carbonate. This paper describes the thermal decomposition of the precipitate studied by TG-DTA equipment and XRD. During the calcination of the basic zinc carbonate three different reactions occur, decomposition of Zn5(CO3)2(OH)6, CaZn(CO3)2 and CaCO3, yielding a final product containing 61.9% ZnO and 38.1% CaO.

3:00 PM
EAF Dust Processing with a Combination of Hydro- and Pyro-Metallurgical Techniques: *≈ke Sandstrˆm*[1]; Qixing Yang[1]; Nourreddine Menad[1]; Bo Lindblom[1]; [1]LuleÂ University of Technology, Process Metall., LuleÂ SE-971 87 Sweden

One representative dust sample generated in the EAF process was treated by a combination of hydrometallurgical and pyrometallurgical techniques. The EAF dust, containing mainly ZnFe2O4, ZnO, Fe3O4 and Mn3O4, was treated first by hydrochloric acid leaching at pH=3 to dissolve 63% of the zinc. The leaching solution was purified by cementation with zinc dust followed by precipitation of zinc with sodium carbonate. The zinc hydroxo-carbonate (Zn5(CO3)2(OH)6) formed was subsequently calcined to zinc oxide. The pyrometallurgical treatment of the leaching residue was then studied using an induction heating system to simulate important operations of EAF smelting of the residue in combination with steelmaking. The leaching residue was mixed with carbon powder to obtain a test mixture with 52.1% of ZnO.Fe2O3 and 17.8% of C. The mixture, the scrap and slag former were charged together in a crucible and heated to attain a melt temperature around 1650°C. Several mixture smelting tests were performed to examine effects of mixture amounts on Zn balance and Fe recovery. Based on the test results, suggestions were made on the EAF recycling of the leaching residue.

3:20 PM Break

3:40 PM
Mechanism of Enrichment of Gallium from Zinc-Leaching Residue by Cold-Bonded Coal-Based Direct Reduction Process: Guanghui Li[1]; Tao Jiang[1]; Zhucheng Huang[1]; Yufeng Guo[1]; Yongbin Yang[1]; [1]Central South University, Sch. of Resources Procg. & Bioeng., Changsha, Hunan 410083 China

In the paper, the recovery technology of gallium from zinc-leaching residue by cold-bonded coal-based direct reduction process following magnetic separation was developed. The results show that the scattered metal Ga can be concentrated obviously after the reduction of zinc-leaching residue briquette and magnetic separation of reduced product. At the same time, the volatilization of Zn in the residue is enhanced during the reduction. When the residue, bearing 20.58% Zn, 19.14% Fe and 570g/t Ga, was reduced with coal at 1100∞C for 150min, the results of Zn volatilization of 98.42% and Ga recovery of 89.10% were obtained in reduction. After magnetic separation of the reduced product, the concentration of Ga in sponge iron is 2164g/t, and the separation recovery of Ga reaches 92.42%. The enriched mechanism of Ga during reduction-separation process was studied by using electron microscope,scanning electron microscope, energy spectrum and mineralogical analysis. The investigations show that there is no formation of independent phase of Ga during reduction process. The Ga occurs mainly in metallic iron phase (the concentration of Ga in metallic iron phase is 0.04%~0.4%), in addition, there exists a little Ga in poorly crystalline sulphide (0.08%~0.15% Ga). But Ga does not exist in most of glass and silicate phase. The affinity of Ga for metallic iron is the enrichment basis of Ga in reduction, and it is also in favor of recovering Ga during magnetic separation.

4:00 PM
Technology Development on Decontamination & Metals Recovery of Fly Ashes from Municipal Waste Incineration: *Kazuyuki Kikuta*[1]; Kazuhiro Kojima[1]; Takafumi Tsujimoto[1]; [1]Metal Mining Agency of Japan, Tech. Dvlp., Toranomon 1-24-14, Minato-ku, Tokyo 105-0001 Japan

MMAJ, a governmental organization in Japan, has carried out a 4 year project for the development of the decontamination & metals recovering technology for the fly ashes from municipal waste incineration. Heavy metals (zinc, lead and copper) are recovered as raw materials for the non-ferrous metals production, particularly for Imperial Smelting Process (ISP) without formation of any waste material to be disposed. More than 500 thousands tonnes a year of fly ashes generation from the municipal waste incineration has been one of the main social issues in Japan, because most of them need to be disposed to the registered areas after decontamination, and occupy 5% of the municipal waste disposed. The segregation, floatation and chemical process are applied to the treatment of the primary fly ash from municipal waste incineration, and MF (Mitsui-type blast furnace) Process after the chlorine removal to the secondary fly ash.

4:20 PM
Calcinations of Sludge from Waste Water in Surface Finishing with No Generation of Hexavalent Chromium: *Ryokichi Shimpo*[1]; Kazunori Kato[1]; Noriyuki Takatsu[1]; Shigeo Hoshino[1]; [1]Musashi-Institute of Technology, Dept. of Mech., Tamadutumi 1-28-1, Setagaya-ku, Tokyo 158-8557 Japan

Much amount of sludge is produced from the waste water treatments in surface finishing industry. There are many problems for the abandonment of the sludge because of the high water content and heavy metals included. It is often observed that hexavalent chromium, which is toxic, is generated in the course of heat treatments of the sludge for reducing its amount by dispersing water. In this study, conditions of the sludge calcinations for reducing water contents have been examined. Maximum hexavalent chromium generation was observed in the temperature range from 673 to 773K, at which most of the crystalline water decomposed from the sludge. When the heating temperature became higher than 1173K, the amount of the hexavalent chromium extracted from the calcinated sludge became very low, less than 1mg/kg sludge. When the sludge was heated with iron oxides, Fe2O3 or FeO, iron ferrite has been synthesized which may be used as a magnetic material. The amounts of hexavalent chromium generated in the heat treatment with iron oxides were not much different from those without iron oxides.

4:40 PM
Membrane Technology Assists New Zinc Refinery in Townsville, Australia to Become Zero Discharge: *Larry Allen Lien*[1]; [1]Osmonics, Industl. Process & Wastewater, 760 Shadowridge Dr., Vista, CA 92083-7986 USA

Zero Discharge from industrial manufacturers has been a much talked about goal of environmental groups, governments and industry for past decade. A new zinc refinery in Townsville, Australia has economically become zero discharge with the use of several membrane systems. Membrane technology was needed because the zinc refinery wastewater after conventional lime precipitation still contained 30-50 ppm of boron and could not be discharged as originally planned. Using this post lime precipitation wastewater as a feed source, now saturated in calcium sulfate by nature of lime addition to low pH sulfuric acid refinery waste waters, a novel designed two pass membrane processes-nanofiltration followed by reverse osmosis proved to be cost effective. As a result the refinery produces high quality boiler feed water and dramatically reduced the size of an evaporation pond originally designed by 90%. The savings using membrane technology were two-fold. First, and foremost, a $20M US savings resulted in a much smaller evaporation pond. Secondly, the reuse of wastewater as high quality boiler feed, eliminated the need for ion exchange and permits for ground water needed for refinery make-up. The ion exchange elimination would have paid for the $1M US wastewater plant would have paid for the system in one year by itself.

Sensors and Control in Materials Processing - I

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee
Program Organizer: Stavros A. Argyropoulos, University of Toronto, Department of Materials Science and Engineering, Toronto, Ontario M5S 3E4 Canada

Monday PM Room: 1A
March 3, 2003 Location: San Diego Convention Center

Session Chair: Stavros A. Argyropoulos, University of Toronto, Dept. of Matls. Sci. & Eng., Toronto, Ontario M5S 3E4 Canada

2:00 PM
Strategies for Quenching Sensors and Control: *Richard D. Sisson*[1]; Md. Maniruzzaman[1]; Shuhui Ma[1]; [1]Worcester Polytechnic Institute, Matls. Sci. & Eng., 100 Institute Rd., Worcester, MA 01609 USA

The current status of quenchant characterization and quenching sensor technology will be reviewed. The need for a real time quenching sensor will be presented and discussed. Based on these technologies and industry needs some strategies for the control of a quenching process will be presented and discussed. These strategies will include simple statistical process control as well as intelligent control via real time sensor data, process models and agitation actuators.

2:25 PM
Determination of Hydrogen in Molten Aluminium and its Alloys Using an Electrochemical Sensor: Carsten Schwandt[1]; *Derek J. Fray*[1]; [1]University of Cambridge, Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Hydrogen is a very important impurity in molten aluminium as it can readily be picked up from the environment and, on subsequent solidification, can cause porosity. It is, therefore, very important that the hydrogen content on the aluminium is known, prior to casting. A novel sensor is described which consists of a calcium zirconate electrolyte with a solid state reference of metal/metal hydride. The sensor is operated in the current reversal mode in which a potential is applied, the current is measured, the potential is reversed, the current re-measured, and the ratio of the currents recalculated. The current ratio is a function of the hydrogen content of the melt and this was shown to give a faster response rather than operating the sensor in the Nernstian mode. There was good agreement between the readings given by the sensor and the results given by the Alscan analyser.

2:50 PM
Development of a Computer-Based Data Acquisition System for Die Temperature Control: *Henry Hu*[1]; Fang Chen[1]; Xiang Chen[1]; Yeou-li Chu[2]; Patrick Cheng[2]; [1]University of Windsor, Dept. of Mechl., Auto. & Matls. Eng., Windsor, Ontario N9B 3P4 Canada; [2]Ryobi Die Casting (USA), Inc., Dept. of R&D, Shelbyville, IN 46176-9720 USA

In high pressure die casting processes, proper temperature control of a die yields a high casting production and superior quality components. Die temperature is usually influenced by various die design and process parameters such as size and location of internal water cooling lines, flow rates of cooling lines, and pouring temperature of molten metal. Among them, the cooling water flow rate plays a major role in controlling thermal pattern of the die. In order to develop an effective control strategy, data of localized die temperature must be collected and analyzed. In this study, a computer-based data acquisition system (DAS) for die temperature control has been designed, and established. The developed DAS has three channels for temperature signals and one channel for water flow rate signals, and is capable of displaying data in both curve format and value format. Implementation of the DAS in a laboratory setup has also been attempted. It should be pointed out that a control mechanism can be potentially integrated into this system.

3:15 PM
Sensor Development for Sorting and Quality Control of Non-Ferrous Scrap Metal: *Marian Bogdan Mesina*[1]; Tako P.R. de Jong[1]; Wijnand L. Dalmijn[1]; [1]Delft University of Technology, Fac. of Appl. Earth Scis., Resource Eng., Mijnbouwstraat 120, Delft, Zuid Holland 2628RX The Netherlands

In our modern society the recycling of waste and discarded products becomes more and more important due to various economical and environmental factors. At present, manual sorting and mechanical separation are used for the separation process of discarded materials. Sensor technology is an alternative for material characterisation and identification. The advantage in using sensors consists in a full automation of the material inspection and better product qualities. This paper describes new possibilities for non-ferrous scrap material characterisation and identification using sensors based on the electromagnetic field. A dedicated sensor evaluates the interaction between an alternating electromagnetic field and the material under inspection. The output signal depends on the type of non-ferrous material. At Delft University an electromagnetic prototype sensor for on-line inspection of non-ferrous metals was developed in co-operation with industry. Based on the electrical conductivity of non-ferrous metals, the electromagnetic sensor can easily differentiate between low and high conductive non-ferrous metals. The paper presents the results of the experiments and the possibility to integrate such sensors in non-ferrous scrap metal recycling and process control.

3:40 PM
Dual Energy X-Ray Transmission Imaging: Applications in Metal Processing: *Tako P.R. de Jong*[1]; [1]Delft University of Technology, Appl. Earth Scis./Raw Matls. Procg., Mijnbouwstraat 120, Delft 2521 CR The Netherlands

Dual energy X-ray transmission imaging is extensively applied for safety luggage inspection. Its advantages are its rapid scanning rate (25-100 cm/sec) at approximately 1 mm resolution and simultaneous thickness independent material identification. This is especially advantageous for secondary scrap metal inspection and automatic sorting. Other applications include metal product inspection, bale control, and waste processing. Recent progress in development of an automatic on-line sorter for bulk solids is presented and discussed. Besides various non-metal applications (solid fuels, wastes etc.) it appeared particularly effective in light metal sorting. In addition as inspection tool better control of difficult to monitor solid bulk streams, bales and other related materials becomes possible.

Surface Engineering in Materials Science - II: Nanotechnology and Property Evaluation II

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA; A. Agarwal, Plasma Processes, Inc., Huntsville, AL 25811-1558 USA; Narendra B. Dahotre, University of Tennessee-Knoxville, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; C. Suryanarayana, University of Central Florida, Mechanical, Materials & Aerospace Engineering, Orlando, FL 32816 USA

Monday PM Room: 7A
March 3, 2003 Location: San Diego Convention Center

Session Chairs: Caroline R. Aita, University of Wisconsin, Matls. Eng., Milwaukee, WI 53201-0784 USA; Arvind Agarwal, Plasma Processes, Huntsville, AL 35811-1558 USA

2:00 PM Invited
Nanocrystalline MCrAlY Bond Coat for Thermal Barrier Coating (TBC) Applications: Leonardo Ajdelsztajn[1]; Julie M. Schoenung[1]; *Enrique J. Lavernia*[1]; [1]University of California-Davis, Cheml. Eng. & Matls. Sci., 2017 Engr. II, Davis, CA 95616 USA

This work describes recent progress in improving the oxidation behavior of the bond coat using HVOF nanostructured MCrAlY coatings. NiCrAlY and CoNiCrAlY powders were cryomilled and HVOF and/or plasma (LPPS) sprayed onto Ni-based alloy. Oxidation experiments were performed on the coating to form the thermally grown oxide layer (TGO). The formation of the oxide phases on top of the bond coat after heat treatment at 1000°C was analyzed (morphology and composition) for different heat treatment times. In the nanostructured coatings, the presence of a homogeneous α-Al2O3 layer was observed. The nanostructured characteristic of the coating and the presence of Al2O3 within the cryomilled powders enhance the nucleation of the TGO alumina layer which protects the coating from further oxidation and avoids the formation of mixed oxide protrusions presented on the coating sprayed using the as-received powder.

2:30 PM
Aluminum Based Nanostructured Composite Coatings: Processing, Microstructure and Wear Behavior: *Arvind Agarwal*[1]; S. Seal[2]; S. Wannaparhun[1]; K. Rea[3]; N. Dahotre[4]; [1]Plasma Processes, Inc., 4914 Moores Mill Rd., Huntsville, AL 35811-1558 USA; [2]University of Central Florida, AMPAC, MMAE, Orlando, FL 32816 USA; [3]University of Central Florida, Orlando, FL 32816 USA; [4]University of Tennessee-Knoxville, Matl. Sci. & Eng., Knoxville, TN 37932 USA

Hypereutectic aluminum alloy reinforced with nano-grained alumina is a lightweight material with potential application in several industries. The reinforcement with nano-size ceramic and Si grains result in a significant increase in strength, stiffness and wear resistance. In the present work, plasma spraying has been employed to deposit nanostructured Al-Al2O3 composite coating on 6061 Al substrate. This work focusses on the processing of nanostructured composite coating, its microstructural characterization and sliding wear behavior.

2:50 PM
Nano-Structured Porous Oxide Coating on Fine FeCrAl Alloy Fibers: W. Fei[1]; *S. Seal*[2]; S. Kuiry[3]; N. Quick[1]; [1]University of Central Florida, MMAE, 4000 Univ. Blvd., Orlando, FL 32816 USA; [2]University of Central Florida, AMPAC, MMAE, 4000 Univ. Blvd., Orlando, FL 32816 USA; [3]University of Central Florida, AMPAC, 4000 Univ. Blvd., Orlando, FL 32816 USA

Recently, fine FeCrAl fibers have attracted attention in hot gas filtration system. FeCrAl fiber media had shown high efficiency, low pressure drop and high temperature strength. However, because of the high gas permeability, the performance of filtering sub-micron size particles needs to be improved. Nano-structured porous Zirconia and Yttria-stabilized-zirconia (YSZ) coating was successfully applied on fiber media to increase the surface area by sol-gel method using selected precursors. The sub-micron size pores are supposed to provide sites for trapping particles. Different parameters like precursor-water ratio, chelating agent-alkoxide ratio and viscosity in the sol-gel process were varied to optimize the YSZ coating. The coating morphology and thickness was investigated by SEM and Focused Ion Beam. The surface area increase was measured by BET method with N2 adsorption. TEM was employed to check the interface structure between coating and substrate.

3:10 PM Break

3:30 PM Invited

Properties of Wear Resistant Spray Coatings Based on Nanocrystalline Cermet Precursor Powders: *Thomas Klassen*[1]; Nico Eigen[1]; Xiumei Qi[1]; Eckhard Aust[1]; R¸diger Bormann[1]; Frank G‰rtner[2]; Heinrich Kreye[2]; [1]GKSS Research Center, Powder & NanoTech., Inst. for Matls. Rsrch., Max-Planck-Str. 1, Geesthacht 21502 Germany; [2]University of the Federal Armed Forces Hamburg, Dept. of Mechl. Eng., Holstenhofweg 85, Hamburg 22043 Germany

Thermal spraying of wear-resistant coatings is a common process for the refinement of parts in numerous applications. Properties of the coatings critically depend on their microstructure, which in turn is determined by the microstructure of the initial powders used in the spray process. In this study, novel coatings produced from nanostructured TiC-Ni based cermet powders are evaluated. High-energy milling was optimized to achieve larger quantities of spray powders with homogeneously dispersed hard phase particles with a mean size of about 30 nm. The coatings were processed by HVOF and VPS spraying on low carbon steel substrates and properties are compared to microcrystalline coatings based on agglomerated and sintered powder of similar compositions. The results show that the composition and microstructure of the powder feedstock as well as the spraying conditions have a significant influence on the final coating microstructure. Grain growth during thermal spraying can be largely suppressed by optimized process parameters as well as by additional cooling of the substrates. In view of potential applications, nanostructured thermal spray coatings have been investigated with respect to their tribological properties. Depending on the processing conditions and the kind of wear, the novel layers based on nanostructured precursor powders show comparable or improved wear resistance. Results indicate that the wear mechanism of nanostructured coatings is determined by wedge formation and material delamination. Thus, wear of nanostructured coatings appears to be more homogeneous, and therefore, nanostructured coatings are promising for applications where low surface roughness is required, e.g. in the paper industry.

4:00 PM

Investigations of Microstructure and Properties of DC Sputtered Copper Films Containing Insoluble Molybdenum: *Tai-Nan Lin*[1]; Jinn P. Chu[2]; Guoyi Tang[1]; J. Michael Rigsbee[1]; [1]North Carolina State University, Dept. of Matls. Sci. & Eng., CB 7907, Raleigh, NC 27695 USA; [2]National Taiwan Ocean University, Inst. of Matls. Eng., 2, Pei-Ning Rd., Keelung 202 Taiwan

The microstructure and properties of Cu films containing insoluble Mo in as-deposited and annealed conditions have been studied by DC dual-gun magnetron sputter system. In our previous study, using RF magnetron sputtering, Cu films with Mo concentrations up to 25 at% have been deposited. The Cu-Mo films consist of non-equilibrium supersaturated solid solutions of Mo in Cu and have nanocrystalline microstructures. Compared to RF sputter deposition, Cu-Mo films were DC co-sputtered onto Si (100) substrates. The film compositions were calculated based by the relative sputter rates of both targets and vary from 0 to 50 at% Mo. As-deposited films were subsequently vacuum-annealed at temperatures ranging from 200 to 900°C. To prevent reaction of the coating with Si, a MoN layer was introduced onto the Si before $Cu_{1-x}Mo_x$ deposition. X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM), atomic force microscopy (AFM) and secondary ion mass spectrometry (SIMS) were used to characterize microstructure, texture, and surface morphology.

4:20 PM

Nanopore Formation in B2 Aluminide by Vacancy Clustering: *Tomohide Haraguchi*[1]; Kyosuke Yoshimi[1]; Hidemi Kato[1]; Shuji Hanada[1]; Akihisa Inoue[1]; [1]Tohoku University, Inst. for Matls. Rsrch., 2-1-1, Katahira, Aoba-ku, Sendai 980-8577 Japan

It is confirmed that nanopores which have several tens nm in diameter is formed near surfaces after annealing at 723 K of rapidly-solidified FeAl ribbons with B2-structure. Nanopores are observed after the heat treatment by SEM and AFM, and during in-situ heating experiments by TEM. The pore shape depends on surface orientation. The pore density of surfaces is increased with an increase in Al content, corresponding to the composition dependence of excess vacancy concentration determined by density and lattice constant measurements. An irreversible exothermic peak around 800 K is also observed in DSC cyclic measurements. Thus, the nanopore formation is considered to be due to excess vacancy clustering. Furthermore, similar phenomenon was also observed in B2 NiAl. Such nanopore formation process by vacancy clustering is a unique technique to control surface nanostructure.

4:40 PM

Electromagnetic Properties of Planar Surfaces and Emissivity: Osman Yildirim[1]; [1]Turkish Airforce Academy, R&D Ctr., Hava Harp Okulu, AR-GE Subesi, Yesilyurt, Istanbul, TR 34800 Turkey

As is well-known, dielectric properties of a planar surface have been extensively studied using laboratory techniques. The standard impedance condition is the simplest way of simulating the material properties of a planar surface. This method is only accurate if the coating is very thin or lossy. To improve the accuracy, generalized impedance conditions can be used to simulate the material properties of a planar surface. The accurate solution of a scattering problem via planar surface depends on its thickness, the wavelength of incident wave, and material properties of the surface. Since boundary condition is local in character, accuracy decreases when the surface is illuminated at near grazing angle. Accuracy also decreases if the surface has a curved shape. In this study, the Taylor series method is applied to a planar slab. This method gives reasonable solution even if slab has a curved shape.

Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties: Point Defects, Dislocations and Deformation of Metals, Ceramics and Intermetallics - II & Point Defects, Dislocations and Deformation of Metals, Ceramics and Intermetallics - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Amit Misra, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Harriet Kung, Los Alamos National Laboratory, Materials Science & Technology Division, Los Alamos, NM 87545 USA; Stuart A. Maloy, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Michael Nastasi, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Ricardo Schwarz, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Monday PM Room: 10
March 3, 2003 Location: San Diego Convention Center

Session Chairs: Stuart A. Maloy, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Altaf H. Carim, US Department of Energy, Div. of Matls. Scis. & Eng. USA

2:00 PM Invited

Carbon-Vacancy Interactions in Austenitic Alloys: *R. Gibala*[1]; J. A. Slane[2]; C. Wolverton[3]; [1]University of Michigan, Ann Arbor, MI 48109 USA; [2]Worthington Industries, Columbus, OH USA; [3]Ford Research Laboratory, Dearborn, MI 48121-2053 USA

Several research results suggest the binding energy of carbon-vacancy (c-v) complexes is of the order 40-60 kJ/mol in fcc Fe-base alloys. Data on point-defect anelasticity, self diffusion, high-temperature creep, strain aging, strain-age hardening, radiation damage and point-defect modeling are examined and discussed. Each type of result offers supporting evidence for large c-v binding. Quantitative evaluations of the c-v binding are made from the effect of carbon on the self diffusion of fcc Fe and imply a binding energy of ~40 kJ/mol. First-principles density functional calculations are used to determine more directly the binding energy for nearest-neighbor c-v pairs and give a result of ~35 kJ/mol.

2:30 PM Invited
Atomistic Calculations on the Role of Dislocations in Deformation: *Michael Baskes*[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

Deformation has been the cornerstone of the career of Dr. Terry Mitchell. Understanding the role of dislocations in the deformation process has been his lifelong goal. Over the last few years computational materials science has progressed to the point where it can actually contribute to understanding this complex problem. This presentation will review our recent work in this area for two very different materials of which Dr. Mitchell is quite familiar, molybdenum disilicide and copper. Using a modified version of the Embedded Atom Method (EAM) that includes angular forces, potentials are developed for $MoSi_2$. The potentials describe the structural and elastic properties in reasonable agreement with experiment. These potentials are used to calculate the core structures and resistance to glide for four straight dislocations, which are observed in $MoSi_2$. In contrast to previous calculations in materials with simple crystal structures, such as fcc metals, it was necessary to use molecular dynamics at elevated temperature to obtain any dislocation mobility. It is found that only one of the dislocations, a/2<111>(110) with predominantly edge character, has any significant mobility at reasonable stresses. In one case, the dislocation dissociates into seven partials at high shear strain and the response is asymmetric with respect to the direction of the applied shear strain. Copper is a prototype fcc material. In the bulk material, dislocations dissociate and easily move on {111} slip planes. The calculations presented here will emphasize the interaction of lattice dislocations with grain boundaries under the influence of shear stress. Using an EAM potential we find that dislocations easily glide and become incorporated into existing grain boundaries at very low stress levels. At slightly higher levels of stress, dislocations are nucleated at these grain boundaries. Depending upon grain orientation and size, we see either nucleation of a partial dislocation and its motion across an entire grain leaving a stacking fault or nucleation of a dissociated dislocation and motion of this entity across the grain. This work was supported by the Office of Basic Energy Sciences, US Department of Energy.

3:00 PM Invited
Solution Hardening and Softening: A New Model Based on Double Kink Nucleation on Partials: *Peter M. Anderson*[1]; Terrence Mitchell[2]; Michael I. Baskes[2]; Shao-Ping Chen[3]; Richard G. Hoagland[2]; Amit Misra[2]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210-1179 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., PO Box 1663, MS G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Nucl. Matls. Tech. Div., NMT-16, G721, Los Alamos, NM 87545 USA

Terry Mitchell introduced this group of authors to the interesting experimental observation that alloying elements such as vanadium, niobium, chromium, and aluminum decrease the yield stress of molybdenum disilicide at 500C, but tungsten additions increase the yield stress. Subsequent measurement of stacking fault energies based in the dissociation of 1/2<110> dislocations into two 1/4<110> partials revealed that vanadium, niobium, chromium, and aluminum all lower the stacking fault energy while tungsten increases the energy. First principles atomic calculations confirm this experimental trend in stacking fault energy. Armed with this information, the group developed a model of dislocation mobility based on the nucleation of kink pairs along the leading partial. This presentation will discuss the model features and resulting predictions of yield stress versus alloying element in molybdenum disilicide. As remarked by Terry Mitchell, the agreement between model and experiment is ìquite remarkableî.

3:30 PM Break

4:00 PM Invited
Influence of Interstitial Content, Strain Rate, and Temperature on the Mechanical Response of Tantalum: *George T. Gray*[1]; Shuh Rong Chen[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA

Terry Mitchell has made seminal contributions to the fields of work hardening, interstitial content, deformation twinning, substructure evolution effects on the mechanical response in refractory metals, to name a few. This talk will discuss experimental results illustrating the role of interstitial content, strain rate, temperature, and substructure response on the dynamic mechanical behavior of tantalum. Increasing interstitial content and strain rate is shown to significantly increase the dynamic flow strength of Ta. The pronounced influence of the Peierls-Nabarro stress on the mechanical response of tantalum and its hardening during shock prestraining is presented. Finally, the defect evolution in tantalum is discussed as a function of temperature and strain rates ranging from quasi-static to shock loading. Work conducted under the auspices of the US Department of Energy.

4:30 PM Invited
Rapid Formation of Stacking Faults by Electron-Hole Recombination and Degradation of 4H-SiC High Power Devices: *Pirouz Pirouz*[1]; Augustinas Galeckas[2]; Jan Linnros[2]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., 510 White Bldg., 10900 Euclid Ave., Cleveland, OH 44106-7204 USA; [2]Royal Institute of Technology (KTH), Dept. of Microelect. & Info. Tech., Kista, Stockholm SE 16440 Sweden

Bipolar devices fabricated from 4H-SiC for high-power applications, such as megawatt PiN diodes, degrade rapidly under forward biasing. This degradation has been found to be associated with a rapid increase in the density of stacking faults throughout the active region of the device. The problem is likely a phenomenon in semiconductors called ìRecombination-Enhanced Defect Interactionî (REDI) whereby the generation of excess minority carriers and subsequent electron/hole recombination at a defect site leads to the dissipation of the recombination energy to promote a certain defect reaction. In this work, REDI has been investigated in 4H-SiC diodes by determining the energy levels of partial dislocations and stacking faults. In particular, by measuring the dislocation velocity in the presence and absence of excess minority carriers, the energy level of non-radiative point defects on the dislocation lines has been determined. Following the presentation of experimental results, the REDI mechanism will be discuss ed in terms of a particular point defect on the partials, proposed to be responsible for trapping the carriers.

5:00 PM Invited
Characterization of Plastic Deformation Around Vickers Indents on Monocrystalline Substrates: *Pedro D. Peralta*[1]; Reina Martinez[1]; Robert Dickerson[2]; Pat Dickerson[3]; [1]Arizona State University, Mech. & Aeros. Eng., Main Campus, MC 6106, Tempe, AZ 85287-6106 USA; [2]Los Alamos National Laboratory, MST Div., MST-8, MS G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, MST Div., MST-6, MS G770, Los Alamos, NM 87545 USA

The deformation surrounding Vickers indents on pure copper and MoSi2 monocrystals has been characterized for two orientations of the indenter diagonals using electron and scanning probe microscopy. Orientation Imaging Microscopy (OIM) was used to map changes on the local crystallographic orientation of the surface surrounding the indents, Transmission Electron Microscopy (TEM) was employed to study the dislocation structure and the surface topography around these indents was characterized using Atomic Force Microscopy (AFM). The results indicate that sink-in and pile-up behavior depend strongly on the in-plane crystallographic orientation of the diagonals of the indent and is related to local multiplicity of slip. Regions with multiple slip show larger lattice rotations and surface sink-in due to local hardening, whereas lower density of dislocations leads to pile-ups. The implications of this behavior on mechanical property measurement from microhardness are discussed. Research supported under NSF grant #CMS-0084948 and by Los Alamos National Laboratory.

The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research: Mechanical Properties:Theory and Experiment

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Japan Institute of Metals, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Julia R. Weertman, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Katherine T. Faber, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Morris E. Fine, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Wayne King, Lawrence Livermore National Laboratory, San Ramon, CA 94583-2496 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Ben Mori, Tokyo 168-0081 Japan

Monday PM Room: 9
March 3, 2003 Location: San Diego Convention Center

Session Chairs: Morris E. Fine, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA; D. N. Seidman, Northwestern University, Matls. Sci. & Eng. Dept., Evanston, IL 60208 USA

2:00 PM Invited

What and How are Dislocations Created During Plastic Deformation?: *Masao Doyama*[1]; Y. Kogure[1]; T. Nozaki[1]; Y. Kato[1]; [1]Teikyo University of Science and Technology, Uenohara, Yamanashi 409-0193 Japan

By the plastic deformation of single crystals the question ìwhere and how dislocations are createdî has been an important problem to solve lattice defects. Copper single crystals with and without notch were prepared. The specimens were pulled, compressed and bent, using the molecular dynamics with an embedded atom potential. When a specimen with a notch was pulled, partial dislocations were created near the tip of the notch as we expected. When a specimen without notch was pulled partial dislocations were created near the grip at the ends of the specimen and then other dislocations were created compensating the bend of the specimen. When specimens were compressed, many partial dislocations were created with or without a notch. For a bending, partial dislocations were created near the compression surface, particularly near the wrinkles of the slip planes. It was quite hard to create partial dislocations on a smooth extended slip plane. Thermal vibrations may start partial dislocations near the surfaces. In case of copper always partial dislocations were created instead of complete dislocations because of the low value of stacking faults. These are clearly shown using the simulation of X-ray Lang method.

2:30 PM Invited

Deformation of PST Crystals With and Without Constraint: Kyosuke Kishida[2]; Toshiyuki Morita[3]; Haruyuki Inui[1]; *Masaharu Yamaguchi*[1]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan; [2]National Institute for Materials Science, Tsukuba 305-0047 Japan; [3]Daido Steel Company, Ltd., Nagoya 457-8545 Japan

Polysynthetically twinned (PST) crystals of gamma/alpha-2 two-phase alloys were deformed in compression with and without constraint and in tension without constraint in a wide temperature range. Deformation structures in gamma domains of six different orientation variants were examined by transmission electron microscopy and operative slip and/or twinning systems were determined. On the basis of these results, macroscopic plastic strain tensors of PST crystals were derived. They were found to well describe the anisotropic deformation of PST crystals. The anisotropy of deformation is typically observed when the loading axis is parallel to the lamellar boundaries. Strain continuity at domain and lamellar boundaries is one of the most important factors determining not only operative slip and/or twinning systems for each orientation variant in the gamma phase but also the tensile properties of PST crystals.

3:00 PM

Dislocation-Free Plastic Deformation of Crystalline Metals: *Michio Kiritani*[1]; [1]Hiroshima Institute of Technology, Sch. of Eng., Miyake 2-1-1, Saeki-ku, Hiroshima 731-5193 Japan

Plastic deformation of crystalline metals without involving dislocations is confirmed to occur during elongation of thin films to fracture, and possibility of dislocation-free deformation is suggested during high-speed deformation of bulk samples. Systematically organized experiemntal results are enough to consider new mechanism. They are, in the case of thin films, confirmation of the absence of the operation of dislocations, formatin of anomalously high density of vacancy defects, high internal stress more than 10 GPa measured from elastic strain. In the case of high-speed deformatin of bulk samples, they are the transition of heterogeneous distribution of dislocations at low-speed deformation to random distribution at high-speed deforamtion, production of high-density of vacancy clusters by high-speed deformation, and estimated stress during deformatin at high-speed more than several 10 GPa. Discussions are made on atomistic mechanism of new mode of deformation without dislocation.

3:30 PM

Correlations Between High-Temperature Deformation and Microstructure: *Farghalli A. Mohamed*[1]; [1]University of California, Cheml. Eng. & Matls. Sci., 916 Eng. Tower Bldg., Irvine, CA 92697-2575 USA

High-temperature deformation in materials is often highly sensitive to microstructure. Accordingly, seeking a correlation between mechanical behavior and microstructure represents an effective approach that can clarify issues or settle controversies related to high-temperature deformation. While there are several microstructural techniques that can be adopted to explore the presence of such a correlation, transmission electron microscopy (TEM) has proven to be a powerful tool that can provide guiding information regarding the details of the microstructure that accompany deformation. This paper discusses examples of the application of TEM to several areas of high-temperature deformation, which include creep behavior in solid-solution alloys, stress changes during creep, creep behavior in powder metallurgy SiC-Al composites and their matrices, and creep behavior in refractory materials.

3:50 PM Invited

Environmental Effects on Long Fatigue Cracks: *A. K. Vasudevan*[1]; R. L. Holtz[2]; I. W. Kang[2]; K. Sadananda[2]; [1]Office of Naval Research, Matls. Div., 800 N. Quincy St., Arlington, VA 22217 USA; [2]Naval Research Laboratory, Matls. Tech. Div., Washington, DC 20375 USA

In the Unified Approach to fatigue crack growth rates, we use two-parameter approach involving amplitude ΔK and peak stress intensity, K_{max}. By considering these two parameters it is shown that one can analyze most of the fatigue phenomena without the need of crack closure concept. Effects of environment naturally manifest through K_{max} driving force. These effects are dominant at low ΔK values or at slow crack growth rates. Hence, they play a significant role in determining total fatigue life of a component. A systematic analysis of the effects of superposition of environmental effects of fatigue crack growth is made and the behavior is classified. A detailed discussion will be presented taking experimental examples from the literature.

4:20 PM

Interaction of Deformation Induced Dislocations on Recrystallization Behavior in Aluminum Bicrystal with Originally Twin Boundary: *Keizo Kashihara*[1]; Fukuji Inoko[2]; [1]Wakayama National College of Technology, Dept. of Mechl. Eng., 77 Noshima, Nada, Bobo, Wakayama 644-0023 Japan; [2]The University of Tokushima, Dept. of Mechl. Eng., 2-1 Minamijosanjima, Tokushima 770-8506 Japan

Deformation and recrystallization experiments are performed using an aluminum bicrystal specimen with twin boundary at initial orientation. The bicrystal specimen was deformed to 48% strain, and annealed under certain condition. The coexistence of the strain induce boundary migration (SIBM) and the <111> rotated recrystallized grains (RGs) is recognized. The SIBM takes place along an original grain boundary (GB), whereas the <111> rotated RGs are formed at deformation bands (DBs) away from the GB. The mechanisms of the SIBM and the <111> rotated RGs are argued based on the interactions of dislocations introduced into their formation sites. Especially, focusing on the SIBM, it is found the accumulation of the edge dislocations substantially generates the SIBM, whereas the screw dislocations are difficult to contribute to it because of the GB cross slip inheriting from the special geometry between the component grains.

4:40 PM

Micromechanics of High-Rate Compression Failure of Ceramics: *Sia Nemat-Nasser*[1]; Sai Sarva[1]; [1]University of California-San Diego, CEAM, 4207 EBUI, 9500 Gilman Dr., La Jolla, CA 92093-0416 USA

Ceramicsí failure modes range from brittle to ductile depending on the deformation conditions. The micro-mechanisms of their compression failure are examined over a broad range of deformation rates, from quasi-static to ballistic strain rates. Recent advances in experimental techniques to study these phenomena are presented. Data on damage initiation and evolution in ceramic armor materials are used to decipher the essential feature of failure phenomena. Under moderate confining pressures and at moderate deformation rates, brittle failure involves initiation of micro-cracks at dominant micro-flaws and pre-existing micro-cracks, and their subsequent interactive growth, leading to axial splitting, faulting or a mixture of brittle-ductile failure. Under great confining pressures, common in ballistic impact on the other hand, classical crack-growth models seem inadequate for representing the actual failure initiation and evolution. Computational simulations of the early stages of impact response of ceramics show development of extremely high shear stresses within the target ahead of the projectile, a state conducive to pulverization. Transmission electron microscopy of recovered Al_2O_3 powder of impact-penetrated ceramics shows extensive twinning with sub-micron spacing. Dynamic compression of ceramics has also shown extensive dislocation activities. These observations are used to identify potential mechanisms of pulverization under high compressions.

5:00 PM

Relationship Between Stress-Strain Potentials and Maximum Shear and Zero Shear Trajectories for Mode III Crack: *Johannes Weertman*[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Camous Dr., Evanston, IL 60208 USA

We show in this paper that for the mode III crack (or any antiplane strain problem) the trajectories of constant stress potential and constant strain potential are the trajectories across which the resolved shear stress is either zero or has its maximum value. The analysis offers an alternative and more simple check to the condition that for a power law hardening material these trajectories satisfy the radii of curvature condition $d(1/R_F)/dF - m d(1/R_T)/dT + (1- m)(1/R_F)(1/R_T) = 0$ where m is the work hardening coefficient and R_F and R_T are the radii of the trajectories (labeled finger and thumb trajectories) and dF and dT are differentials along the trajectories.

The MPMD Fourth Global Innovations Symposium: Energy Efficient Manufacturing Processes: Novel Molten and Semi-Molten Materials Processing

Sponsored by: Materials Processing and Manufacturing Division, *Program Organizers:* Toni G. Marechaux, National Research Council, National Materials Advisory Board, Washington, DC 20418 USA; Iver E. Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Chris Cockrill, DOE Seattle Regional Office, Seattle, WA USA

Monday PM — Room: 5A
March 3, 2003 — Location: San Diego Convention Center

Session Chairs: Iver E. Anderson, Iowa State University, Ames Lab., Ames, IA 50011-3020 USA; Toni Grobstein Marechaux, National Research Council, Natl. Matls. Advisory Board, Washington, DC 20001 USA

2:00 PM Welcome

2:05 PM Invited
Energy Efficient Near-Net Shape Manufacturing - Semi-Solid Processing Routes: *D. Apelian*[1]; M. Makhlouf[1]; [1]Worcester Polytechnic Institute, Metal Procg. Inst., 100 Institute Rd., Worcester, MA 01609 USA

When rheocasting was first discovered, it was believed that one had to break up the dendritic structure during the freezing process either by mechanical stirring or via magneto-hydrodynamic forces to obtain a cellular structure. In the recent past, we have discovered that one may obtain a refined SSM structure without breaking up the dendritic structure, but rather by creating an environment where copious nucleation can occur near the liquidus temperature of the alloy, and with limited growth of the formed nuclei. Essentially, SSM structures develop by controlling the nucleation and growth processes during the early stages of freezing. The various SSM processing routes available will be reviewed from physical metallurgy considerations, as well as reviewing structural evolution during processing. The emphasis will be on the attainment of energy efficiency by the use of SSM processing. Challenges and opportunities for SSM processing will also be presented.

2:30 PM Invited
Spray Forming: An Energy Saving and Process Efficient Technique: *Enrique J. Lavernia*[1]; Yaojun Lin[1]; Yizhang Zhou[1]; [1]University of California-Davis, Davis, CA 95616 USA

Spray forming technique has attracted considerable attention, not only from the standpoint of metallurgical advantages, but also as a potential for near net-shape fabrication with energy and cost savings. In this paper, principles and processes of spray forming to manufacture different geometry parts (i.e., tubes, rings, and plates/strips/sheets) are reviewed and compared with those by conventional techniques. Less processing steps are required in spray forming, compared with conventional techniques. For seamless tube/pipe or ring production, the reduction of processing steps is attributed to the forming method of seamless tubes/pipes or rings in spray forming and microstructural advantages (fine grains/second phases, low micro-segregation, absence of macro-segregation and high density) in as-sprayed materials. For production of plates/strips/sheets, the reduction of processing steps is due to microstructural advantages in as-sprayed materials. As a result of reduction of processing steps and the associated reduction of equipment investment, energy and production cost can be saved when spray forming is used to fabricate the preceding products. The advantages of spray forming in energy and cost savings over powder metallurgy are also analyzed for production of highly alloyed materials. In addition, spray forming can be also utilized to fabricate complex molds/dies. Compared with the conventional fabrication procedure of molds/dies, production cost and time can be saved significantly. Finally, disadvantages of spray forming are analyzed and the approaches to further improve microstructure and mechanical properties of spray-formed materials are discussed.

2:55 PM Invited
New Atomisation Process for Metal Powder: *Volker Uhlenwinkel*[1]; L. Achelis[1]; S. Sheikhaliev[1]; S. Lagutkine[1]; [1]University of Bremen, Inst. for Werkstofftechnik, Bremen 28359 Germany

A new atomisation process has been developed which combines pressure and gas atomisation. The melt leaves the pressure nozzle as a hollow cone with a thin film thickness. After this pre-filming step, the film is atomised by a gas stream delivered by a ring nozzle. The objectives of this new atomisation process are to achieve a smaller size distribution and less specific gas consumption compared to conventional gas atomisation. Both leads to a higher efficiency and less costs. First results on the atomisation of tin and tin alloys will be shown. The mass median diameter from different experiments are between 20 and 100 µm. Standard deviations of far below 2.0 have been achieved.

3:20 PM Break

3:35 PM Invited
LENSô Processing for Enhanced Manufacturing Efficiency and Energy Savings: *John E. Smugeresky*[1]; Richard Grylls[2]; David M. Keicher[2]; [1]Sandia National Laboratories, 8724, MS 9402, Bldg. 941-1191, Livermore, CA 94551-0969 USA; [2]Optomec Design Company, Albuquerque, NM 87123 USA

Laser Engineered Net Shaping (LENSô) is an energy efficient additive forming process for structural materials that reduces the need for machining and the amount of starting material to make parts. It is a model based, paperless technology requiring no tooling or material removal to achieve useful shaped functional objects. It has an inherent capability to engineer composition, microstructure, and properties simultaneously while shaping the material. Results have demonstrated the ability to enhance mechanical properties via rapid solidification and refinement of the microstructure, like strength enhancement without loss of ductility. Each of these features makes a contribution to reducing energy usage in the manufacture of structural parts. Another benefit is the potential to enable the use of todayís advanced material systems, especially for high temperature applications, where formability of the new materials have limited their applications. We will review the essential features of the process, examples of savings in number of processing steps for stainless steels, demonstrating enhanced properties by control of the processing conditions. Work supported by the Optomec NSF Project and the US Department of Energy under contract DE-AC04-94AL85000.

4:00 PM
Finite Element Modeling of Ablation Phenomena and Thermal Stress Evolution a Unique Application of Dual Laser Cutting of Ceramics: Ravindra Akarapu[1]; *Ben Q. Li*[2]; Al Seagull[1]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Pullman, WA 99164 USA; [2]Washington State University, Sch. of Mechl. & Matls. Eng., Sloan 207, Pullman, WA 99164 USA

A 3-D numerical model is developed for the ablation phenomena and thermal stress evolution during dual laser cutting of alumina. Dual laser machining is of interest because of the potential to reduce and stall premature fractures that often occur as the supporting section is continuously reduced by the cut. The dual beam numerical model development is based on the finite element solution of thermal conduction with ablation resulting from an applied laser source. To model the removal of materials due to ablation, a fixed grid finite element method is employed by which material elements at temperature above the melting point are deleted from further calculations. Compared with the front tracking method by which the ablation moving interface is precisely tracked in time and which is useful for simple geometries, the present method has an advantage of modeling more complex ablation geometries such as those induced by a dual laser power source. The thermal model is integrated with a stress model to predict the evo lution of thermal stresses, which are developed during laser cutting as a result of strong temperature gradient near the laser source. Finite element model development and implementation are discussed and computed results are presented for an industrial dual laser cutting system for ceramic materials.

4:25 PM Invited
US Energy Requirements for Aluminum Production: Historical Perspective, Theoretical Limits, and New Opportunities: *William T. Choate*[1]; John A.S. Green[2]; [1]BCS, Inc., 5550 Sterrett Place, Ste. 306, Columbia, MD 21044 USA; [2]Aluminum Consultant, 3712 Tustin Rd., Ellicott City, MD 21042-4826 USA

The US aluminum industry is the worldís largest, handling over 10.7 million metric tons of metal and producing about $39 billion in products and exports annually. Energy reduction in the US aluminum industry is the result of technical progress and the growth of recycling, and these two factors have contributed to a total of 58% energy reduction in the past forty years. Nonetheless, by many measures, aluminum remains one of the most energy intensive materials to produce and is the largest consumer of energy on a per-weight basis of all industries. The aluminum industry has large opportunities to further reduce its energy intensity. This talk will present an historical perspective of the subject, will speculate on the theoretical limits to energy efficiency, and will describe current practices. This knowledge can help identify and understand process areas where significant energy reductions and environmental impact improvements can be made.

4:50 PM
Projects that Reduce Energy in the Metals Industry: *Robert DeSaro*[1]; [1]Energy Research Company, 2571-A Arthur Kill Rd., Staten Island, NY 10309 USA

Partnerships among industry and the Department of Energy (DOE) can have a multiplying affect in bringing innovative technology to the marketplace with commensurate energy savings, productivity increases and emissions reductions. Plant assessments are a good example of how DOEís resources are used to address plant improvements where the plant personnel are too overwhelmed or lack the expertise to tackle. These assessments have produced a minimum of $1 million energy savings yearly for the participants; savings that would never have materialized otherwise. Inert anode development is an example of a long-term technology development that few aluminum companies would conduct themselves. This development could lead to greenhouse gas emissions reduction of 9.1 million metric tons of carbon equivalent in the US, and an energy reduction up to 25%, or 5.6 x 1010 kWhr annually. Laser diagnostics for aluminum companies is an example of developmental work the end-users has neither the capacity nor the interest in developi ng, yet could greatly benefit from. This paper explores these issues with case studies.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Kinetics & Thermodynamics

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors
Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Monday PM	Room: Solana
March 3, 2003	Location: San Diego Marriott Hotel

Session Chairs: Derek J. Fray, University of Cambridge, Matls. Sci. & Metall., Cambridge CB2 3QZ UK; Hong Yong Sohn, University of Utah, Metallurgl. Eng., Salt Lake City, UT 84112-0114 USA

2:00 PM Keynote
Thermodynamics and Fluid-Solid Reaction Kinetics: Effects on the Rate and Activation Energy: *Hong Yong Sohn*[1]; [1]University of Utah, Metallurgl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

The purpose of this article is to critically and quantitatively analyze the effect of chemical equilibrium on the overall rates of fluid-solid reactions. It is shown through a mathematical analysis that a reaction with a small equilibrium constant (a positive standard free energy of reaction) is more likely to be rate-controlled by pore diffusion or mass transfer than a reaction with a large equilibrium constant. The overall reaction rate of the former also tends to be slow. Furthermore, the apparent activation energy of such a reaction approaches the standard enthalpy of reaction, rather than the true activation energy of the chemical reaction. The Law of Additive Reaction Times developed by the author is shown to apply to equilibrium-limited fluid-solid reactions and to be useful in quantitatively analyzing the behavior of such reactions.

2:35 PM
Kinetics of Gaseous Reduction of Mn3O4: *Rodney J. Ishak*[1]; Tor Lindstad[2]; [1]Norwegian University of Science and Technology, Matls. Tech. & Electrochem., Alfred Getz vei 2, Trondheim N-7465 Norway; [2]SINTEF, Matls. Tech., Alfred Getz vei 2, Trondheim N-7465 Norway

The reduction of Mn3O4 to MnO has been investigated in a thermobalance apparatus.The charge was composed of manganese ore, decomposed by heating to mainly Mn3O4 and coke. The material was charged to a crucible, which was suspended in the balance. 100% CO-gas was distributed through a grid in the bottom of the crucible. Two sizes of ores were used; 2,4-4,8 mm and 6,7-9,5 mm. Coke particles was in the size range 4,8-6,7 mm. Experiments were conducted in the 900-1100∫C temperature range. The composition of the product gas was monitored continuously. External mass transport between bulk gas and particle surface does not seem to limit the rate of reduction. Assuming that both diffusion through product layer and chemical reaction at the interface between MnO and Mn3O4 are rate controlling the date was tried both in a shrinking core model and in a grain model using regression. The grain model gives the best physical description of the process, because the reactant is a porous media. With this model the effective diffusivity was calculated to be in the 1,5-4?10-5 m2/s range. Pore size was mainly between 0,1 and 10 □m.

3:00 PM
Reaction Kinetics of Some Carbonaceous Materials with Carbon Dioxide: *M. Kawakami*[1]; T. Ohyabu[1]; T. Takenaka[1]; S. Yokoyama[1]; [1]Toyohashi University of Technology, Dept. Production Sys. Eng., Tempakucho-aza-Hibarigaoka 1-1, Toyohashi 441-8580 Japan

In relation to the designing of new-type blast furnace, the reaction of carbonaceous materials with CO2 is expected at as low temperature as possible. In order to get a highly reactive carbonaceous material, the reaction kinetics was investigated by measuring such quantities as the reaction rate from weight loss, the amount of CO adsorption and specific surface area by BET method. The reaction rates were in the order of Bintyo char, metallurgical cokes, graphite and glassy carbon. If the rate was converted to the rate per unit area, however, that of Bintyo char was smaller than that of graphite. Thus, it is suggested that the rate of Bintyo char is influenced by the pore diffusion of CO2. The rate had a positive relation with the amount of CO absorption, showing that the rate determining step would be the desorption of CO from the active side on the surface.

3:25 PM
Vapor Pressure Measurements for the $FeCl_2$-$ZnCl_2$ System by the Transpiration Method: *Sang-Han Son*[1]; Fumitaka Tsukihashi[2]; [1]The University of Tokyo, Matls. Eng., 7-3-1, Hongo, Bunkyo, Tokyo 113-8656 Japan; [2]The University of Tokyo, Grad. Sch. of Frontier Scis., 7-3-1, Hongo, Bunkyo, Tokyo 113-0033 Japan

The vapor pressure of $ZnCl_2$ in the $FeCl_2$-$ZnCl_2$ system was measured by the transpiration method at 873K and 917K. The vapor pressure data were interpreted to indicate the formation of complex ions in the $FeCl_2$-$ZnCl_2$melts. It is presumed that the complex molecule $FeZnCl_4$ as well as $FeCl_2$ and $ZnCl_2$ exists in the vapors in equilibrium with molten $FeCl_2$-$ZnCl_2$ mixtures. The activities in the $FeCl_2$-$ZnCl_2$ system are shown a negative deviation from Raoultís law. On the basis of those data, thermodynamic properties for the $FeCl_2$-$ZnCl_2$ system were discussed.

3:50 PM Break

4:10 PM Invited
The Kinetics of the Oxidation of Zinc Vapour by Carbon Dioxide and Water Vapour on Quartz, Zinc Oxide, Sinter and Coke Substrates: Antony Cox[1]; *Derek J. Fray*[1]; [1]University of Cambridge, Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

The re-oxidation of zinc vapour in the Imperial Smelting Process is a very important step in the overall process. There have been various studies of the reactions between zinc vapor and oxidising gases and, generally, the same pattern is observed in that at low temperatures a massive deposit is formed whilst at higher temperatures or higher carbon monoxide pressures, a much finer deposit is observed. This work reports new studies on the oxidation of zinc vapour on sinter and carbon so as to reproduce the conditions found in the shaft of the Imperial Smelting Furnace. It was found that the zinc oxide deposited preferentially on coke and sinter rather than on silica. The presence of sulfur resulted in the formation of zinc sulfide on top of the zinc oxide. These observations are discussed in terms of the reactions taking place in the shaft of the zinc blast furnace.

4:40 PM
Effect of P_2O_5 or Na_2O Addition on the Reaction Rate of CO_2 Dissociation with Fe_xO Containing Molten Oxides: *Hiroyuki Matsuura*[1]; Fumitaka Tsukihashi[1]; [1]The University of Tokyo, Grad. Sch. of Frontier Scis., 7-3-1 Hongo, Bunkyo, Tokyo 113-0033 Japan

The kinetic data of CO-CO_2 reaction with molten oxides are important for the analysis of refining process of metals. The rate constant has been expressed as a function of the composition of ferrous and ferric oxides. The rate constant is affected by the addition of small amount of P_2O_5 and Na_2O which are a surface active compounds in the molten oxides. In the present work, the reaction rate of CO_2 dissociation on the surface of Fe_xO-base molten oxides containing P_2O_5 or Na_2O was measured by isotope exchange method at 1773K. The effect of P_2O_5 or Na_2O addition on the reaction rate was investigated. The rate constant decreased with increasing P_2O_5 content and the residual rate constant was observed at high P_2O_5 content. The rate controlling step of CO_2 dissociation was discussed.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Aqueous and Electrochemical Processing I

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Monday PM Room: Pacific
March 3, 2003 Location: San Diego Marriott Hotel

Session Chairs: Yasuhiro Awakura, Kyoto University, Dept. of Matls. Sci. & Eng., Kyoto, 606-8501 Japan; Zhang Duomo, Central South University, Metall. Sci. & Eng., Changsha, Hunan 410083 China

2:00 PM Invited
Preparation of CdS Nanoparticles by Hydrochemical Method and their Surface Modification: *Zhang Duomo*[1]; Li Qihou[1]; [1]Central South University, Metall. Sci. & Eng., Zuojalong, Changsha, Hunan 410083 China

A novel homogenous precipitation method, including microwave heating, spray feeding, solution conductivity controlling and freeze drying, was developed for the preparation of CdS nanoparticles. The effects of precipitation conditions on the structures and morphologies of CdS particle were discussed. Under the experimental conditions, blende-type CdS was obtained in natrate or sulphate solution, and wurtzite-type CdS was obtained in chloride solution. After solvent-thermal treatment, the amorphous CdS particle, which produced from high concentration solution, turn to double cone or tetrahedron shape. In the precipitation process, the bifunctional thiols of CH3CSNH2 modified the surface of CdS particles and the induced counter ion AOT-SO3- increased the stability and dispersion of CdS particles. The produced particles (4nm to 100nm) can be used to prepare nonlinear optics and self-assemble catalysts.

2:35 PM
Dispersion Characteristics from Two Types of Bubble Generators Commonly Used in Flotation Columns: *Ramiro G. Escudero*[1]; Francisco J. Tavera[1]; [1]Universidad Michoacana de San Nicol·s de Hidalgo, Instituto de Investigaciones Metal˙rgicas, Dept. de Metalurgia Extractiva, Santiago Tapia 403, Morelia, Michoac·n 58000 MÈxico

Over almost forty year has past since the introduction of column flotation. Column technology has grown into several non-mineral applications such as effluents treatment, soils recovery, and de-inking of recycled paper among others. Recent studies have demonstrated that particular characteristics of the dispersion (bubble size, gas holdup, bubble surface area flux), are needed for a given flotation column duty. In this work, two kind of spargers were tested (i.e., internal and external), using a 4 inch column lab, in a two phase system. Results shown the external sparger provides a dispersion with characteristics that include those produced by the two internal spargers.

3:00 PM
Formation Behavior and Corrosion Characteristics of Anodic Films on Mg-Al Alloys in NaOH Solutions: *Seong-Jong Kim*[1]; Masazumi Okido[1]; Yoshihiro Mizutani[1]; Ryoichi Ichino[1]; Shoji Tanikawa[2]; Saori Hasegawa[2]; [1]Nagoya University, Grad. Sch. of Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan; [2]Nakanihon Die Casting Company, Ltd., Kakamihara 504-0957 Japan

Magnesium is easy to recycle because impurities are easily removed. Recently, there has been great interest in using Mg in automobile parts, mobile personal computers, etc. However, Mg must be surface treated to prevent corrosion, since it is very active. One of the most efficient surface treatments of Mg is chromate-conversion coating; however, this method causes many problems for the environment, humans, and recycling. Therefore, we studied a non-chromate method of anodizing Mg in NaOH solutions. In this study, the formation behavior and corrosion characteristics of anodic oxide films on pure Mg and Mg-Al alloys were investigated, focusing on the effects of anodizing potential, Al content, temperature, and NaOH concentration. Pure Mg and Mg-Al alloys were anodized for 10 min at 3, 10, 40, and 80 V in NaOH solutions. Mg(OH)2 was generated by an active dissolution reaction at the specimenís surface, and product was affected by temperature, while MgO generation increased with NaOH concentration. Moreover, the current density after anodizing for 10 min at a constant potential decreased with increasing Al content in Mg-Al alloys.

3:25 PM
Electrochemical Couple Behavior of Manganese Dioxide with Ferrous/Ferric in Acidic Chloride Medium: *Guo Xueyi*[1]; Li Qihou[2]; Huang Kai[2]; Zhang Duomo[2]; Qiu Dingfan[3]; [1]The University of Tokyo, Ctr. for Collaborative Rsrch., Komaba 4-6-1, Meguro-Ku, Tokyo 153-8505 Japan; [2]Central South of University, Col. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China ; [3]Beijing General Institute of Mining & Metallurgy, Beijing 100044 China

The electrochemical couple behavior of manganese dioxide with ferrous/ferric in acidic chloride medium was investigated. It was found that the dissolution of MnO2 is due to the electrochemical couple between the MnO2/Mn2+ and Fe2+/Fe3+ in the solution. The driving force for the electrochemical couple reaction originates from their rest potential difference. The reduction of MnO2 is electrochemically kinetic controlled, whereas the conversion of Fe2+ to Fe3+ is diffusion control. The factors, including the Mn2+, Fe2+, Fe3+, Cl- concentration, pH of the solution, and the temperature, have much influence on the reaction rate of the coupled process. Further, the kinetic equation for the couple reactions was derived in term of Butler-Volmer electrochemical Equations and it was found that the theoretical analysis was quite consistent with the experimental results. This study will be useful to guide the practical leaching of manganese nodule from Deep Ocean and manganese dioxide ore in land.

3:50 PM Break

4:05 PM Invited
Electrodeposition of Thin-Layered CdTe Semiconductor from Basic Aqueous Solutions: *Kuniaki Murase*[1]; Tetsuji Hirato[1]; Yasuhiro Awakura[1]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Yoshida-hommachi, Sakyo-ku, Kyoto 606-8501 Japan

Cadmium telluride (CdTe) semiconductor has been well-investigated for n CdS/p-CdTe heterojunction solar cell material, since its direct band gap of 1.44 eV is suitable for energy conversion from sunlight into electricity. In this talk, a new electrochemical processing of thin-layered CdTe using ammoniacal basic aqueous electrolytes is reviewed. The cathodic electrodeposition of stoichiometric CdTe with a flat and smooth surface morphology took place from the basic electrolytes at potentials positive of the Nernst potential for bulk-Cd deposition and negative of that for bulk-Te. In this potential region, deviation from stoichiometric composition of CdTe electrodeposited was controllable by the Cd(II)/Te(IV) concentration ratio, pH, or concentration of ammonia, a complexing agent, of the electrolytes. These deposition behaviors were well accounted for in terms of potential-pH diagram for the Cd-Te-NH_3-H_2O system calculated by a combination of those for Cd-NH_3-H_2O and the Te-H_2O systems.

4:40 PM
Cation Exchange Properties of Zeolites Obtained from Coal Fly Ash by Alkali Hydrothermal Treatment: *Norihiro Murayama*[1]; [1]Kansai University, Dept. of Cheml. Eng., Fac. of Eng., 3-3-35 Yamatecho, Suitashi, Osaka 564-8680 Japan

Syntheses of zeolitic materials are carried out from coal fly ash by alkali hydrothermal treatment, as part of the reuse and recycle technologies of coal ash. Cation exchange capacity and acid resistance, which are very important in the practical use of cation exchanger in aqueous solution, are measured for the reaction products obtained from coal fly ash. Cation exchange properties of the reaction products are investigated for various cations such as K+, Na+, NH4+, Ca2+, Mg2+, Pd2+, Cd2+ and so on. The change in crystallization degree and surface texture are noticed before and after cation exchange operation. From these results, the characteristic and its mechanism of cation exchange are clarified for the various zeolitic materials obtained from coal fly ash.

5:05 PM
The Morphology and Size Control of Cobalt Particles Produced from Concentrated Aqueous Solution: Chen Song[1]; Liu Zihong[1]; Ai Kan[1]; [1]Central South University, Sch. of Metall., Yuelunanlu, Changsha, Hunan 410083 China

Cobalt basic carbonate particles were precipitated from 1.0mol cobalt nitrate or cobalt sulfate solution with ammonia carbonate. By controlling the zeta-potential of particles and the ironic strength of the solution, the uniform particles with ellipsoid or rod shapes were obtained. During the precipitation process, the interface intensities were diminished to the minimum, and therefore the Oswald aging and secondary nucleation were both inhibited. The morphology and size control by zeta-potential seems to the result from thermodynamics rather than classic crystalline kinetics. The particle sizes were in the range of 0.3-0.4 micrometer. After thermolysis, the spindle-type Co3O4 particles were produced. The morphologies of Co3O4 particles were similar to that of their precursors.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: General I

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Monday PM	Room: Point Loma
March 3, 2003	Location: San Diego Marriott Hotel

Session Chairs: G. A. (Tony) Eltringham, BHP Billiton, Houston, TX 77056 USA; Takahiko Okura, Nikko Techno Service Company Ltd., Tokyo 105-0001 Japan

2:00 PM Keynote
Production of Elemental Sulphur from Non-Ferrous Smelter Gas: *Takahiko Okura*[1]; [1]Nikko Techno Service Company, Ltd., 10-1 Toranomon 2 Chome, Tokyo 105-0001 Japan

Most of non-ferrous raw materials are associated with sulphur. To extract the metals, the sulphur is recovered, typically as sulphuric acid and liquid SO2. On the other hand, natural gas may contain over 20% of hydrogen sulphide (H2S) and a huge quantity of elemental sulphur is produced together with oil refining in the world. A large fraction of the sulphur mined with natural resources is eventually returned to the earth as fertilizers, by-products of desulphurisation and neutralisation processes, or as fugitives to the atmosphere. However, the supply and demand for the sulphur is in the imbalance. Such situation urges to develop a new smelting process without sulphuric acid production. In this paper, the global sulphur balance and the technologies that recover the elemental sulphur are briefly reviewed. The core of the paper is a proposal to convert SO2 gas to the elemental sulphur form using organic materials. Some results of thermodynamic calculation and experiments will be presented.

2:35 PM Invited
Physical Chemistry of Reactive Ball Milling: *Geoffrey Alan Brooks*[1]; [1]McMaster University, Dept. of Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

Reactive ball milling has been proposed as a new route for producing carbides from oxides through carbothermic reduction. It is claimed that through milling oxide and graphite powders to the nanoscale, amorphous materials with very high reactivity are formed, that allows for easy transformation of the starting material to another in a subsequent thermal process. This paper will examine the thermodynamics of these proposed routes, particularly the effect of scale and localised temperatures on the stability of phases. Methods for calculating the effect of particle size on chemical stability will described and problems associated with distinguishing kinectic and thermodynamic effects in these processes will be discussed.

3:05 PM
Investigation in Different Reducing Agents for the Pyro-Metallurgical Treatment of Steel Mill Dusts: *J¸rgen Antrekowitsch*[1]; [1]University of Leoben, Dept. of Nonferrous Metall., Franz-Josef-Strafle 18, Leoben 8700 Austria

Nowadays nearly the half of the worlds zinc production are used for galvanizing. Most of the galvanized products return after their life span as scrap to the steel mill. During the steel process the zinc is collected in the flue dust. It is astonishing, that between 40 and 50% of the zinc produced are brought into the steel processing cycle by coated scrap, while only 3 to 5% of the zinc production can be covered by the recycling of the zinc-bearing steel mill dusts. Responsible for this worse ratio is the fact, that established recycling technologies suffer from high energy consumption, low zinc yields, halogen problems and hardly any iron recovery. A development of new, or an optimization of current recycling procedures for the dusts is unalterable because of increasing zinc amounts in these residues and rising costs for the disposal. Different pyrometallurgical recycling technologies are tested in rotary kilns and vertical retorts at the Department of Nonferrous Metallurgy, University of Leoben, Austria. Various reducing agents from carbon monoxide to hydrogen and combinations are used. The results should make it possible to draw conclusions that allow an economical and ecological judgement of the different varieties. The goal is to support the affected industry to increase the low recycling rate and prevent a loss of high zinc values in the steel industry filter dusts.

3:30 PM
On the Behavior of Arsenopyrite in the Process of Roasting: *V. A. Luganov*[1]; [1]The K.I. Satpaev Kazak National Technical University, 22 Satpaev str., Almaty 480013 Kazakhstan

Behavior of arsenopyrite under heating was studied by many scientists. It was established that under heating at more than 650°C without air access arsenopyrite dissociates with formation of one atom arsenic. Under vacuum the dissociation process begins at 550°C with formation of elemental arsenic too. The residue of dissociation process contains pyrrhotite and throilite. In the presence of pyrite arsenopyrite dissociates under lower temperature and with higher velocity than without it. Industrial realization of fool scale processing of arsenic bearing raw materials is possible only with the use of standard equipment and oxygen containing blast. In this connection the paper presents the results of the study of the influence of oxygen in the gaseous phase and addition into the charge of pyrite on the process of arsenopyrite decomposition. Thermodynamic analyses has shown that under thermal processing of arsenopyrite in the presence of pyrite and limited quantities of oxygen in the gaseous phase format ion of arsenic sulfides is possible according to the following reactions: FeAsS+FeS2=2FeS+0.25As4S4 (1), FeAsS+0.75FeS2+2.8125O2=0.875Fe2O3+0.25As4S4+1.5SO2 (2). Other mechanism of reaction also possible. For example: FeAsS+0. 5FeS2+2O2=0.5Fe3O4+0.25 As4S4+SO2 (3). Thermogravimetric and technological investigations of the process have shown: - in the process of thermal processing there is no decrepitation of arsenopyrite particles; - the specific surface and the porosity of dissociating arsenopyrite particles changes extremely with a maximum depending on dissociation degree; - addition of pyrite into the charge decreases diffusional limitation of the process; - the presence of limited quantities of oxygen in the gaseous phase also decreases diffusional resistance of the process; - the main products of arsenopyrite dissociation in the presence of pyrite and oxygen are arsenic sulfides, pyrrhotite and magnetite; - consecutive dosage (addition by parts) of pyrite makes it possible to increase the degree of arsenic sublimation and to obtain cinder with high sulfur pyrrhotite. The technological investigations have confirmed the results of thermodynamical calculations and kinetic research. Chemical, x-ray, electron microscopic analysis and the BET method were used in the course of the investigations.

3:55 PM Break

4:05 PM Keynote

The Contribution of Pyrometallurgy to Sustainable Development: *W. J. Rankin*[1]; [1]CSIRO Minerals, Bayview Ave., Clayton 3169 Australia

The fundamental issue facing the world is achieving continued economic growth with social equity within the natural limits of the Earthís eco-systems. The basic material and energy needs of the world cannot be met with current ways of addressing these! The demand for metals will continue into the future because their unique properties make substitution by other materials impossible in many applications and this demand will continue be met from primary metal production and recycling. However, the recycling rate will increase and the usage pattern of metals will change to reflect the environmental impact of their production and use. In the overall supply chain of material needs, resource processing is a critical stage for the potential release of gaseous, liquid and solid emissions since it is in the processing stage that ores are most physically and chemically transformed. Pyrometallurgy has an important role in meeting the metal needs of the future since some metals can really only be produced pyrometallurgically; the energy required to produce some metals pyrometallurgically is often significantly less than by other methods; most metals are most efficiently recycled pyrometallurgically; and the release of harmful and toxic elements into the biosphere can often best be controlled through pyrometallurgical processing. The development of strategies for pyrometallurgical processing for the future lies in the recognition that sustainable development calls for large step improvements in value delivered per net unit of environmental impact over the entire supply chain. Pyrometallurgy will need to respond to this challenge by contributing to reducing the environmental impact of meeting metal needs using three broad strategies: ìend-of-pipeî approaches to convert emissions into benign materials for storage or disposal; flowsheet redesign to limit or eliminate emission production; and improved recycling technologies. A number of examples are discussed to illustrate how the sustainability driver is already influencing the technological development of pyrometallurgical processes and how these will develop further as the sustainability driver strengthens.

4:40 PM Invited

Some Challenges on Nitride Metallurgy: Synthesis of Complex Nitrides, Phase Equilibria and Chemical Potential Measurements: *Toru H. Okabe*[1]; Osamu Ishiyama[2]; Hisanori Yamane[3]; K. T. Jacob[4]; Yoshio Waseda[3]; [1]The University of Tokyo, Inst. of Industl. Sci., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8505 Japan; [2]Nippon Steel Corporation, 6-3 Otemachi 2-Chome, Chiyoda-ku, Tokyo 100-8071 Japan; [3]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls. (IMRAM), 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan; [4]Indian Institute of Science, Dept. of Metall., Bangalore 560 012 India

As part of a systematic study on the thermodynamic properties of complex nitrides, phase equilibria of the system Li-M-N (M = Mg, Al, Ga) at 900 K were investigated. An attempt to determine the thermodynamic properties of complex nitrides was made by using the galvanic cell method, which utilizes LiMgN as an electrolyte under nitrogen atmosphere. The electromotive force (Emf) of the cell at temperatures between 800 and 1100 K was measured. While the Emf of the cell at 900 K was measured to be 0.137 V, data discrepancies of ±0.03V were found. It was difficult to determine temperature dependence of the Emf. The Gibbs energy change for the reaction, 1/3 Li3N (s) + 1/3 Mg3N2 (s) = LiMgN (s), at 900 K was determined to be $\Delta G_o{}^r$ (LiMgN) = -13.2 (±2.9) kJ/mol. Using thermodynamic information on the binary nitride from literature, the standard Gibbs energy of LiMgN was derived to be $\Delta G_o{}^f$ (LiMgN)= -121 kJ at 900 K. The thermodynamic stability range o f nitride was discussed by constructing isothermal chemical potential diagrams that provide a better understanding of the stability region of the LiMgN phase. This diagram was found to be in good agreement with the isothermal phase diagram determined by the alloy equilibration method. Although the obtained data includes large uncertainties, the results provide a better understanding of the thermodynamic stability of nitrides. The potential application of this complex nitride to nitrogen chemical sensors or a medium for controlling nitrogen potential is also shown.

5:10 PM

Processing of Antimony Raw Materials in Matte Melts: *O. N. Mustyatsa*[1]; V. A. Lata[1]; [1]Kazakh Academy of Sciences, Inst. of Metall., Almati Kazakhistan

Increase in total raw materials balance of antimony production of the share of oxide materials causes the necessity of the development of the new technological schemes for this kind of raw materials, because metal extraction in accordance with the existing technologies is not possible to consider as completely satisfactory. The authors have offered the technology of antimony raw materials processing for metal in one unit by the method of reducing electromelting with production of matte melts and further electrolysis of them. The method of processing includes: -electromelting together with the sodium sulphate and coal with production of the melt of thiosalts and glass melt; -electrolytical isolation of metals from the melts of their thiosalts. Dependencies of antimony extraction from time, temperature, and components ratio have been studied. Kinetic dependency of the contents of admixtures of iron, arsenic, lead and tin in crude metal under various temperature regimes of reducing melts has been defined. It has been shown that increase of temperature, duration of melting leads to accumulation of iron in antimony. For the production of antimony with low contents of iron the maintenance of optimal conditions (with 800-900C, time of melting 20-30 minutes) is necessary. Maximal antimony extraction has been reached under period of time of electrolysis equal to 1.5 hours and was of the order 99.3%. Investigations made show that electromelting of antimony raw materials to thiosalts with further processing of their melt in the electrolyzer to antimony and sulphur is the base of the new electrochemical technology of wasteless processing of antimony raw materials.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Recycling, Waste Treatment and Environmental Issues I

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Monday PM　　Room: Leucadia
March 3, 2003　　Location: San Diego Marriott Hotel

Session Chairs: Nickolas J. Themelis, Columbia University, Earth & Environl. Eng., New York, NY 10027 USA; M. A. Reuter, Delft University of Technology, Dept. of Appl. Earth Scis., Delft 2628 RX The Netherlands

2:00 PM Keynote

Thermal Plasma Treatment of Metal and Material Wastes: *Patrick R. Taylor*[1]; Wenming Wang[1]; [1]Colorado School of Mines, Dept. of Metallurgl. & Matls. Eng., Golden, CO 80401-1887 USA

In the search for the development of a more ìenvironmentally friendlyî and more intensive waste disposal approach to meet more and more stringent environmental regulations, thermal plasma-based processes are considered one of the most promising alternatives to the well-established technologies such as incineration. This paper presents an overview on the research and development status of thermal plasma-based waste remediation technologies. The properties of thermal plasma are described. Due to its unique feature of high-energy intensity and super high temperature, thermal plasma is particularly capable of destroying organic-containing wastes. Its potential and promising application areas to the waste remediation may include: treatment of incinerator ashes; metallurgical dusts (especially, electric arc furnace-dust); medical wastes; radioactive wastes; and other Resource Conservation and Recovery Act (RCRA) listed hazardous wastes. Many research institutes and vendors have been involved in the development of the thermal plasma-based waste treatment technology encouraged by the cleanup programs supported by the Environmental Protection Agency (EPA), the Department of Energy (DoE) and the Department of Defense (DoD). More recently, thermal plasma-based waste treatment technologies (sometimes called plasma-enhanced pyrolysis) are focused more and more on not only just destroying organic wastes but also on how to practically, and economically recover energy contained in the organic waste materials in addition to the recovery of metals in order to offset the high operating costs.

2:35 PM

Modeling of Arsenic Distribution Between Slags and Copper Mattes or Liquid Copper: *Jonkion M. Font*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

The present study was undertaken in an attempt to develop a model that can predict a-priori the behavior of arsenic in a slag system. The arsenic capacity based on the Reddy-Blander model was derived for the acidic and basic melts of the hypothetically $FeO-SiO_2$, $CaO-SiO_2$, $MgO-SiO_2$, $FeO-CaO-SiO_2$, FeO-CaO-MgO and $FeO-CaO-MgO-SiO_2$ systems. The obtained results are summarized as: the arsenic distribution ratio for the hypothetically $FeO-CaO-MgO-SiO_2$ quaternary system was calculated at 1573, 1523 and 1473 K. Furthermore, the a-priori arsenic distribution ratios were found to be in good agreement with the reported experimental data, when the derived ΔG from the experimental data was considered. These results suggest that, the a-priori arsenic distribution model developed here can be used for prediction in a wide range of the $FeO-CaO-MgO-SiO_2$ slag, matte grade, temperature and pSO_2. Such predictions will be very usef ul for understanding the behavior of arsenic in the current and eventually future non-ferrous processes.

3:00 PM Invited

Pyrometallurgical Reactors-Closers of the Recycling Material Cycle: *M. A. Reuter*[1]; A. van Schaik[1]; [1]Delft University of Technology, Dept. of Appl. Earth Scis., Mijnbouwstraat 120, Delft 2628 RX The Netherlands

Modern consumer society is governed by the extensive use of complex multi-component products, of which passenger vehicles are an outstanding example. At the end-of-life these products return, as complex multi-component materials that cannot directly be converted into products once more. However society requires that a maximum of end-of-life products find their way back into the industrial and consumer cycle to ensure sustainable development. In order to capture the influence of rapidly changing design of products on recycling, a dynamic optimisation model has been developed that links product characteristics to the optimal recovery of metal in pyrometallurgical reactors. This model consists of a dynamic part, which imbeds an optimisation model. Whereas the dynamic model predicts the behaviour of the resource cycle over time based on various characteristic distribution functions for the changing lifetime, weight and composition of the car, the optimisation model optimises the recycling of the car as a function of product design, efficiency of the different process steps in recycling (i.e. physical separation and pyrometallurgical metal production), economics and legislation. Therefore, it is argued in this paper that the fundamental thermodynamics and kinetics in the metallurgical reactors play a final crucial role in closing the material cycle. Therefore, based on various fundamental simulations, the interaction between the time dependent variables in product design and the optimisation of the recycling of end-of-life vehicles will be discussed in relationship to the fundamental thermodynamics in metallurgical reactors. This implies that central to the discussion is the feedforward control of the feed to the reactors, their composition and the effect thereof on the performance of the reactor and the quality of the produced metal product. The discussed model has been developed in Matlab.

3:30 PM

Thermodynamics of Iron Reduction from Metallurgical Residues: *Ivan Imris*[1]; Alexandra Klenovcanova[1]; Matej Imris[2]; [1]Technical University of Kocice, Dept. of Power Eng., Fac. of Mechl. Eng., Letna 9, Kosice 041 87 Slovak Republic; [2]Technical University of Kosice, Dept. of Non-Ferrous Metals & Waste Treatment, Letna 9, Kosice 041 87 Slovak Republic

The iron and nickel production in Slovakia generated many tonnes of very fine residues with iron contents vary from 44 to 74 wt.%. In addition the steelmaking dust contains zinc and lead which are prevented inplant recycling. These kind of residues are usually dumping beside the metallurgical plants to make the environmental problems. Therefore the metal recovery from metallurgical residues has become major interest not only from the viewpoint of metals recycling but mainly from the viewpoint of environmental land protection. The thermodynamic analyses and laboratory experiments suggested that the metals could be recovered from metallurgical wastes by reduction roasting and by plasma smelting processes. The iron rich pellets with very high metallization may be obtained from metallurgical wastes by reduction roasting process and by plasma smelting process the inert slag and pig iron could be produced. In both cases the iron rich products are suitable for the steel production. During reduction roasting and plasma smelting processes the high volatile zinc and lead metals were removed and enriched as a secondary dust, Zn-Pb concentrate, which is suitable for processing in zinc and lead smelters. Suggested processes are commercially feasible and environmentally friendly without any solid or liquid waste.

3:55 PM Break

4:10 PM Invited

Simulation of Transport and Chemical Phenomena in Flash Combustion of Municipal Solid Wastes: *Nickolas J. Themelis*[1]; Young Hwan Kim[1]; [1]Columbia University, Earth & Environl. Eng., 500 W. 120th St., #918, New York, NY 10027 USA

Nearly thirty six million tons of municipal solid wastes (MSW) are combusted annually in over one hundred US Waste-to-Energy (WTE) power plants, thus obviating the use of 1.6 billion gallons of fuel oil. One of the advanced WTE processes is the 1-million ton/y SEMASS facility designed by Energy Answers Corp. and operated by American Ref-Fuel in southeastern Massachusetts. Most of the combustion in the three giant combustion chambers occurs while the injected shredded wastes are in flight, i.e., in flash combustion mode. The velocity, temperature, and concentration profiles in the SEMASS combustion chamber were simulated by representing the combustible fraction of MSW by the simplified formula C6H10O4 and using the FLUENT Computerized Fluid Dynamics program to solve the turbulent energy and mass transport equations. The results of this model were used to examine options for increasing the productivity of WTE combustion chambers, such as the effect of oxygen enrichment.

4:40 PM

Thermodynamical Analysis of the Chloridization Reaction in Ash Melting Furnace: *Kokoro Iwasawa*[1]; Nan Wang[2]; Shu Yamaguchi[2]; Masafumi Maeda[1]; [1]The University of Tokyo, Inst. of Industl. Sci., 4th Dept., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8505 Japan; [2]Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya 466-8555 Japan

Various kinds of remelting or pelletizing processes of primary bottom ash and fly ash evolved from the incineration of municipal solid wastes, have been proposed for the volume reduction and detoxification of residues. In the latter issues, it is important to know the behavior of heavy metal elements in the pyrometallurgical processes. The purpose of the present study is to investigate the chloridization reaction of heavy metal in the ash treatment processes from thermodynamic view point. The present authors focus attention on the effect of Na_2SO_4 formation in the ash re-melting furnace. From the evaluation of the Na_2O activity in the Na_2O-SiO_2, Na_2O-CO_2, Na_2O-SO_3, and $Na_2O-P_2O_5$ systems from reported thermodynamic data, it is found that the Na_2O activity is prominently low in the Na_2O-SO_3 system, suggesting that NaCl contained in as hes can work as a chloridizer as the following reaction is favored. $2NaCl + SO_3 + 1/2\ O_2 = Na_2SO_4 + Cl_2$. Further discussion on the reaction between oxides (composed of mainly the Na_2O-SiO_2 system) and salt systems, which are regarded as the $NaCl-Na_2CO_3-Na_2SO_4$ quasi-ternary system, has been made using the potential stability diagrams at various temperatures. Because of strong temperature dependency of $\Delta G°$ for the Na_2SO_4 formation reaction, Na_2SO_4 formed at lower temperatures can be decomposed to Na_2O and SO_3 at higher temperatures by the reaction with SiO_2. The authors proposed a novel processing of solid wastes based on the present results which utilize the formation and decomposition reactions of Na_2SO_4.

5:05 PM

Combustion Modeling of Hazardous Waste Incineration: *Y. Yang*[1]; M. Pijnenborg[1]; M. A. Reuter[1]; [1]Delft University of Technology, Dept. of Appl. Earth Scis., Mijnbouwstraat 120, Delft 2628 RX The Netherlands

Hazardous waste has often very complicated chemical compositions in variety of physical forms. It is normally processed in rotary kiln incinerators. Due to large variations in waste types and difficulties in feed characterization, the complex transport processes within the incinerator are not well understood, and the incineration process meets great challenges in a smooth operation, and expects various uncertainties in the process chemistry and emission control. For better understanding of the incineration process, process simulation was conducted by using Computational Fluid-dynamics (CFD) to characterize temperature and species distribution in the incinerator. As the first step, hazardous waste in various forms is converted to a virtual fuel or fuel mixture with more or less equivalent chemical compositions and heating value. Then the simulation of the combustion process of this virtual fuel in an industrial-rotary-kiln waste incinerator was carried out with a combustion model. The distribution of temperature and chemical species especially the remaining CO in the system is investigated. The results give a good indication on the combustion efficiency and emission level (indicated by CO concentration), and influences from various operating and process parameters.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: Nickel

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Monday PM — Room: Santa Rosa
March 3, 2003 — Location: San Diego Marriott Hotel

Session Chairs: T. A. Utigard, University of Toronto, Matls. Sci. & Eng., Toronto, Ontario M5S 3E4 Canada; Anthony (Tony) E.M. Warner, Inco Technical Services Ltd., Mississauga, Ontario L5K 1Z9 Canada

2:00 PM Keynote

An Overview of the Metallurgy of Nickel-Copper Matte Converting: *Anthony (Tony) E.M. Warner*[1]; Carlos M. Diaz[2]; [1]Inco Technical Services, Ltd., 2060 Flavelle Blvd., Sheridan Park, Mississauga, Ontario L5K 1Z9 Canada; [2]Metallurgical Consultant, 210 Radley Rd., Mississauga, Ontario L5G 2R7 Canada

Inco, Limited has long and varied experience in converting nickel-copper primary smelting mattes over a broad range of both nickel/copper weight ratios and combined nickel-copper-cobalt contents. In addition, at the Copper Cliff Smelter, the composition of the converter product matte has to meet the requirements of the matte separation process while, at the Thompson Smelter, converter matte with a different composition is cast into anodes for nickel electrowinning. Recently, Inco has conducted pilot plant tests of three possible nickel-copper matte continuous converting routes, namely flash converting, oxygen top blowing-nitrogen bottom stirring, and tuyere blowing. This work has generated substantial new information on nickel-copper matte converting chemistry. In this overview paper, the metallurgy of nickel-copper matte converting is reviewed with emphasis on the behaviour of cobalt and on the operating conditions required to meet the iron and sulfur specifications of the product matte. The available relevant thermodynamic information is used to discuss the industrial data.

2:30 PM

Miniplant Oxygen Flash Smelting of Bulk Copper-Nickel Sulfide Concentrate: The Effect of Coke Addition on Process Metallurgy: *Jin Liu*[1]; Anthony E.M. Warner[1]; Torstein Utigard[2]; Carlos M. Diaz[2]; Mustafa Fezzani[1]; [1]INCO Technical Services, Ltd., 2060 Flavelle Blvd., Sheridan Park, Mississauga, Ontario L5K 1Z9 Canada; [2]University of Toronto, Dept. of Matls. Sci. & Eng., 184 College St., Toronto, Ontario M5S 3E4 Canada

At its Copper Cliff Smelter, Inco, Limited processes a bulk copper-nickel concentrate in two Inco oxygen flash furnaces. The furnace matte is converted to low iron matte for further processing, and the molten converter slag is recycled to the flash furnaces. The flash furnace slag is discarded. Natural gas or, in recent years natural gas combined with coke, supplies a minor proportion of the process heat requirements, thus limiting the flash furnace matte grade to the target 45% CuNiCo. Coke addition to the furnace offers a potential means of establishing a reducing barrier on the surface of the molten bath. Such a practice would be conducive to producing sulfur deficient (partially metallized) matte. At the same iron content, the iron activity of sulfur deficient matte is higher than that of regular matte. A supernatant coke barrier could, therefore, be expected to lead to higher nickel and cobalt recoveries, reduction or even elimination of magnetite furnace bottom buildup and increased sulfur elimination as SO2 in the flash furnace. Preliminary experiments were conducted in a miniplant flash furnace to investigate the feasibility and merits of this process scheme. This paper presents the experimental results and discusses key aspects of the possible operation of the Inco flash furnace with a coke barrier separating the freeboard from the molten bath.

2:55 PM

The Operation of the INCO Flash Furnace Uptake: Combustion of H2S and Formation of Uptake Buildup: *Jin Liu*[1]; Anthony E.M. Warner[1]; Geoff Osborne[2]; *Darryl Cooke*[2]; Ralph Slayer[1]; [1]INCO Technical Services, Ltd., 2060 Flavelle Blvd., Sheridan Park, Mississauga, Ontario L5K 1Z9 Canada; [2]INCO, Ltd., Copper Cliff Smelter Techl. Services, Copper Cliff, Ontario P0M 1N0 Canada

The 15m high INCO flash furnace uptake was designed as a reaction chamber with the objective of destroying the H2S contained in the furnace off-gas by combustion with oxygen at about 1300∫C. Until recently, the oxygen required for this purpose was injected through afterburners located at the bottom of the uptake. An undesirable side effect of this practice was the formation of build up accretions on the inside walls of the uptake. This buildup had a negative impact on both the furnace on line time and the control of the flash smelting process. In late 2001, the afterburners were relocated to the roof of the uptake. As a result, about 80% of the uptake accretion buildup disappeared with obvious beneficial effects for the operation of the furnace. This paper discusses key flash smelting aspects that affect the operation of the flash furnace uptake such as the dusting rate, the formation of H2S in the freeboard of the furnace, and the impact of using coke to supply some of the heat required by the process. The mechanism of buildup formation and its dependence on uptake afterburners location are also examined. Prof. Yazawaís sulfide smelting thermodynamic relationships proved instrumental in understanding the conditions that influence the formation and the elimination of H2S and uptake buildup.

3:20 PM

Nickel and Copper Behaviour in the Process of Autogenous Smelting of the Concentrate after High-Grade Matte Separation: *L. Sh. Tsemekhman*[1]; A. G. Ryabko[1]; L. B. Tsymbulov[1]; G. P. Miroevskiy[1]; A. N. Golov[1]; [1]AO ìGipronickelî, 1 Grazhdansky Pr., Saint-Petersburg 195220 Russia

At Severonickel Combine JS the process of autogenous smelting of copper concentrate after high-grade matte separation in a stationary unit with upper oxygen blow is being brought to a commercial level. Due to heat deficit fuel oil is put on together with the blow. Processes for autogenous smelting of the concentrate with crude blister and blister copper and liquid silicon slag have been developed and tested. Ni:Cu ratio in the slag is higher that in solid slag, formed in crude blister copper converting. Conduct of Ni, Cu and Co forms of metals in slag as well as gas mode of the -process have been studied.

3:45 PM Break

3:55 PM Invited

Laboratory One-Step Bath Smelting of Nickel Concentrate to a Low Iron Matte: *N. J. Tannyan*[1]; T. A. Utigard[1]; [1]University of Toronto, Matls. Sci. & Eng., Toronto, Ontario M5S 3E4 Canada

Laboratory bath smelting tests were conducted to determine the feasibility of smelting nickel-copper concentrate directly to a low iron matte in one step. Concentrate, air/oxygen and a silica flux were fed to a matte-slag bath kept in MgO crucibles at 1300°C. Various oxygen enrichments and smelting rates were used to maintain 0.5 to 2 wt% iron in the product matte. The slag losses are comparable to those obtain under similar industrial conditions. Due to the highly oxidizing conditions required to produce mattes with low iron contents, it is not recommended to target iron contents below about 2% in the matte. The specific bath smelting rates were equal to or higher than those in various industrial bath smelting processes.

4:20 PM

Improving the Operating, Maintenance and Repair Practices of an Inco Flash Furnace at the Copper Cliff Smelter: *Randy Lawson*[1]; W. Peter Lee[2]; [1]Copper Cliff Smelter Complex, Copper Cliff, Ontario P0M 1NO Canada; [2]Inco Technical Services, Ltd., 2060 Flavelle Blvd., Mississauga, Ontario L5K 1Z9 Canada

Incoís Copper Cliff Smelter in Sudbury, Ontario operates two (2) Inco flash smelting furnaces treating a copper-nickel (bulk) concentrate produced by the areaís milling operations. Daily concentrate throughput averages ~4800 tonnes at a grade ~21% CuNiCo by mass. The two new flash furnaces began the bulk smelting operation around 1993 as part of the Incoís SO2 abatement project. Over the years, a number of specialized repair procedures have been developed with the intention of increasing on-line time, productivity and longevity (vessel integrity) between major turn-arounds. Some of the more significant improvements and repair procedures are highlighted in this paper with the aim of providing other furnace operators some ideas for development of their respective technologies.

4:45 PM

Direct Sulfation of Nickel Laterite Ores Using SO2-Rich Gases: D. Papazoglou[1]; *W. J. Rankin*[2]; [1]WMC, Ltd., Kwinana Nickel Refinery,

Kwiana, WA 6167 Australia; [2]CSIRO Minerals, Clayton, VIC 3169 Australia

This work was undertaken to investigate the thermodynamic and kinetic feasibility of using hot sulfur dioxide bearing gases from smelting processes to selectively sulfate the nickel in nickel laterite ores for subsequent extraction in water. The environmental benefits of this approach, as opposed to releasing the gas to the atmosphere, and the economic benefit of directly utilising the SO2 rather than converting it to sulfuric acid are strong drivers for such a process. A thermodynamic analysis indicated that selective sulfation of the nickel could be feasible under certain conditions and these were explored through a series of laboratory scale experiments. Laterite samples were reacted at temperatures between 500 and 800∞C with an equilibrated mixture of sulphur dioxide, oxygen and nitrogen in a fixed bed reactor. Tests were conducted on saprolitic ore from Bulong, Western Australia, limonitic ore from New Caledonia and garnieritic ore from Indonesia. The main variables examined were reaction temperature, st rength of the sulphating atmosphere, reaction time and particle size. Nickel extractions were highest from the limonite and relatively low from the other ores. The maximum selective extraction of nickel from the limonite in a single step sulfation was 73%. A two-step process was also identified which involved removing the reaction products by leaching and then sulphating again at the same conditions. This increased the extraction for the limonite ore to around 84% Ni, 95% Co and 2% Fe. Extraction from the garnierite ore was strongly dependent on particle size. The 106-212 mm fraction yielded only 15% Ni extraction, in a single step sulfation, whereas the <30 mm fraction yielded 66%. In contrast, the limonite ore showed an increase from 72% to 74% Ni extraction over the same range. Maximum nickel extractions from the saprolite ore were generally below 60%, but increased to around 70% with the two-step process. Direct sulfation of laterite could be feasible in locations where sulfide smelting or roasting operat ions are relatively close to laterite deposits. Such a location is in the Kalgoorlie region of Western Australia.

5:10 PM

An Investigation on the High-Mg-Content Slag in Jinchuan Nickel Flash Smelting: Wan Zhi[1]; Wan Wei[1]; [1]Jinchuan Non-Ferrous Metals Company, Ltd., Jinchang, Ganshu 33000 China

High-Mg-content concentrates are usually treated in Jinchuan flash smelting furnace. Because the viscosity of high-Mg-content slag is very high, the operation of flash smelting becomes very difficult. After adjusting the ratio of FeO/SiO2 and operation conditions, the flash smelter works smoothly. The nickel content in the slag can decrease to 0.18-0.22%.

Notes

Actinide Materials: Processing, Characterization, and Behavior: Plutonium

Sponsored by: Light Metals Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS), LMD-Reactive Metals Committee
Program Organizers: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA; Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Tuesday AM	Room: 4
March 4, 2003	Location: San Diego Convention Center

Session Chair: Sean M. McDeavitt, Argonne National Laboratory, Cheml. Tech. Div. Matls. Dvlp. Sect., Argonne, IL 60439-4837 USA

8:30 AM Keynote
An Overview of Plutonium: Metal, Ceramics, and Chemistry: *Siegfried S. Hecker*[1]; [1]Los Alamos National Laboratory, MST-DO, MS G754, Los Alamos, NM 87545 USA

Plutonium is the most complex element. It sits near the middle of the actinide series and appears to occupy a unique position with no analogues. In the solid state, the 5f electrons in plutonium are caught in an abrupt transition between being bonding and being localized, giving rise to very unusual propertiesñespecially its instability with temperature, pressure, and chemistry. It is highly reactive in moist air and strongly reducing in solution, forming multiple compounds and complexes in the environment and during chemical processing. In molecular bonding, the proximity of the 7s, 6d, and 5f electrons in valence orbitals generates a strong competition among these configurations while the 5f electrons extend sufficiently far to favor highly directional bonding. Plutonium transmutes by radioactive decay, damaging its crystalline lattice and leaving behind helium and other transmutation products in solids and causing radiolytic effects in solution or in organic materials.

9:15 AM
Thermal Expansion and Gr¸neisen Ratio of Invar-Like δ-Phase Pu-Ga Alloys: *Andrew C. Lawson*[1]; Joyce A. Roberts[2]; Barbara Martinez[3]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS H-805, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Los Alamos Neutron Sci. Ctr., MS H845, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Nucl. Matls. Tech. Div., MS E574, Los Alamos, NM 87545 USA

Recently we showed that the thermal expansion behavior of Pu-Ga follows the Weiss two-state model for the invar effect developed for Fe-Ni alloys. In δ-phase Pu, the thermal expansion behavior is determined by a competition between the normal lattice expansion with the expansion associated with the thermal occupation of two localized states, a lower-energy higher volume state δ_1, and a higher energy lower volume state δ_2. Higher temperature favors the occupation of the lower volume state. Using model parameters appropriate for Pu-Ga, we discuss the consequences of the invar model for these alloys: atomic volume, thermal expansion and Gr¸neisen constant versus temperature and Ga concentration. The competition between two thermal expansion mechanisms leads to anomalous behaviors for these quantities.

9:35 AM
Thermodynamic Assessment of the Stability Properties of Pu-Based Alloys: Application of the Calphad Methodology: *Patrice E.A. Turchi*[1]; Patrick G. Allen[1]; Shihuai Zhou[2]; Zi-Kui Liu[2]; Larry Kaufman[3]; [1]Lawrence Livermore National Laboratory, Dept. of Chem. & Matls. Sci., PO Box 808, Livermore, CA 94551 USA; [2]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16803 USA; [3]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Cambridge, MA 02139 USA

The CALPHAD approach has been applied to the study of the equilibrium thermodynamic properties of Pu-X alloys (X=Al,Fe,Ga,Ni). Predictions are made on the low temperature phase diagrams of Pu-Ga and Pu-Al for which controversy has been noted in the past. The validity of the assessed thermodynamics is discussed by comparing predicted heats of transformation with measured values from DSC analysis. An overall picture for the stability properties of Pu-Ga and Pu-Al that reconciles the results of past studies carried out on these alloys is proposed. Additional assessment will be presented for the Pu-Ni system. Finally preliminary results are presented on TTT diagrams for some of the reactions occurring in Pu-Ga alloys, and suggestions are made for further studies. This work was performed under the auspices of the US Department of Energy by the University of California Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

9:55 AM
Mechanical Behavior of Delta-Phase Plutonium-Gallium Alloys: *George C. Kaschner*[1]; Michael G. Stout[1]; Siegfried S. Hecker[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-DO, MS G754, Los Alamos, NM 87545 USA

In this report, we present a constitutive model that predicts the yield strength and ultimate tensile strength (UTS) of delta-stabilized plutonium-gallium alloys (Pu-Ga). The model accounts for the effects of temperature, strain rate, grain size, and gallium (Ga) concentration. The coefficients in the model are based on ambient-pressure quasi-static data that were published in the open literature. Our model has been validated against approximately 50 different experiments for both yield and ultimate strength. The predicted yield strengths agreed with the experimental data to within a ±1 standard deviation of 15%. The gallium concentration has the greatest affect on the yield strength. There was a 50% increase in yield strength ranging from Pu-1 at % Ga to Pu-6 at % Ga. The grain size also produced a measurable strengthening effect, typical of face-centered-cubic (fcc) metals. The yield strength for a material with 10 µm grain size was 15% greater than for 50 µm grain size. Finally, we found that there were no observable yield strength effects from different amounts of either iron and nickel impurities or carbon concentrations.

10:15 AM Break

10:35 AM
Electron Backscatter Diffraction of Plutonium-Gallium Alloys: *Carl J. Boehlert*[1]; Thomas G. Zocco[2]; Roland K. Schulze[2]; Jeremy N. Mitchell[2]; Ramiro A. Pereyra[2]; [1]Alfred University, Sch. of Ceram. Eng. & Matls. Sci., CEMS/McMahon Hall, 2 Pine St., Alfred, NY 14802 USA; [2]Los Alamos National Laboratory, Nucl. Matls. Tech., MS G721, Los Alamos, NM 87545 USA

An experimental technique has been developed to characterize reactive metals, including plutonium and cerium, using electron backscatter diffraction (EBSD). Microstructural characterization of plutonium and its alloys by EBSD had been previously elusive primarily because of the extreme toxicity and rapid surface oxidation rate associated with plutonium metal. The experimental techniques, which included ion-sputtering the metal surface using a scanning auger microprobe (SAM) followed by vacuum transfer of the sample from the SAM to the scanning electron microscope (SEM), used to obtain electron backscatter diffraction Kikuchi patterns (EBSPs) and orientation maps for plutonium-gallium alloys are described and preliminary microstructural observations based on the analysis are discussed. Combining the SEM and EBSD observations, the phase transformation behavior between the d and e structures was explained. This demonstrated sample preparation and characterization technique is expected to be a powerful means to further understand phase transformation behavior, orientation relationships, and texture in the complicated plutonium alloy systems.

10:55 AM
Influence of Fe on Elevated Temperature Phase Transformations in Pu-0.6 Wt.% Ga: *Daniel S. Schwartz*[1]; Thomas G. Zocco[1]; Ramiro A. Pereyra[1]; Michael Ramos[1]; [1]Los Alamos National Laboratory, NMT-16, PO Box 1663, MS G721, Los Alamos, NM 87545 USA

Low concentrations of Fe are thought to have a significant effect upon phase transformations and homogenization kinetics in lean Pu-Ga alloys above 400C. In order to study this effect in detail, differential scanning calorimetry (DSC) and optical microscopy were used to examine phase transformations in Pu-0.6 wt.% Ga containing ~300 wppm Fe. A series of specimens cut from a casting of Pu-0.6 wt.% Ga + 300 wppm Fe were given 24 hour anneals at temperatures ranging from 450C to 510C. The specimens were subsequently scanned in the DSC from RT to 580C. The α-β and δ-ε solid state transformations and the Pu_6Fe melt transition were examined in detail. Material annealed above ~480C showed significant qualitative and quantitative differences compared to material annealed at the lower temperatures. The observed differences and their ramifications will be discussed in detail.

11:15 AM
Atomistic Determination of the Transport Properties of Liquid Plutonium-Gallium Alloys: *Frank J. Cherne*[1]; Michael I. Baskes[1]; [1]Los Alamos National Laboratory, MST-8, Struct. & Prop. Rel., PO Box 1663, MS G755, Los Alamos, NM 87545 USA

The transport properties of liquid plutonium-gallium alloys are calculated using equilibrium and non-equilibrium techniques. The potentials chosen for our study are based on the modified embedded atom method. These potentials have been shown to be representative of Pu/Ga system. The calculated transport coefficients, diffusivity and viscosity, are evaluated across the entire composition range (0-100% gallium). The calculated viscosity is found to be much greater than the available experimental data. The range of temperatures evaluated are between 950 K and 1700 K.

11:35 AM
Atomistic Modeling of the Phase Stability in the Pu-Ga System: *Krishna Muralidharan*[1]; M. I. Baskes[1]; Marius Stan[1]; S. G. Srinivasan[1]; [1]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

The plutonium (Pu)-gallium (Ga) system has a complex equilibrium phase diagram consisting of about twenty phases. There is still considerable disagreement in the literature regarding the existence of a eutectoid point in the Pu-Ga phase diagram. In this work, we calculate the Pu rich (XGa < 0.25) part of the phase diagram using a combination of molecular dynamics (MD) and thermochemical calculations, with the recently developed MEAM potential used to represent the interatomic interactions. This potential was shown to be quite reliable in predicting the stable phases of the pure Pu and the Ga systems. The phase diagram is constructed by calculating the free energies of the various phases as a function of temperature and composition. The resultant phase diagram will be compared and contrasted to existing experimental data.

Alumina and Bauxite: Red Mud Operations

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: David Kirkpatrick, Kaiser Aluminum & Chemical Group, Gramercy, LA 70052-3370 USA; Steve Rosenberg, Worsley Alumina Pty, Ltd., Process Chemistry Group, Collie, West Australia 6225 Australia; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday AM — Room: 3
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: David Kirkpatrick, Kaiser Aluminum & Chemical Group, Gramercy, LA 70052-3370 USA; Steve Rosenberg, Worsley Alumina Pty Ltd., Process Chem. Grp., Collie, W. Australia 6225 Australia

8:30 AM
Influence of Bayer Liquor Composition on the Measurement of Slurry Velocity Using an Electrochemical Flowmeter: *AndrÈ Leclerc*[1]; Alexandre Perron[1]; Guy Simard[1]; Michel J. Gagnon[1]; Guy Peloquin[2]; [1]UniversitÈ du QuÈbec ‡ Chicoutimi, Dept. Scis., 555 Blvd. de lÍUniversitÈ, Chicoutimi, QuÈbec G7H 2B1 Canada; [2]Alcan International Ltd., Jonquiere, Quebec G7S 4K8 Canada

A knowledge of the flow velocities in the feed well of sequential gravity settlers of the Bayer process is essential for their optimal design and operation. The velocities range from 0 to 50 cm/s and the characteristics of the Bayer liquor vary between settlers. An intrusive electrochemical flowmeter described previously may be used to measure slurry velocities in the 5 to 50 cm/s range with a precision of 10% by monitoring an electrical current. This current depends not only on the fluid velocity but also on the characteristics of the Bayer liquor. The principal physical parameter is the temperature since the electrochemical reaction rates and physical properties are temperature dependant. The chemical parameters are the concentrations of the major solutes that partake in the electrochemical reaction or that affect the activies of the reagents. To use this flowmeter, calibration curves relating the current as a function of the temperature and the concentrations of sodium hydroxide, carbonate and aluminate must be available. Typical concentration curves are presented and discussed.

8:55 AM
A Fractal Model for the Aggregate Size Distributions Generated During Red Mud Flocculation: *Michel J. Gagnon*[1]; Andre Leclerc[1]; Guy Simard[1]; Guy Peloquin[2]; [1]Universite du Quebec at Chicoutimi, Dept. Scis., 555 Blvd. Universite, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International Ltd., 1955 Blvd. Melon, Jonquiere, Quebec G7S 4K8 Canada

In the Bayer process, flocculation is produced in the gravity settlers to enhance the settling of the fine particles of red mud. Depending upon many physical and chemical parameters, the aggregates generated demonstrate specific density-size distributions. In this study, a fractal model of the flocculation process is introduced. Based on reliable experimental data and on physical calculation, the model presented helps to envisage the initial formation of the aggregates, and to interpret the subsequent interactions between the aggregates in terms of a global flocculation process. A good agreement is obtained between the model and the experimental data.

9:20 AM
Effect of Organics on the Separating Efficiency of Flocculates in Red Mud Sedimentation: *Zhijian Lu*[1]; [1]Henan Filiale of China Aluminium Company, Tech. Dept., Shangjie Dist., Zhengzhou, Henan 450041 China

Adding flocculates is an important method to facilitate the separating of red mud slurry in alumina refinery, while the existence of organics will disturb their separating efficiency. The effect of seven organics (benaenedicarboxylic sodium, phenol, oxalic sodium, formic sodium,acetic sodium, yellow and black humic acid) on overfall clarity, underflow L/S and sedimentation rate in red mud separating procedure by using three anionic flocculates such as A-600, HPAM, PAS-1 and one nonionic flocculate (PAM) had been researched respectively in this paper. It is illustrated that the existence of organics can significantly decrease the separating efficiency of flocculates. The optimum concentration of flocculates in slurry increase with the rising of organics and the negative effect of organic canít be eliminated when its content higher than a certain value which is different for every organic. However, A-600 has best separating effect for all experiments while the effect of nonionic flocculate PAM is indistinctive.

9:45 AM
Security Filtration Operation in CVG-Bauxilum: Ricardo Alfredo Galarraga[1]; [1]CVG-Bauxilum, Lado Rojo II, Zona Industrial Matanzas, Puerto Ordaz, BolÌvar 8015 Venezuela

The segurity filtraciÛn area (38 area), is essential in the Bayer process of alumina production. In CVG-Bauxilum this area to consist in 16 Kelly filters of 36 m3 of capacity and 372 m2 of filtration area by filter. Around of 19 years of plant operation has been maked changes structural and operational for improve the performance. The changes of mesh of the filtres frames, the increase the flow across of the frames, the time the operation of the filters, the frequency of washed acid and caustic, the cloths life and the relation of TCA are some changes done in the industrial area. This work involve the more important changes done until today for improve the area for to serve the levels of production demanding.

10:10 AM Break

10:20 AM
Settling Properties and Utilization of Red Mud Produced in Bayer Process with an Excessive Addition Lime: *Zhao Hengqin*[1]; Feng Ansheng[1]; Li Jie[2]; Liu Yexiang[2]; [1]Zhengzhou Institute of Mulitipurpose Utilization of Mineral Resources, No 328 Longhai W. Rd., Zhengzhou, Henan 450006 China; [2]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

Good settling property of red mud is one of what contribute to smooth production of alumina. In this paper, the study has been performed on the settling properties of red mud produced in Bayer Process with an excessive addition lime. The results indicated that the settling property of red mud produced in the Bayer Process with an excessive addition lime are slightly poorer than that in conventional Bayer Process, but the former red mud with smaller ratio of L/S, and can meet the requirements of production process of alumina. As for red mud produced in the above improved Bayer Process,its utilization has also been studied, and the research results showed that the red mud can be used as raw material of silicate cement.

10:45 AM
Conversion of Conventional Wet Disposal of Red Mud into Thickened Tailing Disposal (TTD) at Nalco Alumina Refinery, Damanjodi: *S. K. Banerjee*[1]; [1]National Aluminium Company, Ltd., Dir. (Proj. & Techls.), Bhubaneswar 751013 India

National Aluminum Company (NALCO) is a leading company in India to successfully implement the environmental friendly Thickened Tailing Disposal (TTD) System for disposal of red mud produced from itís Alumina Refinery. Earlier, the red mud was disposed of in a pond using conventional wet disposal method with a solid concentration of 25-30%. The TTD system, introduced recently under the expansion program, is operated with high rate thickeners and positive displacement pumps to dispose of the red mud at a solid concentraion of 55-65% on the existing pond. The introduction of this new system has resulted in substantial savings in capital cost in construction of a new pond apart from saving in energy cost for recycling of water from the red mud pond. The detailed engineering for the system was done by Engineers India Tld. and the system was supplied by M/S Envirotech, Netherlands at an approximate cost of USD 15 million.

11:10 AM
Experience with Thickened Disposal of Red Mud: *Pitabas Das*[1]; D. Roy[1]; [1]National Aluminum Company Ltd., R&D, Orissa State 00085 India

Due to the obvious environmental impact emanating from disposal of Red Mud, it has always posed to be a sticky problem for Alumina Plants. Ever tightening environmental regulations have been forcing Alumina producers worldwide to continuously look for better and safer methods of disposing the red mud generated by them. As most pollution controllers consider traditional method of disposal in wet form, a potential threat to environment, the trend now is to switch over to a dry disposal mode, or otherwise termed as the Thickened Tailings Disposal (TTD) system. National Aluminium Company (NALCO) in India has recently commissioned a TTD System. This paper elaborates the advantages and disadvantages experienced with the system so far.

11:35 AM
Problems of Red Mud DisposalñIs Romelt an Answer?: *S. K. Misra*[1]; J. S. Saluja[2]; [1]National Aluminum Company, Ltd., Rsrch., Dvpt. & Eng., P/1, Nayapali, NALCO Bhawan, Bhubaneswar 751013 India; [2]Romelt Sail India, Ltd., A-47, Friends Colony (East), New Delhi-65 110065 India

Bauxite Residue (Red Mud) is the major waste from Alumina Refineries which are contaminated with alkali and pose serious environmental hazards. NALCOís alumina refinery having an annual capacity of 1.575 Million tones per annum disposes red mud in specially designed ponds around its refinery. The Bauxite having 25-30% iron content in the form of oxides offers tremendous scope for waste utilization and mitigation of environmental hazards. Several methods for extraction of iron from low grade ores and wastes are now available. Moscow Institute of Steel and Alloys (MISA) after intensive research for iron making have developed the Romelt Technology which accepts the microfine iron oxides without any pretreatment. NALCO after getting promising results from the laboratory testing of its red mud are now taking steps for preparation of a feasibility report for a Romelt Plant which will substantially eliminate the problem of waste disposal apart from maintaining the ecological balance.

Aluminum Reduction Technology: Environmental - HF and PFC

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Martin Segatz, VAW Aluminum Technology, Bonn D-53117 Germany; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday AM　　Room: 6B
March 4, 2003　　Location: San Diego Convention Center

Session Chair: Jerry Marks, J. Marks and Associates, Kansas City, MO 64064 USA

8:30 AM
The Effect of Pot Operation and Work Practices on Gaseous and Particulate Fluoride Evolution: *G. P. Tarcy*[1]; [1]Alcoa Inc., Hall Process Improvement, Alcoa Techl. Ctr., 100 Alcoa Dr., Alcoa Ctr., PA 15069-0001 USA

Alcoa has measured fluoride evolution (amount of fluoride in the duct off a single pot) intermittently since 1980. In 1982 and 1983, continuous real time measurements were made of gaseous HF and total fluoride on a series of single pots ranging from 20 kA to 275 kA. The information was cataloged as a function of pot operations including alumina feeding, anode effect, crust integrity, anode change, bath chemistry changes and metal tapping. The studies showed that the single most important factor to both gaseous HF evolution and particulate evolution was the integrity of the crust. As the crust integrity deteriorated into open holes, the amount of HF evolution increased up to a limiting rate while the particulate evolution increased without any indication of a limit being reached. The studies also showed that under ideal operations (good crust integrity and no upsets), approximately 16% of the fluoride evolved from the pot was due to feeding of alumina and the evolution associated with this was due to water on the material, not fluoride or crust breaking. Anode effect and the method by which they are terminated also impact the fluoride evolution, as does anode change and the method by which the anode is covered. Metal tapping has a very short-lived high evolution rate associated with this practice.

8:55 AM
The Impact of Open Holes on Vapor-Phase Fluoride Evolution from Pots: *Michael L. Slaugenhaupt*[1]; Jay N. Bruggeman[1]; Gary P. Tarcy[1]; Neal R. Dando[2]; [1]Alcoa Inc., Hall Process Improvement, Alcoa Techl. Ctr., 100 Alcoa Dr., Alcoa Ctr., PA 15069-0001 USA; [2]Alcoa Inc., Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Ctr., PA 15069-0001 USA

Pot operating and tending practices are the primary determinants of fluoride evolution. Fluctuations in fluoride evolution have direct consequences on pot gas scrubber loads, which can impact scrubber maintenance and capture efficiency. Variability in fluoride evolution can also have dramatic implications on bath chemistry control. Both of these factors motivate continuing efforts to quantify the affect of pot operating practices on fluoride evolution. Gaseous fluoride evolution measurements were made at several smelters and quantitatively demonstrate a strong correlation between the amount of vapor-phase fluoride evolved from individual pots and the total area of open holes in the crust. Data from pots of different operating types suggests that it is possible to normalize the data to allow predictions for other pot sizes and geometries. In many cases, the additional fluorides lost due to holes in the crust comprise the largest fraction of the total gaseous evolution.

9:20 AM
In-Plant PFC Monitoring: Technology Options and Performance Concerns: *Neal R. Dando*[1]; [1]Alcoa Inc., 100 Technical Dr., Alcoa Ctr., PA 15069-0001 USA

Commercial aluminum production is the largest emission source of the perfluorocarbon gases (PFCs) carbon tetrafluoride(CF4) and hexafluoroethane (C2F6). Given the high global warming potentials of these two gases, concerted efforts exist to monitor and reduce future emissions of PFCs from aluminum smelters. Several commercially available technologies exist for enabling capable off-line and on-line in-plant monitoring of PFC emissions. The local conditions in aluminum smelters (duct gas composition, magnetic fields, etc.) present a hostile environment for most instrumentation, which can compromise the accuracy and stability of the analysis method. This paper will detail a comparative evaluation of the advantages and disadvantages of different technologies for off-line and/or on-line in-plant PFC monitoring. Practical concerns regarding sample handling and method implementation will be discussed with respect to each monitoring approach.

9:45 AM
Tunable Laser Diode Technology for Continuous HF Monitoring in Potrooms: *Carl Kamme*[1]; [1]Opsis, Inc., 1165 Linda Vista Dr., San Marcos, CA 92069 USA

A tunable diode laser (TDL) provides an excellent tool for potroom open path measurements of gaseous HF. The TDL provides quick and exact concentration data for HF from one or several potrooms. Monitoring is provided for reporting purposes, and also process control. Particular events in the smelting process can be measured and evaluated in terms of gaseous emissions, which allows for savings and process improvements. Technology now supports automatic system alignment of the optical laser beam, which compensates for any changes in building structure caused by thermal stress or crane movements.

10:10 AM Break

10:20 AM
A Streamlined Portable Mid-IR TDL Based System for On-Site Monitoring of PFCís from Potroom Exhaust Ducts: *H. A. Gamble*[1]; D. R. Karecki[1]; G. I. Mackay[1]; H. I. Schiff[1]; [1]Unisearch Associates, Inc., 96 Bradwick Dr., Concord, Ont. L4K 1K8 Canada

Maintaining comprehensive and up to date records of the quantities of perfluorocarbons (PFCís) emitted from potroom exhaust ducts is an issue of outstanding concern to primary producers of aluminium worldwide. Unisearch has previously demonstrated the utility of mid-IR tunable diode laser based instrumentation for performing detailed, accurate, on site measurements of PFCís (CF4 and C2F6). The Unisearch TAMS-150 has been used for on-site measurements of PFCís at primary aluminium smelters across Canada, over a period of time spanning more than ten years. Unisearch now presents a more streamlined version of the instrument, the EMS-150, capable of performing the same experiment and of producing similar or better quality results. The instrument has been developed at Unisearch in response to ongoing industry needs, and is available either as a commercial instrument or on a contract measurement basis.

10:45 AM

Protocol for Measurement of Tetrafluoromethane and Hexafluoroethane from Primary Aluminum Production: *Jerry Marks*[2]; *Ravi Kantamaneni*[1]; *Diana Pape*[1]; *Sally Rand*[3]; [1]ICF Consulting, 1850 K St. NW, Ste. 1000, Washington, DC 20006 USA; [2]J. Marks and Associates, Kansas City, MO 64064 USA; [3]US Environmental Protection Agency, 1200 Pennsylvania Ave. NW, 6202J, Washington, DC 20460 USA

Two perfluorinated compounds (PFCs), tetrafluoromethane (CF4) and hexafluoroethane (C2F6), are produced periodically during primary aluminum production by the Hall-Heroult process. The atmospheric concentration of these two compounds is currently about 81 parts per trillion by volume (pptv) CF4 and 3 pptv C2F6 based on projections of previously reported measurements. While the annual amount of CF4 and C2F6 released to the atmosphere is small in comparison to other greenhouse gases, the environmental impact of each yearís emissions will persist for generations due to their long atmospheric lifetimes, estimated at 50,000 and 10,000 years, respectively. The measurement of PFCs is required to assist with developing the most accurate greenhouse gas emissions inventories. These inventories can be used to support company benchmarking and process improvement activities, to facilitate reporting under the United Nations Framework Convention on Climate Change, and to assist with the implementation of emissions trading mechanisms. This paper describes a measurement protocol to assist with developing accurate PFC inventories based on the Intergovernmental Panel on Climate Change (IPCC) Tier 3b method. The protocol has been produced with an aim to foster consistency in smelter-specific sampling programs. The procedure described here provides a method for making accurate measurements of CF4 and C2F6 emissions and relating those measurements to anode effect process data. A methodology is presented for summarizing the collected data and calculating IPCC Tier 3b emission factors, which provide for the most accurate calculation of long term PFC emissions from aluminum production. The protocol provides the necessary guidance to allow individual facilities to develop detailed plans for sampling and analysis based on plant specific technology, anode effect data, and chosen measurement instrumentation.

11:10 AM

Measuring Reporting and Verifying GHG Emissions from Aluminum Smelting Operations: *Kenneth Joseph Martchek*[1]; [1]Alcoa Inc., ABSS, 201 Isabella St., ACC-5B08, Pittsburgh, PA 15212 USA

Emissions of gases such as CO2 which retain heat in our atmosphere from bauxite refining, anode production, aluminum smelting and indirectly from electricity to power aluminum electrolysis has been estimated worldwide at 350-400 million metric tons of carbon dioxide equivalents per year. In particular, persistent greenhouse gases, CF4 and C2F6, are emitted during ìanode effectsî and the worldwide industry has responded by reducing these perfluorocarbon emissions by 46% since 1990. Nevertheless, accurately measuring, reporting and verifying all significant greenhouse gas emissions from aluminum operations is becoming increasingly important because of emerging schemes related to taxation, allowances, emissions trading, regulatory and reporting requirements. This paper highlights recent efforts in developing a robust and coherent greenhouse gas emissions inventory for alumina refining, carbon consumption and aluminum electrolysis in todayís regime of developing corporate, regional, state and international protocols. This paper will highlight practical ìlessons learnedî from a pilot third-party verification of greenhouse gas emissions measurements and reporting at an alumina refinery, an aluminum smelter and a dedicated coal-based electricity generator. In particular, this paper will discuss issues such as data quality, records retention and integration of emissions measurement into a locationís ISO 14,001 compliant Environmental Management System.

11:35 AM

PFC Emissions Measurements from Aluminium Pechiney Smelters: *CÈline Martin*[1]; Elisabeth Couzinie[1]; [1]Aluminium Pechiney, LRF, BP 114, Saint-Jean-de-Maurienne, Cedex 73303 France

Emissions of CF4 and C2F6 were measured with a photoacoustic gas spectrometer for three different AP smelters: 300 kA PFPB pots, 93 kA PFPB pots and 105 kA SWPB pots. Gases were directly extracted from the exhaust ducts between the pots and the dry Gas Treatment Centre to obtain continuous measurements. Fugitive emissions were considered to be insignificant for the modern PFPB pots whereas samples of gases were collected from the roof exhaust gas of the SWPB smelter. In this case, a collection efficiency was determined for PFC during anode effects. For each smelter, emissions measured were related to the anode effect overvoltage in order to be confronted to the Aluminium Pechiney equation. For the 300 kA PFPB pots, anode effect overvoltage and anode effect duration were also recorded in order to compare the Pechiney Overvoltage method and the Slope method. Relevance and accuracy of both methods is discussed.

Carbon Technology - I

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Amir A. Mirchi, Alcan Inc., Arvida Research and Development Centre, Jonquiere, QC G7S 4K8 Canada; Don T. Walton, Alcoa Inc., Wenatchee Works, Malaga, WA 98828-9784 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday AM	Room: 6D
March 4, 2003	Location: San Diego Convention Center

Session Chair: Frank Cannova, BP, Petroleum Coke Marketing, Long Beach, CA 90802 USA

8:30 AM

Formulation/Structure and Properties of Carbon Anodes from Coal-Tar Pitch/Petroleum Pitch Blends: Marta PÈrez[1]; *Marcos Granda*[1]; Ricardo SantamarÌa[1]; Roberto GarcÌa[1]; Rosa MenÈndez[1]; [1]Instituto Nacional del CarbÛn, CSIC, Chem. of Matls., C/Francisco Pintado Fe, 26, Oviedo, Asturias 33011 Spain

During the last decade much effort has been devoted to overcoming environmental problems derived from the use of traditional coal-tar pitch in Sˆderberg pot rooms. To this end, coal-tar pitch/petroleum pitch blends have been developed with the aim of obtaining a low PAH content. This would result in lower PAH emissions. The use of these blends in the preparation of carbon anodes requires a new paste formulation. This is because blends are usually more fluid than coal-tar pitches. For this reason, modifications in the granulometric coke fractions and binder percentage must be made. This paper reports on the formulation of pastes from coal-tar pitch/petroleum pitch blends and on the structure and properties of the resultant carbon anodes.

8:55 AM

Precise Measurement of Density of Coal Tar Oil and Liquid Binder Pitch: *Kwangeui Yoon*[1]; Myoung Won Lee[1]; Dong Joon Lee[1]; Se In Yang[1]; [1]DC Chemical Company, Ltd., R&D Ctr., 587-102, Hakik-Dong, Nam-Gu, Incheon 402-772 Korea

The density of liquid pitch is an essential factor for the mass of the pitch amount at elevated temperature. To operate anode plant at optimum condition, accurate weight measurement of liquid binder pitch is very important. However there has been no adequate system to measure the precise density at high temperature and not much data in public. We modified the conventional density measurement system of buoyant force to a system with high temperature measuring cell which could measure the liquid density at high temperature with very high accuracy. Using this system, we measured high temperature densities of coal tar fractions. Also densities of various coal tar binder pitches were measured in the temperature range of 190-210°C where liquid pitch is practically handling. We tried to correlate these high temperature density with their typical properties.

9:20 AM

Worldwide Pitch Quality for Anodes: *Raymond Perruchoud*[1]; Kirstine Hulse[2]; Markus Meier[1]; Werner Fischer[1]; [1]R&D Carbon, Ltd., Sierre 3960 Switzerland; [2]Alusaf Empageni S. Africa

A worldwide survey made on aluminium smelter supplies provided valuable information on the typical values and specifications for coal tar pitch based binders. An overview of ISO analytical standards used for characterizing pitch is given. The evaluation of the last decodes in the coke oven and tars distillers industry is addressed. The future requirements of the Al-industry are discussed.

9:45 AM
Change of Focus: Use of Refinery Simulation Tools for Coke Quality Related Predictions: *Eberhard Lucke*[1]; [1]KBC Advanced Technologies, Inc., Process Consulting, 580 Westlake Park Blvd., Ste. 1150, Houston, TX 77082 USA

Simulation models help to optimize refineries and petrochemical plants. The same principle can be used to optimize the anode grade coke chain. Potential benefits of this application are increased process efficiency, reduced energy consumption and reduced raw material cost. This paper discusses the results of using a standard Delayed Coker model to predict coke related quality parameters and the effect of process changes on these parameters. Besides a representative anode grade base case the following case studies are analyzed: -Decreased Recycle Ratio -Decreased Drum Pressure -Ramped Coil Outlet Temperature -Blending of 5% S+ Crude Oil. The predicted impact on yield structure, energy consumption and product qualities is presented. The possibility of using the coke quality data with commonly known correlations to estimate calcined coke qualities is evaluated. The discussion of these results shows the gap to best practice and leads to necessary measures for improvement.

10:10 AM Break

10:20 AM
Importance of Granulometry in Calcined Petroleum Coke: *Ravindra Narayan Narvekar*[1]; Ajit Sambhaji Sardesai[1]; Asht B. Prasad[1]; [1]Goa Carbon, Ltd., Production, Dempo House, Campal, Panaji, Goa 403001 India

Goa Carbon Limited, flagship company of DEMPO group, is one of Indiaís largest calciners having three rotary kilns in India and wide customer network across the globe. GCL has sieved out different grain sizes from one CPC lot and analysed the same for purity, structure and porosity parameters. This exercise has been repeated for different origin CPC lots with widely varying sulphur content. It has been observed that there is significant variation in chemical composition for different grain sizes belonging to same CPC lot indicating that some of the impurities have tendency to concentrate in finer grains. Significant variation in the structure and porosity parameters also seen among different grain sizes of same CPC lot indicating difference in Calcination levels undergone by them under otherwise identical conditions. These findings are utilised as a tool to emphasize proper sample preparation especially when sieving, crushing, grinding operations are involved.

10:45 AM
Classification of Pores in Prebake Anodes Using Automated Optical Microscopy: *Lorentz Petter Lossius*[1]; Stein R□rvik[2]; Harald A. ÿye[3]; [1]Hydro Aluminium Teknologisenter ≈rdal, PO Box 303, ÿvre ≈rdal N-6882 Norway; [2]SINTEF Applied Chemistry, Sem SÊlands veg 12, Trondheim N-7465 Norway; [3]Norwegian University of Science and Technology, Dept. of Chem., Sem SÊlands veg 12, Trondheim N-7491 Norway

The porosity influences anode properties as strength and reactivity. There are several identifiable types of pores; the most important are calcination cracks and gas bubble pores in the petrol coke and mixing and baking pores formed during production. An automatic image analysis method for classification has been developed to allow separate quantification of these different types of pores. The paper will present the method and show examples of pore distributions in green and baked anodes, dependent on recipe, pitch level, vibration forming parameters and process temperatures. It is especially useful to quantify the mixing porosity to study effects of the production process.

11:10 AM
Anode Quality Improvements at the Valesul Smelter: *Gustavo Franca*[1]; Carlos Mesquita[2]; Les Edwards[3]; [1]Valesul Aluminio S/A, Carbon Plant, Estr. Aterrado do Leme, 1225-Sta. Cruz, Rio de Janiero 23579-900 Brazil; [2]Billiton; [3]CII Carbon, LLC, 700 Coke Plant Rd., Chalmette, LA 70044 USA

Valesul Aluminio S/A is a 93,000 tonne per year smelter located in the suburbs of Rio de Janeiro, Brazil. Historically, Valesul has used a low sulfur petroleum coke for anode manufacture. In a drive to improve anode quality and performance, Valesul started an experiment in 2001 to evaluate the cost and potential benefits associated with using an alternative, higher sulfur coke. The paper describes the methodology and results of a four month plant trial, which simultaneously produced low and high sulfur anodes. High and low sulfur anodes were sent into two different sectors of the potline. Improvements in both anode quality and potline performance demonstrated the significant benefits possible when switching coke qualities in a smelter. Valesul is now evaluating the capital cost and return on a project which would increase its coke storage capacity and allow it to run with a blend of low sulfur and high sulfur cokes.

Cast Shop Technology: DC Casting II

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; David H. DeYoung, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday AM Room: 6C
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Christian Pluchon, Pechiney Group, Voreppe 38341 France; Laurens Katgerman, Delft University of Technology, Fac. of Cheml. Eng. & Matls. Sci., Delft 2628 AL Netherlands

8:30 AM Cancelled
Evaluation of Commercial CFD Packages Used in Mathematical Modeling of Direct Chill Casting of Aluminum Alloys: *Daniel Paul Cook*[1]; Christopher Thompson[1]; [1]Virginia Commonwealth University, Mechl. Eng. Dept., 601 W. Main St., PO Box 843068, Richmond, VA 23284-3068 USA

8:55 AM
Modelling of Fluid Flow Phenomena During DC Casting of Aluminium Alloys: *Laurens Katgerman*[1]; Bart Venneker[2]; Jan Zuidema[2]; [1]Delft University of Technology, Lab. of Matls., Rotterdamseweg 137, Delft, ZH 2628 AL The Netherlands; [2]Netherlands Institute for Metals Research, Rotterdamseweg 137, Delft, ZH 2628 AL The Netherlands

The major drive for casting modelling is to improve the insight how process parameters affect casting performance, in order to prevent casting defects. Reliable calculations of macrosegregation during casting of alloys depend on accurate modelling of the associated physical mechanisms. In solving the solute concentration equation, the accuracy of the velocity field is thus of great concern. Several discretisation schemes for the convection terms are examined on their ability to correctly predict macrosegregation. The occurrence of oxides can give major problems during aluminium casting and processing. During transfer from conventional metal treatment systems to the casting station pick up of inclusions can occur. In our simulations, we have assessed quantitatively some of the upstream fluid flow effects in relation to filling behaviour of the DC mould cavity and number of inclusions trapped in the launder system. Results of numerical simulations of fluid flow with discrete particles of different mass and distribution are given.

9:20 AM
Detailed Modelling of a Metal Distributor by Means of a Combined Numerical and Physical Approach: *RenÈ Kieft*[1]; Sjaak van Oord[1]; Ferry Frinking[1]; Dico Bal[1]; Hugo van Schoonevelt[1]; [1]Corus RD&T, PB 1000, IJmuiden 1970 CA The Netherlands

During the VDC casting process of ingots, the metal flow into the mould is mostly distributed by using a glass cloth distributor. The design of the distributor strongly determines the generated flow pattern within the liquid pool and this on its turn effects the ingot properties after solidification. For an accurate prediction of the flow pattern in the liquid pool it is necessary to model the flow through the distributor very precisely. Furthermore, the flexible character of the distributor bag demands for taking into account the deformation of the distributor caused by the flow. In most cases such a detailed modelling is unfeasible. Therefore the 3D velocity field at the outflow ports of the distributor was measured by means of an advanced measuring technique called Particle Image Velocimetry. The measurements were performed within a water model. The measured distributor outflow data was then used as inlet boundary conditions in the numerical fluid flow model. The results show that the details of the flow caused by the flexible distributor bag could be captured accurately. The influence of these details on the flow characteristics within the sump are elucidated by comparing the results with calculations in which the distributor was fully modelled. From the results it can be concluded that for a valid prediction of the flow patterns in the sump it is obligatory to incorporate all details caused by the flexible distributor bag.

9:45 AM
Effect of Water Quality and Water Type on the Heat Transfer in DC Casting: *Laszlo I. Kiss*[1]; Thomas Meenken[1]; Andre Charette[1]; Yves Lefebvre[2]; Robert LÈvesque[3]; [1]UniversitÈ du QuÈbec ‡ Chicoutimi, DSA, 555 Blvd. de líUniversitÈ, Chicoutimi, Quebec G7H 2B1 Canada; [2]BetzDearborn Canada, Inc., 3451 Erindale Station Rd., Mississauga, Ontario L5A 3T5 Canada; [3]Alcoa Aluminium Smelter in Baie-Comeau, 100, rte. Maritime, Baie-Comeau, Quebec G4Z 2L6 Canada

In the recent years many efforts have been directed toward the analysis of the influence of surface roughness, surface quality water temperature and composition on the heat transfer coefficients between the solidifying ingot and cooling water during the secondary cooling in DC casting. An experimental technique was developed earlier to follow closely the variations of the surface heat flux without modifying the surface properties of the solid. The method was used to analyze the effects of the type and composition of water and that of the cleanliness of the solid surface on the heat extracting capacity of the cooling water.

10:10 AM Break

10:20 AM
Thermomechanical Analysis of the DC Casting Start-Up Phase for AA5182: *Neill J. McDonald*[1]; Joydeep Sengupta[2]; Mary A. Wells[2]; Daan M. Maijer[2]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15232 USA; [2]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Recently thermo-mechanical finite element (FE) models based on fundamental principles have emerged as powerful tools to predict the temperature distribution and stress-strain fields in the solidifying aluminum ingot during the Direct Chill (DC) casting process. A critical aspect of these process models, is the way the constitutive behaviour of the material is modeled in the solid state as this can influence the stress, strain and butt curl predictions during the casting process. This paper compares four different methods of modelling constitutive behaviour, in the solid state, available in the commercial FE package, ABAQUS, namely: elastic-plastic, elastic-rate dependent plastic, creep, and combined creep and rate dependent plastic using data measured on as-cast AA5182 at strain rates and temperatures consistent with those typically seen during DC casting. The comparison of the different methods was done using a 2-D uncoupled axisymmetric thermal and stress model of an aluminum billet during the start-up phase of the DC casting process.

10:45 AM
Computing Free Surface Film of Liquid Metal During Mould Filling: *Jiawei Mi*[1]; [1]The University of Birmingham, Sch. of Eng., IRC in Matls. Procg., Elm Rd., Edgbaston, Birmingham, England B15 2TT UK

The mould filling of gravity top pouring and tilt pouring of an Al-4.5% Cu alloy has been studied by using a real-time X-ray video radiography system and a computational fluid dynamic code (Flow 3D). The liquid metal flow exhibited tranquil, turbulent or very chaotic phenomena due to the different filling conditions used in the experiment. The moving free surface of liquid metal was accurately tracked and various flow features were precisely presented by modeling. Based on an optimized filling sequence (free from surface turbulence), a method was developed to quantify the potentially entrained free surface film for the mould fillings that experienced free surface turbulence. The defects of the castings which experienced different filling phenomena were measured and analyzed. It revealed that the increase of the potentially entrained surface film resulted in the monotonous increase of the number and the average size of the defects in the castings.

11:10 AM
Mathematical Modelling of Water Ejection and Water Incursion During the Start-Up Phase of the DC Casting Process: *J. Sengupta*[1]; D. Maijer[1]; M. A. Wells[1]; S. L. Cockcroft[1]; A. Larouche[2]; [1]University of British Columbia, Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Alcan International Ltd., Arvida R&D Ctr., 1955 Mellon Blvd., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada

A comprehensive mathematical model has been developed to describe heat transfer during the start-up phase of the Direct Chill casting process. This 3-D thermal model, based on the commercial finite element package ABAQUS, includes primary cooling to the mould, secondary cooling to water and ingot base cooling. The algorithm used to account for secondary cooling to water includes boiling curves that are a function of surface temperature, water flow rate and position relative to the point of water impingement. Based on experimental measurements and observations, the algorithms used to describe secondary cooling and ingot base cooling have been augmented to include the physical phenomena of water ejection from the ingot surface and water incursion between the ingot base and bottom block surface. The model has been validated against temperature measurements obtained from two 711 x 1680 mm AA5182 ingots, cast under different conditions (non-typical ìcoldî practice and non-typical ìhotî practice). Comparison of the model predictions with the thermocouple data indicates that this 3-D thermal model correctly describes the heat flow in the early stages of the casting process throughout the ingot and bottom block.

11:35 AM
Understanding DC-Cast Microstructures Using Laboratory-Based Solidification Techniques: *Mike William Meredith*[1]; [1]Alcan International Ltd., Banbury Labs., Southam Rd., Banbury, Oxon OX16 2SP UK

Many of the microstructural features that exist in a DC-cast Al alloy ingot survive subsequent processing steps and contribute to the final properties of the product. For example, the formation of constituent particles may affect ìfir-treeî generation and, hence, surface quality in commercial purity alloys and downstream texture development in beverage can sheet. It is important, therefore, to understand which of the microstructural features generated during solidification are most important and, where feasible, how to control or influence them. Investigating commercially produced material, however, is expensive so it is becoming increasingly important to study solidification microstructure formation from within the laboratory. To this end, there are a number of tools available (for example, controlled directional solidification, DC-casting simulation, laboratory scale DC-casting, numerical simulations). This paper aims to assess how accurate and reliable such methods are and the precautions that should be taken when comparing the results of these to full-scale DC-cast material.

Computational Methods in Materials Education: New Computational Courses

Sponsored by: TMS-Education Committee
Program Organizers: Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, State College, PA 16082-5006 USA; Mark D. Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA

Tuesday AM — Room: 13
March 4, 2003 — Location: San Diego Convention Center

Session Chair: Mark D. Asta, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208-3108 USA

8:30 AM Invited
Graduate Course in Materials Modeling at Princeton: *David J. Srolovitz*[1]; [1]Princeton University, Princeton Matls. Inst., 70 Prospect Ave., Bowen Hall, Princeton, NJ 08544-5211 USA

Computational Materials Modeling is a regular graduate course for first year graduate students at the Princeton Materials Institute. It is designed to familiarize the graduate students with a wide range of simulation methodologies from first-principles to atomistic to microstructural to continuum. The course focuses on understanding the methods such that each student is able to write a simple computer simulation code. During this course, the students are assigned four projects which require them to write their own simulation codes. I will discuss the philosophy of the computer simulation course, the subject matter covered, as well as the mechanisms employed to make it possible for students to write their own simulation codes.

9:00 AM Invited
Introduction to Modeling and Simulation: A Multidisciplinary Approach to Computational Materials Education: *Adam C. Powell*[1]; Sidney Yip[2]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139-4301 USA; [2]Massachusetts Institute of Technology, Dept. of Nucl. Eng., 77 Massachusetts Ave., Rm. 24-208, Cambridge, MA 02139-4301 USA

Introduction to Modeling and Simulation, first taught in Spring 2002, is a broad overview of computation for processing-structure-property relationships. This subject draws lecturing faculty from the departments of Materials Science and Engineering, Nuclear Engineering, Applied Mathematics, Civil and Environmental Engineering, Health Sciences and Technology and Chemical Engineering, with undergraduate and graduate student representation from a similar diversity of backgrounds. First half lectures cover modeling methodologies with assignments spanning lengthscales from electronic structure of simple molecules, to percolation clusters, to Ising models, to finite difference modeling of heat conduction and phase change. Students spend the second half working on term projects, with lectures covering recent research breakthroughs making use of methodologies taught in the first half. This talk will focus on experiences in managing the diverse backgrounds of students and faculty, both in terms of differences in approach between departments, and also varying mathematical and computational backgrounds of the students.

9:30 AM Invited
A New Course Integrating Computational and Experimental Methods in Materials Engineering: *James B. Adams*[1]; Shahriar Anwar[2]; [1]Arizona State University, Cheml. & Matls. Eng., PO Box 876006, Tempe, AZ 85287-6006 USA

In response to feedback from our Advisory Board, we created a new course, MSE 394 Computational and Experimental Methods in Materials Engineering. This course is intended to follow our introductory materials course. The course was intended to provide students with an integration of experimental and computational skills, as will be expected by future employers. This course includes an introduction to Fortran programming, UNIX, LabView programming/data acquisition, crystal building/modeling, OOF (microstructure evaluation and mechanical behavior modelling), Design of Experiments (concrete optimization), 4-point probe measurements, and magnetic bubble behavior. Throughout the course, computational and experimental methods are integrated as much as possible.

10:00 AM Break

10:30 AM Invited
Object Oriented Finite Element Modeling (OOF) of Microstructures in a New Course in Materials Engineering: *Shahriar Anwar*[1]; James B. Adams[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA

We are teaching a new course entitled Computational and Experimental Methods in Materials Engineering aimed at the sophomore/junior level in materials engineering. One of the topics covered in this course is microstructure-based simulation using finite element analysis techniques. The tool that we use is a public domain software called OOF (Object Oriented Finite Element) developed at the National Institute of Science & Technology (NIST). We will discuss our implementation of OOF in this course, as well as improvements and ongoing development based on feedback from our students.

11:00 AM Invited
Incorporating Computational Materials Research into Graduate Courses: A Hands-On Approach: *Diana Farkas*[1]; [1]Virginia Tech, Matls. Sci. & Eng., Blacksburg, VA 24061 USA

We describe a hands on approach to incorporating computational materials research into the graduate curriculum. The approach involves the students actually using atomistic simulation codes as a homework tool. The approach has been used in a graduate course on computational techniques in materials science, and is also appropriate as a teaching tool in other courses, such as dislocation theory, and fracture mechanics. The examples used involve calculations ranging from simple defects, such as surface energies and simple vacancies to dislocation core structure and simulations of fracture.

11:30 AM Invited
Facing the Challenge of Biological Materials: From DNA to Self-Assembled Bilayers: *Rob Phillips*[1]; [1]Caltech, Div. of Eng. & Appl. Sci., 1200 E. California Blvd., Pasadena, CA 91125 USA

Model building in materials science is a crucial element of the drive to construct a new generation of materials based upon fundamental understanding as opposed to enlightened empiricism. One class of materials of ever increasing importance is that of ìbiological materialsî ranging from the synthesis of artificial proteins to the exploitation of molecular motors and beyond. Successful use of this broad class of materials represents a truly interdisciplinary endeavor and poses intriguing challenges from the standpoint of education since it draws from biology, chemistry, physics, materials science and other domains as well. This talk will examine some of the beautiful models that have been set forth recently to respond to experiments in single molecule biology and to show how such models (and their computational incarnations) and the associated experiments can be brought to the classroom setting.

Computational Phase Transformations: Fundamental Properties of Surfaces and Interfaces

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Tuesday AM Room: 11B
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Wolfgang Windl, The Ohio State University, Dept. of Matls. Sci. & Eng., Columbus, OH 43210 USA; Jeffrey J. Hoyt, Sandia National Laboratories, Matls. & Process Modlg., Albuquerque, NM 87122 USA

8:30 AM Invited
Accelerated Molecular Dynamics: *Arthur F. Voter*[1]; [1]Los Alamos National Laboratory, Theoretl. Div., MS B268, Los Alamos, NM 87545 USA

A significant problem in the atomistic simulation of materials is that molecular dynamics simulations are limited to nanoseconds, while important reactions and diffusive events often occur on time scales of microseconds and longer. Although rate constants for slow events can be computed directly using transition state theory (with dynamical corrections if necessary), this requires first knowing the transition state. Often, however, we cannot even guess what events will occur. For example, in vapor-deposited metallic surface growth, surprisingly complicated exchange events are pervasive. I will discuss recently developed methods (hyperdynamics, parallel replica dynamics, and temperature accelerated dynamics) for treating this problem of complex, infrequent-event processes. The idea is to directly accelerate the dynamics to achieve longer times without prior knowledge of the available reaction paths. I will present our latest method developments and some recent applications, including metallic surface growth and deformation of carbon nanotubes.

9:00 AM Invited
Computing the Kinetic Coefficient in Solid-Liquid Systems from Molecular Dynamics Simulations: *Jeffrey J. Hoyt*[1]; Deyun Sun[2]; Mark D. Asta[2]; Alain Karma[3]; [1]Sandia National Laboratories, Matls. & Process Modlg., Bldg. 897, MS 1411, Albuquerque, NM 87122 USA; [2]Northwestern University, Dept. of Matls. Sci. & Eng., Robert R. McCormick Sch. of Eng. & Appl. Sci., 2225 N. Campus Dr., Evanston, IL 60208 USA; [3]Northeastern University, Physics Dept., 111 Dana Rsrch. Ctr., 110 Forsyth St., Boston, MA 02115 USA

The kinetic coefficient is the constant of proportionality between the velocity of a solid-liquid interface and the interface undercooling. The value of the kinetic coefficient and its dependence on growth direction are critical parameters in continuum modeling of dendritic solidification. In the present work three methods of extracting the kinetic coefficient from molecular dynamics simulations will be discussed. The technique of fluctuation kinetics tracks fluctuations of the solid-liquid interface for a system maintained at the melting point. A correlation function describing the time dependence of the k-space fluctuation amplitude can then be used to derive the kinetic coefficient. A second method, free solidification, employs constant NPT simulations and monitors the interface position as a function of time. The free solidification technique will be discussed paying particular attention to the means by which pressure is controlled. Finally, the imposed pressure technique employs solid-liquid systems main tained at the melting point, but with high pressures applied normal to the interface. The pressure induced undercooling is obtained from the Clausius-Claperyon relation. All three techniques will be compared for the case of pure Ni modeled using interatomic potentials of the embedded atom type and rapid solidification simulations will be used to study the phenomenon of disorder trapping in Ni-Al alloys.

9:30 AM Invited
Atomistic Simulations of Alloy Solid-Liquid Interfaces: *Mark D. Asta*[1]; Deyan Sun[1]; Jeffrey J. Hoyt[2]; Alain Karma[3]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208-3108 USA; [2]Sandia National Laboratories, Albuquerque, NM 87122 USA; [3]Northeastern University, Boston, MA 02115 USA

We demonstrate the application of Molecular-Dynamics and Monte-Carlo simulation methods to the calculation of solid-liquid interface properties relevant for the evolution of solidification microstructure in metallic alloys. Interfacial free energies, and their associated crystalline anisotropies, are computed from capillary fluctuation spectra derived from equilibrium molecular-dynamics simulations. We further discuss how interface mobilities in alloy systems can be extracted from analyses of capillary-fluctuation relaxation kinetics. These approaches are applied to studies of solid-liquid interface properties in both Cu-Ni and Al-Cu alloy systems modeled by many-body interatomic potentials of the embedded-atom form. In order to examine effects of solute upon both kinetic and thermodynamic interface properties, calculated results for alloys are compared in both cases to corresponding values in the associated pure solvent materials.

10:00 AM Break

10:15 AM Invited
Direct Calculations of Crystal-Melt Interfacial Free Energies for Simple Materials: *Brian B. Laird*[1]; [1]University of Kansas, Chem., 1251 Wescoe Hall Dr., Lawrence, KS 66045 USA

Using a recently developed molecular-dynamics simulation method [Davidchack and Laird, Phys. Rev. Lett. 85,4751 (2000)], we determine the crystal-melt interfacial free energy for the hard-sphere and Lennard-Jones model systems as functions of interfacial orientation.In addition, it is shown that Turnbullís rule for the crystal-melt interfacial free energy of close packed metals can be explained with quantitative accuracy solely using the hard-sphere model, indicating that this quantity is primarily entropic in origin.

10:45 AM Invited
The Dependence of the Anisotropic Solid-Liquid Interfacial Free Energy on Interatomic Potentials: *James R. Morris*[1]; Xueyu Song[2]; [1]Iowa State University, Ames Lab., USDOE, Metal & Ceram. Scis., Ames, IA 50011 USA; [2]Iowa State University, Dept. of Chem., 303 Wilhelm, Ames, IA 50011 USA

We have calculated the orientation dependence of the solid-liquid free energy, examining the effect of interatomic potentials. The free energy calculation examines the equilibrium fluctuations of the interface, using molecular dynamics simulations of the coexisting phases. The interfacial free energy of aluminum was calculated using both the Embedded Atom Method potentials of Ercolessi and Adams, and of Mei and Davenport, with the modification from Sturgeon and Laird. We compare the results with each other and with experimental results, and also compare the liquid structure calculated using these potentials with experimental results. We have also examined the interfacial free energy in a number of model systems, including the Lennard-Jones interaction and purely repulsive systems, to see how changing the potential affects the interfacial properties. Finally, we will present preliminary results on the direct calculation of the free energies of the solid and liquid phases, using a modified Weeks-Chandler-Anders on approach that eliminates the need for simulations, and explore the possibility of a similarly direct calculation of the interfacial free energy.

11:15 AM Invited
Interface Modeling: Chao Jiang[1]; Long-Qing Chen[1]; *Zi-Kui Liu*[1]; [1]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA

The key issue in integrating bulk thermodynamic/kinetic modeling and kinetic simulation is the modeling of interfaces separating two phases of the same structure or different structures. In this presentation, various interface modeling techniques will be reviewed and discussed in the frame work of the interface types, thermodynamic models, thermodynamics of solute segregation, and migration of interfaces. Particular attention will be paid to the integration of the CALPHAD-type thermodynamic and kinetic modeling with the phase-field-type diffuse interface.

Defects and Deformation of Crystalline Solids - in Honor of Dr. Man H. Yoo - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
Program Organizers: Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Department, Houghton, MI 49931-1200 USA; Sean R. Agnew, University of Virginia, Materials Science and Engineering Department, Charlottesville, VA 22904-4745 USA; K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA

Tuesday AM — Room: 17A
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: David Srolovitz, Princeton University, Mechl. & Aeros. Eng., Princeton, NJ 08544 USA; Hamish Fraser, Ohio State University, Dept. of Matls. Sci. & Eng., Columbus, OH 43210 USA

8:30 AM Invited
The Relationship Between the Onset of Plastic Flow in Nearly Perfect Silicon Samples and the Brittle to Ductile Transition: *David P. Pope*[1]; [1]University of Pennsylvania, Dept. of Matls. Sci. & Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

Nearly perfect single crystalline Si wafers have been extensively used in the past ten years to study dislocation activity at the BDTT because the absence of pre-existing dislocations in these samples means that all dislocations are generated at the transition. However to date, most of the experiments have been conducted on notched or pre-cracked samples because it is widely believed that the BDT results from some type of crack-tip phenomenon. We believe that the BDT occurs when a critical combination of stress and temperature results in the generation of an avalanche of dislocations in the material, permitting general yielding. In this view, a crack-tip is not necessary for the transition but it may serve as the stress concentrator that precipitates the avalanche of dislocations. To test this hypothesis, experiments were performed on a large number of dislocation free, notch free silicon specimens, produced using photolithography. These silicon beams were tested in bending at various elevated temperatures an d strain-rates and reveal a strain-rate dependent brittle-to-ductile transition in these defect-free samples. Dislocation etch-pit analysis of the sample surfaces show that massive dislocation activity occurs prior to yielding in the highest stressed portions of the beam. In addition, the scanning electron microscope images reveal a qualitative increase in dislocation activity above the BDTT, in agreement with the observed upper yield point behavior. The Khantha-Pope-Vitek model of the BDT is proposed to address these new observations.

8:55 AM
Dislocation-Solute Atom Interactions in Mo-W and Ta-W Alloys Studied by a Kinetic Monte Carlo Model: *Chaitanya S. Deo*[1]; David J. Srolovitz[1]; Wei Cai[2]; Vasily V. Bulatov[2]; [1]Princeton University, Mechl. & Aeros. Eng., Princeton, NJ 08544 USA; [2]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., Livermore, CA 94550 USA

Crystal plasticity in metals and alloys depends on the nature of interactions between dislocations and solutes in addition to the dislocation behavior that is present in pure metals. Dislocation Dynamics (DD) simulations require mobility laws that prescribe the behavior and velocity of a dislocation as a function of solute concentration, temperature and applied stress. We present a kinetic Monte Carlo (kMC) simulation of the glide of a single <111> oriented screw dislocation on the (011) plane in Mo and Ta in the presence of substitutional tungsten (W) atoms. The kMC simulation is used to develop mobility laws prescribing the dependence of the dislocation velocity on the driving force and on the inhibiting lattice and solute resistance. The W atoms are substitutional impurities distributed in three dimensions around the screw dislocation and create a small distortion in the Ta and Mo lattice. Both short range and long range solute-dislocation interactions are included in the model. The dislocation motion is r epresented by the kink model, which explicitly includes double kink nucleation, kink migration and kink-kink annihilation. We integrate over the initial evolution of the embryonic double kink by treating the double kink as a one-dimensional random walker and obtaining the distribution of the elapsed time before the double kink stabilizes. Incorporation of such a double kink nucleation model is a key to efficient simulations. The dislocation velocity shows an Arrhenius dependence on the temperature as the double kink nucleation process is thermally activated. The solutes inhibit the motion of the dislocation, leading to a reduction in the dislocation velocity, also called dislocation friction.

9:15 AM Invited

Strain Hardening by Mesoscale Simulations: *Ladislas P. Kubin*[1]; [1]LEM, CNRS-ONERA, 29 Av. de la Division Leclerc, BP 72, 92322 Chatillon, Cedex France

Strain hardening can be decomposed into a product of two contributions. One is due to forest hardening that relates the flow stress to dislocation densities, the other stems from the evolution of dislocation densities with time. In order to establish a relation with the continuum modelling of plastic deformation, strain hardening is expressed in the form of a matrix, whose components describe how the critical stress in one slip system depends on the plastic strain in other systems. The way dislocation dynamics simulations can contribute to remove arbitrariness in the definition of the hardening matrix is discussed on several examples. Forest hardening, the related interactions between slip systems and the evolutionary laws for dislocation densities are discussed in materials with low and high lattice friction. This will be illustrated for the case of fcc, bcc and hcp crystals.

9:40 AM

In Situ Studies of Deformation by Nanoindentation: Andrew Minor[1]; Erica T. Lilleodden[1]; Eric A. Stach[2]; *John William Morris*[1]; [1]University of California, Matls. Sci., Evans Hall, Berkeley, CA 94720 USA; [2]Lawrence Berkeley Laboratory, NCEM, 1 Cyclotron Dr., Berkeley, CA 94720 USA

We have used a unique nanoindentation stage mounted on a high-resolution electron microscope at NCEM/LBNL to observe, in situ, the nucleation and development of mechanical deformation during nanoindentation. This talk will focus on the deformation of Al. Indentation in the center of a relatively large grain produces deformation by the spontaneous generation of dislocations, suggesting the ideal behavior of defect-free crystals. Deformation near grain boundaries forces the participation of those boundaries, often through surprisingly large, recoverable excursions. The characteristic deformations will be described, shown and interpreted.

10:00 AM

Anomalous Slip in High-Purity Molybdenum Single Crystals: *Luke L. Hsiung*[1]; David H. Lassila[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., 7000 East Ave., L-352, PO Box 808, Livermore, CA 94551-9900 USA

Dislocation multiplication and propagation in high-purity Mo deformed by anomalous slip have been investigated. The result reveals that the initial screw dislocations associated with grown-in superjogs can act as effective sources for multiplying 1/2[111] and 1/2[1-11] coplanar screw dislocation arrays in the (-101) primary slip plane. The interaction force between the freshly multiplied 1/2[111] screw dislocations and 1/2[1-11] screw dislocations results in the multiplication of 1/2[1-11] screw dislocations, which leads to the formation of 1/2[111] and 1/2[1-11] cross-grid dislocation arrays in the (-101) primary slip plane. It is proposed that the occurrence of {0-11} anomalous slips is intimately related to the onset of a domino event of dislocation propagation caused by the internal stresses originating from the interactions between the cooperative screw dislocation arrays. The internal stresses render the propagation of 1/2[111] and 1/2[1-11] screw dislocations from the primary slip planes onto the {011} planes by a stress-induced cross-slip process, and subsequently resulting in the anomalous operation of slip systems in the {011} planes. This work was performed under the auspices of the US Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

10:20 AM Break

10:40 AM Invited

Computer Simulation of Defect Generation and Properties, and Dislocation-Obstacle Interactions in Metals: *David John Bacon*[1]; [1]The University of Liverpool, Dept. of Eng., Brownlow Hill, Liverpool L69 3GH UK

Recent advances in areas where computer modelling is being used for research into radiation damage, dislocation-obstacle interactions and twinning in metals will be reviewed. Defect production by displacement cascades, and the numbers and arrangements of point defects in these primary events are now well-understood. An unexpected degree of defect clustering has been revealed, and the mobility of defect clusters has been investigated and shown to play a potentially important role in the evolution of microstructure. Atomic-scale modelling is also being used in realistic simulation of the interaction of dislocations with obstacles such as dislocation loops, voids and precipitates, and the information gained can be used to assist continuum-scale modelling of yield and flow. In the field of twinning, simulation has been used to understand the important role of dislocations in interfacial processes.

11:05 AM

TEM Investigation of Nanoindentation Plastic Zones in an Alpha/Beta Ti Alloy: *Gopal B. Viswanathan*[1]; Eunha Lee[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The advent of nanoindentation technique has provided us with the ability to probe a smaller volume of material for mechanical properties such as elastic modulus, yield stress etc. in the areas of thin films, coatings and multiplayer materials etc. In this study, the same technique has been applied to evaluate the mechanical properties of Ti-Al based alloy containing alpha/beta two-phase microstructure. Specifically, nanohardness measurements were obtained from individual alpha and beta phases in these alloys. TEM samples were carefully prepared beneath specific indentations using Focused Ion Beam (FIB) slicing method and the deformation structures from these samples are investigated in detail. Attempts have been made to correlate the trends that are seen in hardness estimates to various microstructural features such as grain size, composition, orientation of individual phases supported by the advent of nanoindentation technique has provided us with the ability to probe a smaller volume of material for mechanical properties such as elastic modulus, yield stress etc. in the areas of thin films, coatings and multiplayer materials etc. In this study, the same technique has been applied to evaluate the mechanical properties of Ti-Al based alloy containing a two-phase ($\alpha+\beta$) microstructure. Specifically, nanohardness measurements were obtained from individual a and b phases in these alloys. TEM samples were carefully prepared beneath specific indentations using Focused Ion Beam (FIB) slicing method and the deformation structures from these samples are investigated in detail. Attempts have been made to correlate the trends that are seen in hardness estimates to various microstructural features such as grain size, composition, orientation of individual phases supported by Orientation Microscopy (OM) and detailed TEM analysis of the deformation structures.

11:25 AM Invited

Anomalous Hardening at High Temperatures in Gamma TiAl: *Sung H. Whang*[1]; Zhijie Jiao[1]; Qiang Feng[2]; Zhongmin Wang[1]; [1]Polytechnic University, Dept. of Mechl. Eng., Six MetroTech Ctr., Brooklyn, NY 11201 USA; [2]University of Michigan, Dept. of Matls. Sci. & Eng., Inst. of Sci. & Tech., M-13, IST Bldg., H. H. Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109 USA

Anomalous increase in yield stress at high temperatures has been observed in a number of intermetallics. The micromechanisms for anomalous yield stress in intermetallic alloys such as $L1_2$ Ni_3Al and Ni_3Ga were relatively well known. On the other hand, gamma TiAl exhibits complex dislocation features and diversified cross-slip profiles, which led to potential multiple-pinning mechanisms. Furthermore, the active dislocation types are different in Ti rich gamma TiAl alloys from those in Al rich TiAl alloys, which makes us to evaluate both roles of superdislocations as well as ordinary dislocations in slip. In the past, in addition to the static and dynamic observation of defect structures, theoretical effort based on the change in kink interaction energy in various slip planes has been carry out to understand the pinning mechanisms of active dislocations. In this presentation, we will present some of the experimental results as well as theoretical calculations, and attempt to shed new lights on the subject of anomalous hardening at high temperatures.

11:50 AM

Dynamic Strain Aging and Strength Anomaly of $L1_2$-Type Intermetallic Alloys at Intermediate Temperature: *Takayuki Takasugi*[1]; Hyabusa Honjo[1]; Yasuyuki Kaneno[1]; Hirofumi Inoue[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

The serrated plastic flow of $L1_2$-type Co_3Ti, $Ni_3(Si,Ti)$ and Ni_3Al intermetallic alloys in forms of poly- and single-crystals was investigated at intermediate temperature by tensile tests in terms of the effects of temperature, strain rate, composition and microstructure. The serrated plastic flow and negative strain rate sensitivity was generally observed at intermediate temperature in which positive temperature dependence of the flow stress has been shown. Such plastic instability was correlated with dynamic strain aging. It is suggested that solutes interact with a decomposed screw dislocation in its inelastic strain field, and modify the core structure of a decomposed screw dislocation, resulting in plastic flow instability. In this article, it is discussed how solutes as well as dislocation core structures affect the dynamic strain aging in $L1_2$ intermetallic alloys.

Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong: Shock Compression

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Marc Andre Meyers, University of California-San Diego, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; George T. Gray III, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA; Kenneth S. Vecchio, UC San Diego, Dept of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA

Tuesday AM Room: 16B
March 4, 2003 Location: San Diego Convention Center

Session Chair: N. N. Thadhani, Georgia Institute of Technology, Matls. Sci. & Eng., Atlanta, GA 30332-0245 USA

8:30 AM
Observations of Common Microstructural Issues Associated with Dynamic Deformation Phenomena: Twins, Microbands, Dynamic Recrystallization, and Grain Size Effects: *Lawrence E. Murr*[1]; [1]University of Texas at El Paso, Metallurgl. & Matls. Eng., 500 W. University Ave., El Paso, TX 79968-0520 USA

Plane wave shock deformation has been shown to produce deformation twins or twin-faults in essentially all metal and alloys. In FCC metals and alloys twinning depends upon stacking-fault free energy (SFE) and a critical twinning pressure; which increases with increasing SFE. For impact cratering where the shock wave is spherical and a prominent shear stress is involved, metals and alloys with high SFE form microbands coincident with {111} planes while low SFE metals and alloys either form mixtures of twins and microbands or twins. Oblique shock loading of copper also produces mixtures of twins and microbands. Both twins and microbands increase in volume fraction with increasing grain size. BCC iron is observed to twin in both shock loading and as a result of impact cratering. Impact craters, shaped charges, and other examples of extreme deformation and flow at high strain rates exhibit dynamic recrystallization as a mechanism of solid-state flow. Deformation twins are also often precursors to this process as well. Examples of these phenomena in FCC materials such as Al, Ni, Cu, stainless steel and brass and BCC materials such as W, Mo, W-Ta, Ta will be presented; with emphasis on transmission electron microscopy.

9:00 AM
Materials Science Under Extreme Conditions of Pressure and Strain Rate: *Bruce A. Remington*[1]; Grant Bazan[1]; Eduardo M. Bringa[1]; Maria J. Caturla[1]; M. John Edwards[1]; Daniel H. Kalantar[1]; Mukul Kumar[1]; Barbara F. Lasinski[1]; K. Thomas Lorenz[1]; James M. McNaney[1]; Stephen M. Pollaine[1]; David B. Reisman[1]; James S. Stolken[1]; Justin S. Wark[2]; Barukh Yaakobi[3]; [1]Lawrence Livermore National Laboratory, Y-Div., HED Prog., PO Box 5001, Livermore, CA 94551-9970 USA; [2]University of Oxford, Oxford UK; [3]University of Rochester, Lab. for Laser Energetics, Rochester, NY USA

Solid state experiments at very high pressures and strain rates are possible on high power laser facilities, albeit over brief intervals of time and small spatial scales. Bragg diffraction on single-crystal surrogates and VISAR measurements establish the ultrahigh strain rates of 10^7-10^8 s^{-1}. Solid-state strength is inferred using the Rayleigh-Taylor instability as a ìdiagnosticî. Temperature and compression in polycrystalline samples can be deduced from EXAFS measurements. Deformation mechanisms can be identified by examining recovered samples. We will briefly review this new area of laser-based materials science research, then present a path forward for carrying these solid-state experiments to much higher pressures, P > 10 Mbar, on the NIF laser facility. This work was performed under the auspices of the US Department of Energy by the Lawrence Livermore National Laboratory under Contract No. W-7405-ENG-48.

9:30 AM
Simulating Shock Propagation in Single Crystal Cu and Al: *Eduardo Marcial Bringa*[1]; Paul Erhart[1]; Nicolai Tanushev[1]; Maria Jose Caturla[1]; Brian D. Wirth[1]; Dan Kalantar[1]; James Stolken[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matl. Scis., PO Box 808, L-353, Livermore, CA 94551 USA

Large scale molecular dynamics simulations with the embedded atom method (EAM) potentials are used to simulate shock propagation in single crystal Cu and Al for different crystal orientations. Simulations were performed for a wide pressure range (2-300 GPa) and agree well with existing experimental data for both metals. Large anisotropies are found for shock propagation in Cu, with different plasticity mechanisms along different directions. Experiments for ìsingle crystalsî deal with samples having a small initial defect density, including point defects, dislocations, etcetera. Therefore, simulations were also run for crystals with vacancies and nano-void distributions before the shocks. The effect of the defects on the Hugoniot, the Hugoniot elastic limit and the resulting plastic deformation will be discussed. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

9:45 AM
High Strain Rate Deformation and Failure in Pre-Shocked Metals: *Geoffrey H. Campbell*[1]; James M. McNaney[1]; Mukul Kumar[1]; James S. Stolken[2]; F. Xabier Garaizar[3]; [1]Lawrence Livermore National Laboratory, Chem. & Matl. Sci. Direct., PO Box 808, MS L-356, Livermore, CA 94550 USA; [2]Lawrence Livermore National Laboratory, Eng. Direct., PO Box 808, L-342, Livermore, CA 94550 USA; [3]Lawrence Livermore National Laboratory, Ctr. for Appl. Scientific Compu., PO Box 808, MS L-561, Livermore, CA 94550 USA

Deformation microstructure evolution is a controlling aspect of metal dynamics in general and many types of failure observed in practice. There are large bodies of scientific information on both microstructural evolution and failure of ductile metals that start with a low defect density; however, less is known about highly defective metals. Shocks induce deformation microstructures with high dislocation densities that are comprised, for the most part, of statistically stored dislocations. This highly defective state allows recovery mechanisms to begin at an earlier point in the deformation behavior, reducing the work hardening rate and increasing the propensity for shear localization. We have induced shocks using laser drives in pure Ta, Ta - 2.5% W alloy, and pure Cu under a variety of conditions. We have measured their mechanical properties and characterized their microstructures in the pristine, shock processed and mechanically strained conditions with a variety of techniques, including EBSD mapping and TEM. We have also performed plane stress fracture mechanics tests with the double edge notched tension specimen. The mechanical response will be discussed in terms of the observed deformation microstructures. This work performed under the auspices of US Department of Energy by University of California, Lawrence Livermore National Laboratory under contract no. W-7405-Eng.-48.

10:00 AM
Atomistic Modeling of Spherical Shocks in Single and Nanocrystalline Copper: *Maria J. Caturla*[1]; Alison Kubota[1]; James S. Stˆlken[1]; James M. McNaney[1]; Bruce A. Remington[1]; Tomas Diaz de la Rubia[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., L-353, PO Box 808, Livermore, CA 94550 USA

Shock propagation has traditionally been studied using continuum models. However, under certain geometries, the boundary conditions can have an impact in the solutions obtained. Such a situation arises when studying spherical shocks. One way of overcoming this limitation is to use molecular dynamics simulations. We have studied the effect of spherical shocks with peak stresses between 5 and 40GPa on a copper target. The initial condition is a copper sphere 35 to 82nm in diameter. The influence of grain boundaries on shock propagation and energy dissipation is studied in nanocrystalline copper spheres. As a result of the applied deformation a void is generated at the center of the sphere. We analyze the formation of the void with respect to the type and strength of the shock applied. We correlate the size of the void with the peak stresses, the size of the original system, and compare to known scaling relations and with experiments. This work was carried out under the auspices of the US Department of Energy by Lawrence Livermore National Laboratory under contract W-7405-Eng-48.

10:15 AM Break

10:30 AM
Heterogeneous Nucleation of Shock-Induced Plasticity and Phase Transformations: *Timothy C. Germann*[1]; Brad Lee Holian[2];

Kai Kadau[3]; Peter S. Lomdahl[3]; [1]Los Alamos National Laboratory, X-7, MS D413, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, T-12, MS B268, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, T-11, MS B262, Los Alamos, NM 87545 USA

Large-scale (10^6 to 10^9-atom) molecular dynamics simulations have been used to study plastic deformation and polymorphic phase transformations under shock loading. In addition to the homogeneous perfect crystals typically studied using smaller-scale simulations, larger samples enable the introduction of specific extended defects, or even nanocrystalline samples. Such heterogeneous nucleation sites are found to substantially lower the Hugoniot elastic limit (as compared with perfect crystals), as well as the phase transformation or detonation threshold pressure for polymorphic and energetic materials, respectively. Three prototypical systems will be discussed: (1) fcc metals, represented either by a generic Lennard-Jones pair potential or by an embedded atom method (EAM) potential for Cu; (2) the bcc-to-hcp transformation of solid iron; and (3) model energetic materials, represented by the diatomic ìABî model bond-order potential.

10:45 AM
Influence of Grain Size on the Dynamic Deformation and Shock-Loading Response of Metals and Alloys: *George (Rusty) T. Gray*[1]; Shuh-Rong Chen[1]; Kenneth S. Vecchio[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

Ron Armstrong has made seminal contributions to the fields of high-rate deformation, constitutive modeling, and grain size effects on metal plasticity. This talk will discuss experimental results illustrating the role of grain size on the dynamic mechanical behavior, substructure evolution, and constitutive modeling of titanium, Monel 400, and zirconium. Increasing grain size is shown to significantly decrease the dynamic flow strength of Zr, Ti, and Monel 400 while increasing work-hardening rates in Ti and Zr due to an increased incidence of deformation twinning. The influence of grain size on dynamic mechanical behavior is discussed in terms of defect generation and storage mechanisms. The incorporation of grain size effects into constitutive models and validation using Taylor cylinder impact testing is presented.

11:00 AM
The Mechanical Properties of 304 Stainless Steel After Explosive Shock Prestraining: *S. A. Maloy*[1]; G. T. Gray[1]; C. M. Cady[1]; R. W. Rutherford[1]; R. Hixson[1]; [1]Los Alamos National Laboratory, MST-8, MS-H809, Los Alamos, NM 87545 USA

The mechanical properties of 304 stainless steel have been investigated in compression and tension at room temperature before and after explosive shock prestraining to a pressure of 35-45 GPa and compared to results from compressive testing of the annealed microstructure at a strain rate of 10-3/s. The microstructure evolution due to ìTaylor-waveî shock prestraining has been investigated using optical metallography and transmission electron microscopy. Mechanical testing results after explosive shock prestraining displays a factor of two increase in yield stress and a large reduction in ductility in tension. Microstructural analyses reveal increased twinning after ìTaylor-waveî prestraining and very little production of deformation induced martensite. The results are compared to previous results on high strain rate testing of 304 stainless steel specimens.

11:15 AM
Deformation Mechanisms at Ultrahigh Pressures and Strain Rates: *James M. McNaney*[1]; Bruce A. Remington[1]; Steven M. Pollaine[1]; K. Thomas Lorenz[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., L-370, 7000 East Ave., Livermore, CA 94550-9234 USA

The use of high-energy lasers to produce shock loading conditions in solid materials is a relatively recent development. Sophisticated 2-D hydrodynamics simulations are used to characterize the transmission of the pressure wave through the material. Experimental verification of the predictions of these calculations is generally limited to VISAR measurements of the back side of thin foils. This work examines the use of microstructure in laser shock-loaded and recovered samples to detect deformation mechanisms and validate predictions based on hydrodynamic simulations of the laser-based drive. In order to test the accuracy of the complex hydrodynamic simulations, direct laser illumination was used to singly and/or doubly shock samples of an aluminum alloy. The material was recovered and subsequently examined metallographically to determine the extent of the ablation crater and the location of melted and refrozen material. Regions of material having been melted and refrozen were observed to have a small grain size (< 2 μm) as compared to that of the bulk material (< 30-40 μm). More detailed examinations, using transmission electron microscopy and/or orientation imaging microscopy were used to determine the presence of coarse slip bands and/or shear bands. Comparisons to results from similar material loaded by a high explosive drive will be made. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

11:30 AM
Laser Induced Shock Compression of Copper: Orientation and Pressure Decay Effects: *Matthew S. Schneider*[1]; Marc A. Meyers[2]; Fabienne Gregori[4]; Bimal Kad[1]; Daniel H. Kalantar[3]; Bruce Remington[3]; [1]University of California-San Diego, Matl. Sci. & Eng., 9500 Gilman Dr., 0418, La Jolla, CA 92093 USA; [2]University of California-San Diego, Dept. of Mech. & Aeros. Eng., 9500 Gilman, 0411, La Jolla, CA 92093 USA; [3]Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; [4]University of Paris 13, Villetaneuse France

Copper monocrystals with [001] and [134] orientations were subjected to ultra-short shock pulses (~10 ns duration) induced by a laser. The deformation structure was significantly dependent on the crystallographic orientation, and distance from laser-impacted surface. The threshold stress for twinning in the [001] was 45 GPa compared with 55 GPa for [134]. Dislocation densities were also different for the two orientations. These results are rationalized in terms of a constitutive description of the slip twinning transition. The higher work hardening rate of the [001] orientation is responsible for increasing the shear stress in the specimens at the imposed strains, to a higher level than in the [134] orientation; this higher stress predisposes the onset of twinning in this orientation assuming both mechanisms are competitive. The orientation dependence of the twinning stress is much lower and expressed by the Schmid factors.

11:45 AM
Orientational Dependence of Shock-Induced Plasticity and Chemistry in Diamond: *Sergey V. Zybin*[1]; Mark Elert[1]; Carter T. White[2]; [1]US Naval Academy, Chem. Dept., Annapolis, MD 21402-5026 USA; [2]Naval Research Laboratory, Code 6189, 4555 Overlook Ave. SW, Washington, DC 20375 USA

Molecular dynamics simulations employing an empirical bond order potential are used to study the orientational dependence of the elastic-plastic shock wave structure and shock-induced chemistry in diamond. The mechanism of shock-induced plasticity within this model is found to depend crucially on the direction of shock wave propagation. For <110> oriented shock waves at piston velocities Up < 4.0 km/s a twinning deformation develops along the <112> direction while in <111> oriented shock wave at Up < 5.5 km/s the twinning is accompanied by considerable shearing in the lateral <110> direction, which favors formation of sp^2 hybridized bonds. However, for higher shock velocities the situation is changed. In the <110> shock waves at piston velocities > 6.0 km/s shear deformation now develops in the transverse <110> direction favoring the formation of layered carbon structures with a considerable number of sp^2 bonds while shock waves in the <111> direction show formation of diamond micrograins separated by amorphous carbon inclusions.

Friction Stir Welding and Processing II: Friction Stir Joining: Process Models

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Thomas J. Lienert, University of South Carolina, Mechanical Engineering Department, Columbia, SC 29208 USA; Rajiv S. Mishra, University of Missouri-Rolla, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Tuesday AM — Room: 7B
March 4, 2003 — Location: San Diego Convention Center

Session Chair: Kumar V. Jata, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA

8:30 AM
Material Flow and Temperature Distribution During Friction Stir Welding of 7050 Aluminum Alloy: *Blair London*[1]; Murray Mahoney[2]; [1]Cal Poly State University, Matls. Eng. Dept., San Luis Obispo, CA 93407 USA; [2]Rockwell Scientific Company, Dept. 202, 1049 Camino dos Rios, PO Box 1085, Thousand Oaks, CA 91358-0085 USA

We used Al-30 vol.% SiC markers machined into one of the faying surfaces of the weld butt joint to track material flow during FSW. Though detailed metallographic studies, we were able to document the flow of material at the centerline, advancing, and retreating sides of the welds at various depths within the plate. We found significant differences in how much the material is strained and distributed in the FSW process with location in the weld. The experimental results are compared with those predicted by our computational model. Other marker materials in friction stir welded metals other than aluminum will also be discussed. The temperatures in the weld zones were measured with small-diameter thermocouples at various distances from the FSW nugget. In our design of experiments, we investigated the effects of weld parameters, location (advancing-retreating sides), and depth within the plate on the temperatures reached in the welding process. These results are compared with our modelís predictions.

8:55 AM
2-Dimensional CFD Modelling of Flow Round Profiled FSW Tooling: *Paul Andrew Colegrove*[1]; Hugh R. Shercliff[1]; [1]The University of Cambridge, Dept. of Eng., Trumpington St., Cambridge CB2 1PZ UK

This paper describes the application of the Computational Fluid Dynamics (CFD) code, FLUENT, to modelling of the metal flow in FSW. The primary goal is to assist in the development of new tools for FSW, though the models also improve the understanding of the deformation and heat generation mechanisms in FSW, and their relationship to the final weld microstructure. Friction stir models to date have focussed on thermal field prediction, with some studies leading to prediction of microstructural evolution, properties and residual stress. A much greater modelling challenge is the prediction and validation of the 3D flow field round the tool. Recent flow models have given some indication of the metal flow for idealised or conventional threaded tools, and some authors have speculated on the likely cause of weld defects. This paper describes progress towards developing a quantitative method of comparing the flow round different practical tool shapes. 2-D flow models have been used to compare four different tool profiles, and to examine the effect of changing rotation and welding speed on the process. To enable this comparison a novel ìslipî model was developed, in which the interface conditions were governed by the local shear stresses. This revealed significant differences in behaviour compared to the common assumption of sticking. The 2D model has demonstrated the viability of the approach for investigating the flow round practical tool shapes, and gives confidence that developing the more computer-intensive 3D models will be justified. The model also gives a first order visualisation of the flow round the central region of the probe in thick welds (i.e. well away from the influence of the shoulder or the probe base).

9:20 AM
Tool Wear and Shape Optimization in the Friction-Stir Welding of Aluminum Metal-Matrix Composites: *R. A. Prado*[1]; L. E. Murr[1]; D. J. Shindo[1]; A. R. Rivera[1]; J. C. McClure[1]; [1]University of Texas at El Paso, Dept. of Metallurgl. & Matls. Eng., 500 W. University Ave., El Paso, TX 79968 USA

In the friction-stir welding (FSW) of most aluminum alloys there is no perceptible wear of the pin tool even after traversing tens of meters of material. In the FSW of aluminum metal-matrix composites (MMCís) such as Al 6061 + 20% Al_2O_3 and Al-A339 + 20% SiC, tool wear has been shown to depend upon rotation and traverse speeds initially; with optimum wear occurring at 1000 rpm. However, as the traverse or actual weld speed is increased (from 1 mm/s to 9 mm/s) tool wear and tool wear rate decline. At 6 mm/s and 9 mm/s weld speeds (and constant rotation speed of 1000 rpm) the MMC FSW wear produces optimized tool shapes after sufficient traverse distances. Tool wear effectively ceases when these optimized shapes are achieved. Consequently, these shapes may provide a basis for designing optimized tools for other FSW systems as well.

9:45 AM
Thermal and Microstructure Modelling in Thick Plate 7075 Friction Stir Welds: Takehiro Hyoe[1]; *Paul Andrew Colegrove*[1]; Hugh R. Shercliff[1]; [1]The University of Cambridge, Dept. of Eng., Trumpington St., Cambridge CB2 1PZ UK

This paper investigates the evolution of precipitation hardening in thick plate friction stir welded aluminium alloy 7075-T7351. As part of a study into metal flow in FSW, thermal histories were produced as a function of position in the weld cross-section using the numerical fluid dynamics code, FLUENT. These were validated by thermocouple observation, and enabled detailed temperature prediction for the different regions in the weld, such as the nugget, thermo- mechanically affected zone (TMAZ) and heat-affected zone (HAZ). Isothermal softening experiments were conducted to capture the temperature-dependent fall in strength, which is due to a combination of precipitate dissolution and coarsening. Subsequent natural ageing was used to determine the extent to which strength could be recovered after welding. The data were fitted to a dissolution model developed for welding of ternary Al-Zn-Mg alloys. In spite of the added complexity of the phases in Cu-bearing alloys such as 7075, the model provides a useful tool for predicting the extent of softening and re-ageing due to the thermal cycles seen in 7075 friction stir welds. The model is applied to predict: (a) the extent of softening ahead of the probe, to investigate whether full dissolution has occurred prior to deformation; (b) the hardness profile across the TMAZ and HAZ. A number of possible complications in the precipitation behaviour, which are not incorporated in the current model, are discussed. It is likely in 7075 that there is an interaction between the dislocation structures and precipitation in the weld nugget and the TMAZ. This may lead to accelerated coarse precipitation (due to the alloyís quench sensitivity) during cooling of the weld, with a consequent decline in properties.

10:10 AM Break

10:20 AM
Advances in Laser Assisted Friction Stir Welding: *G. Kohn*[1]; Y. Shneor[1]; A. Munitz[2]; [1]Rotem Industries, Ltd., Temed Industl. Park, PO Box 9046, Beer-Sheva 84190 Israel; [2]Nuclear Research Center, PO Box 9001, Beer-Sheva 84190 Israel

Among the drawbacks of friction stir welding (FSW) is the need to use powerful fixtures to clamp the workpiece to the welding table, the high force needed to move the welding tool forwards and the relative high wear rate of the welding tool. To overcome these drawbacks a new laser assisted friction stir welding (LAFSW) system has been developed. The system is a combination of a conventional machining center and a Nd: YAG laser-system that are used together during the welding process. Laser power is used to pre-heat a volume of the workpiece ahead of the probe. The workpiece is then joined in the same way as in the conventional FSW process. The high temperature ahead of the rotating tool softens the workpiece and enables joining it without the need of strong clamping fixtures, reduces markedly the force needed to move the welding tool forwards and hence reduces its wear. This paper describes results that have been accumulated since the publication of the first paper. Comparison between the forces needed in FSW and LAFSW are presented for various geometries and materials and possible application of LAFSW in the aviation industry is suggested.

10:45 AM
A New Coupled Heat Transfer Model for Friction Stir Welding: *Mingde Song*[1]; Rado Kovacevic[1]; [1]Southern Methodist University, Dept. of Mechl. Eng., Rsrch. Ctr. of Adv. Mfg., 1500 International Pkwy., Ste. 100, Richardson, TX 75081 USA

In this paper, a new heat transfer model is established for modeling the three-dimensional transient heat-transfer in friction stir welding (FSW). The heat transfer between the tool and the workpiece are coupled at the interface. The heat flux input from the tool shoulder is calculated by the friction work; the plastic deformation heat near the pin is also modeled. The material latent heat is included in the model. An explicit central differential scheme is employed in solving the control equations. The calculation results are compared with the FSW experimental results, the two results are in good agreement. The welds at different welding control conditions are discussed.

11:10 AM
Thermomechanical Processes in Friction Stir Welding: *J. A. Schneider*[1]; A. C. Nunes[2]; [1]Mississippi State University, Dept. of Mechl. Eng., PO Box ME, 210 Carpenter Eng. Bldg., Mississippi State, MS 39762 USA; [2]NASA-MSFC, ED 33, Matls. Process & Mfg. Dept., Huntsville, AL 35812 USA

In friction stir welding (FSW), a rotating pin-tool stirs the edges of a weld seam together. The flow trajectories around the tool are illustrated by a thin (0.0025 in. diameter) tungsten wire marker and explained by a decomposition of the flow field into simpler elements. Two kinds of trajectories, each subjecting the weld metal to a distinct thermomechanical process and imparting a distinct microstructure, can be differentiated. Microstructures observed in a FSW cross-section in an aluminum 2195 alloy are related to their respective strain-temperature histories along their respective flow trajectories.

11:35 AM
Modeling Friction Stir Welding Using an Inverse Problem Approach: Sam G. Lambrakos[1]; *Richard W. Fonda*[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6324, 4555 Overlook Ave. SW, Washington, DC 20375 USA

In contrast to the direct problem approach, which employs assumed values of material properties in coupled equations of energy, momentum, and mass transport to calculate the temperature histories occurring during welding processes, the inverse problem approach uses information about the actual temperature(s) achieved during welding to constrain calculations of the weld thermal field. This talk will discuss the application of this inverse problem approach to friction stir welding and its relationship to the direct problem approach. Finally, the model predictions will be compared to experimental data from thermocouple measurements, thermal simulations of the various regions of the friction stir welds, microhardness measurements, and microstructural characterizations in actual friction stir welds.

General Abstracts: Smelting, Melting, and Effluent Control

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Tuesday AM — Room: 5A
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Robert L. Stephens, TeckCominco Metals Ltd., Trail, British Columbia V1R 4L8 Canada; Adrian Deneys, Praxair Inc., Tarrytown, NY 10591-6717 USA

8:30 AM
Improved Maintenance Techniques for INCOís Two Flash Smelting Furnaces: *Randy E. Lawson*[1]; Darryl D. Cooke[2]; [1]INCO, Ltd., Smelter Maintenance, Smelter Offices, Copper Cliff, Ontario P0M 1N0 Canada; [2]INCO, Ltd., Smelter Techl. Services, Copper Cliff, Ontario P0M 1N0 Canada

INCOís Copper Cliff Smelter houses two Flash Smelting Furnaces to process 100% of the copper-nickel concentrate produced from the Sudbury area mines. On-line time of these furnaces is key to maintaining throughput to the downstream processes. Increased furnace throughput and variations in slag temperature have resulted in accelerated wear of the furnace refractory. In order to minimize the effect of furnace downtime on production, an accelerated cool-down/repair/heat-up schedule was fabricated to fit within the schedule for repairs required to INCOís oxygen plant. The engineering of a unique system for supporting the furnace sidewalls allowed repairs to be completed on the high wear areas of the furnace including the matte tapholes. This paper also discusses the various techniques used for the furnace roof repairs, tie-ins to existing brickwork, co-ordination of mechanical and refractory contractors, replacement of a leaking uptake transition block and the achievements in the cool-down and heat-up schedules.

8:50 AM
Observations on Blister Copper Spitting: *Melissa L. Trapani*[1]; Ross K. Andrews[1]; Dennis Montgomerie[2]; Andrew K. Kyllo[1]; [1]The University of Melbourne, G. K. Williams Ctr. for Extractive Metall., Dept. of Cheml. Eng., Melbourne, Victoria 3010 Australia; [2]WMC Olympic Dam, PO Box 150, Roxby Downs, S. Australia 5725 Australia

ìBlister spittingî is a flow irregularity where the molten blister copper stream - or part thereof - sprays from the taphole exit rather than following a regular flow profile to the collection box. As well as being a general ìhousekeepingî issue, the unpredictable nature of the flow is a threat to operator safety. Also, the aggressive flow pattern may contribute to the mechanical wear of taphole refractory, compromising the integrity of the taphole. This paper brings together a collection of first-hand observations and reports from process personnel, and suggests some theories as to the causes of blister spitting.

9:10 AM
Instrumentation of a Production Taphole: *Melissa L. Trapani*[1]; Ross K. Andrews[1]; Dennis Montgomerie[2]; Andrew K. Kyllo[1]; Neil B. Gray[1]; [1]The University of Melbourne, G. K. Williams Ctr. for Extractive Metall., Dept. of Cheml. Eng., Melbourne, Victoria 3010 Australia; [2]WMC Olympic Dam, PO Box 150, Roxby Downs, S. Australia 5725 Australia

Many smelters have sought to improve their tapping systems, embarking on taphole development programs, which consist of a series of incremental modifications to an original taphole design. Modifying tapholes without sufficiently understanding them has led to the installation of hazardous tapholes, and in some cases dangerous and costly events have occurred. An improved taphole design methodology, developed specifically to avoid this scenario, is currently being applied to a production taphole. This paper provides: a discussion of the major challenges that come with the instrumentation of an industrial taphole, results from the plant trial, and the details of how these results are facilitating an improved taphole design.

9:30 AM Cancelled
Improved Plugging Technology for Water Jackets in FS Furnaces: *Pekka A. Taskinen*[1]; Veikko Polvi[1]; Tuija K. Suortti[1]; Ilkka V. Kojo[2]; Pekka Set‰l‰[3]; Pasi Ranne[4]; [1]Outokumpu Research, PO Box 60, Pori FIN-28101 Finland; [2]Outokumpu Technology, PO Box 862, Espoo FIN-02201 Finland; [3]Outokumpu Harjavalta Metals, Teollisuuskatu 1, Harjavalta FIN-29200 Finland; [4]Outokumpu Poricopper, Kuparitie, Pori FIN-28101 Finland

9:50 AM
Non-Destructive Inverse Determination of Refractory Wall Material Wear Configurations in Melting Funaces: *George S. Dulikravich*[1]; Thomas J. Martin[2]; [1]University of Texas at Arlington, Mech. & Aeros. Eng. Dept., Multidisciplinary Analy., Inverse Design & Optimization (MAIDO) Prog., UTA Box 19018, Arlington, TX 76019 USA; [2]Pratt & Whitney Engine Company, Turbine Discipline Eng. & Optimization Grp., 400 Main St., MS 165-16, E. Hartford, CT 06108 USA

From the existing thermocouples measurements on the outer surface of the hearth section of the furnace, it is possible to obtain both temperature and heat flux distribution on that surface. This provides for the over-specified thermal boundary conditions on this surface that is essential for our inverse algorithm, which can determine the corresponding temperature distribution on the guessed shape of the refractory/melt interface. If we insist that the wall refractory wear surface is at the melt solidus temperature, then only one possible wear surface configuration will have such isothermal condition. The correct shape of the wear surface is determined iteratively with our inverse shape determination algorithm that is based on an elastic membrane concept and Fourier series formulation. This inverse approach to inner wall shape determination is considerably faster than if utilizing an optimization algorithm thus providing for a real-time monitoring tool of wear surface configuration.

10:10 AM Break

10:20 AM
Environmental Management of Selenium: Case Studies: *Karen A. Hagelstein*[1]; [1]Times, Ltd., 1604 Leopard St., Sheridan, WY 82801 USA

This presentation will provide case studies in the environmental management of selenium from the aluminum, recycling, and mining industries. Selenium-containing waste streams, sources, and applicable USA regulations will be discussed. The toxicological and bioaccumulative properties of selenium will be outlined with respect to risk assessments and site specific criteria. Case studies will focus on monitoring, treatment, transport, and disposal practices in managing selenium wastes and emissions from primary and secondary aluminum operations. The sources of selenium such as dross, scrap, aluminum, and steel alloys containing manganese/selenium will be addressed. Secondary aluminum processing solid waste, such as baghouse dust, are subject to additional regulatory requirements if classified as hazardous by the toxicity characteristic leaching procedure (USEPA TCLP). Hazardous air pollutants (HAPs), including particulates and potentially selenium emissions, will be assessed with respect to operational variables, processed materials, treatment/control technologies, and health-based standards.

10:40 AM
Nano-Filtration of Contaminated Solutions Using Avian Keratin Protein: *Manoranjan Misra*[1]; *Piyush Kar*[1]; Ashok Raichur[2]; Carlo Licata[3]; Gautam Priyadarshan[1]; [1]University of Nevada-Reno, Mackay Sch. of Mines, Metall. & Matls. Eng., MS 388, Reno, NV 89557 USA; [2]Indian Institute of Science, Bangalore, Dept. of Metallurgl. Eng., Bangalore India; [3]MaXim Biosystems, 1015 N. Lake Ave., Ste. 313, Pasadena, CA 91104 USA

Avian Keratin Fiber is a form of animal fibrous protein and is derived from feathers of birds such as chicken and turkey and has been projected here as an ideal materials for biosorption and removal of metals from solutions. It is hydrophobic, stable over a wide pH range, and has a high surface area. It is an abundant bioresource and a cheap alternative to conventional adsorbents like activated carbon and ion-exchange resins. This material is primarily b-keratin and is nearly 100% protein in its constitution. The keratin protein fiber is an intricate network of protein fibers that are high in surface area and it consists of nano-sized pores. The nano-structure and the functional groups render the keratin fiber as an excellent nano-filter. The heavy metals that could be effectively removed included lead, copper, cadmium, zinc, chromium, iron, and mercury and precious metals like gold, platinum and palladium from process and drinking water to meet USEPA drinking water standards.

11:00 AM

Efficient Treatment of Complex Wastewaters at Umicore Precious Metals Using Biotechnology: Merijn Picavet[1]; Henk Dijkman[1]; *Johannes Boonstra*[1]; Klaas de Hoop[1]; Bert Daamen[1]; Wouter Ghyoot[2]; [1]Paques B.V., PO Box 52, Balk NL-8560AB Netherlands; [2]Umicore, Kasteelstraat 7, Olen B-2250 Belgium

Paques and Umicore have demonstrated the feasibility of biological treatment of the highly complex Hoboken wastewaters in a 1 m3/h pilot plant at Umicore Precious Metals, Hoboken, Belgium. The process consisted of the following main steps: 1. Arsenic precipitation with biogenic H2S as As2S3. 2. Biotechnological removal of nitrate combined with precipitation of dissolved metals as sulphides (MeS). 3. Biotechnological sulphate removal from the pretreated and denitrified waters using hydrogen as reductant. The sulphide produced was used for metals precipitation in steps 1 and 2. Excess sulfide was biologically oxidised and separated as solid elemental sulphur. Arsenic sulphide produced was easily separated using a tilted plate settler. Metals such as mercury, thallium, copper, zinc, lead, nickel were also removed to below discharge demands. Sulphate and nitrate were removed to low concentrations (<300 mg/l and < 5 mg/l respectively). Selenate was biologically reduced to elemental selenium to concentrations <0.2 mg/l.

11:20 AM

Microwave Heating Characteristics of Pyrite and Microwave Assisted Coal Desulphurization: *Tuncay Uslu*[1]; ‹mit Atalay[1]; [1]Middle East Technical University, Mining Eng. Dept., Ankara 06531 Turkey

In this study, microwave heating characteristics of pyrite mineral and desulphurization of coal by using magnetic separation following the microwave heating were investigated. Different size of pyrite samples were heated in microwave oven and heating characteristics were determined. It was observed that heating rates and maximum attained temperatures increased with increased microwave power level, heating time and reducing particle size. Microwave heated pyrite samples were subjected to magnetic separation and 98% of the pyrite was removed as magnetic product. Coal desulphurization by microwave heating followed by magnetic separation was also studied. Total and pyritic sulphur of coal sample were reduced by 32.82% and 37.46% respectively. With the addition of 7.5% magnetite into coal which is an excellent microwave absorber, the heating characteristics of coal sample was improved and total sulphur and pyritic sulphur content of coal sample were reduced 52.05% and 58.20% respectively by magnetic separation following the microwave heating.

Global Development of Copper and Gold Deposits - I

Sponsored by: Extraction & Processing Division, EPD-Process Mineralogy Committee, EPD-Precious Metals Committee
Program Organizers: Tzong T. Chen, CANMET, Ottawa, Ontario K1A 0G1 Canada; Corby G. Anderson, Montana Tech of the University of Montana, Center for Advanced Mineral & Metallurgical Processing, Butte, MT 59701-8997 USA; Steven L. Chryssoulis, Amtel, London, Ontario N6G 4X8 Canada

Tuesday AM — Room: 1B
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Corby G. Anderson, Montana Tech of the University of Montana, Ctr. for Adv. Minl. & Metallurgl. Procg., Butte, MT 59701-8997 USA; T. T. Chen, CANMET, Ottawa, Ontario K1A 0G1 Canada

8:30 AM

Remediating an Integrated Copper Complex-Problems in the Present and Lessons for the Future: *Sandra M. Stash*[1]; Robert Ray Beebe[2]; Raymond E. Krauss[3]; [1]ARCO Environmental Remediation, 306 E. Park Ave., Anaconda, MT 59711 USA; [2]PO Box 4135, Butte, MT 59702-4135 USA; [3]6969 St. Helena Rd., Santa Rosa, CA 95404 USA

An integrated copper complex includes mining, milling and smelting facilities which are usually at or near a common site, and sized to complement one another in capacity. There may also be an electrolytic refinery serving the complex, but not always totally dedicated to its production. Mines and mills without co-owned smelters are not usually considered integrated. The first marketable product of an integrated complex is blister, or in some cases electrolytically refined copper. The nature of integrated operation brings together all of the environmental problems associated with copper production, but currently operating and newly planned facilities are required to anticipate these problems and meet stringent regulations. Older complexes, however, may have legacies of 50 or more years of operation unencumbered by environmental strictures: nevertheless, they must be remediated. Using examples from closures and remediations of the Anaconda/ARCO facilities in Montana, the authors review some of the problems encountered over more than a decade, and the means for their solutions, bearing in mind that not all of the problems and solutions were technical. Based on real-world experience, some potential pitfalls and lessons for the future are identified.

9:00 AM

Behaviour of Gold During the Electrorefining of Copper: *T. T. Chen*[1]; J. E. Dutrizac[1]; [1]CANMET, 555 Booth St., Ottawa, Ontario K1A 0G1 Canada

Copper sulphide ores and secondary materials contain trace amounts of Au. After smelting, the Au is collected in the copper anodes. In the copper anodes, the gold occurs in solid solution in the copper crystals. During electrorefining, the copper dissolves and the associated gold is released. Most of the gold reports as tiny metallic Au particles in the anode slimes. The Au shows a strong affinity for the selenide phase, and some of the metallic Au appears to have nucleated on the selenide particles. Some gold also appears to dissolve in the sulphate electrolyte, possibly because of the presence of chloride and thiourea, and subsequently to become incorporated as a minor constituent of a complex oxidate phase. Gold also is present both in solid solution in the selenide phase and as a Ag-Au-Cu selenide phase. Decopperizing of the anode slimes in an autoclave concentrates the gold as metallic Au, a Ag-Au selenide phase (Ag3AuSe2) and a Ag-AuSe-Te rich phase; the Ag-Au selenide phase appears to form during decopperizing. Also, the gold seems to be enriched in the original Ag selenide phase.

9:30 AM

The Industrial Nitrogen Species Catalyzed Hydrometallurgical Recovery of Copper and Precious Metals from Chalcopyrite: *Corby G. Anderson*[1]; [1]Montana Tech, Ctr. for Adv. Minl. & Metallurgl. Procg., 1300 W. Park St., Butte, MT 59701-8997 USA

Hydrometallurgical pressure oxidation of copper ores and concentrates is currently an active area of technological development. Of these, an overlooked but, ironically the first industrially proven methodology, utilized nitrogen species catalyzation in the oxidizing pressure leach system to produce copper via SX/EW. In this paper, the history of the system and its application to copper concentrates and ores will be outlined. In particular, the methodology for effective recovery of precious metals from chalcopyrite concentrates will be discussed. Finally, the perceived economics of chalcopyrite concentrate treatment with this unique industrially proven process will be delineated.

10:00 AM

On the Floatability of Gold Grains: *Stephen Chryssoulis*[1]; Stamen Dimov[1]; Daniela Venter[1]; [1]AMTEL, 100 Collip Cir., London, Ontario N6G 4X8 Canada

Accepted wisdom is that gold is naturally floatable in industrial systems, due to the adsorption of hydrocarbons which render hydrophobicity to gold grain surfaces. Such hydrophobicity is usually enhanced by the addition of flotation collectors such as xanthates, mono- and dithiophosphates and mercaptobenzothiolates. The mechanism invoked in enhancing the hydrophobicity of gold is similar to that of pyrite, whereby collector ions are oxidized at the gold surfaces to form the neutral dimers. The non-polar dimers are oily substances. Impurities in the gold (usually Ag and Cu) have been thought to form hydrophilic surface phases. Microbeam surface analysis of floated and rejected free gold grains from a number of commercial plants and pilot plant testwork has shown silver (the most common impurity in gold) and sulphur invariably in greater concentration on floated gold grains, implying that they assisted gold flotation. Further, collector loading measurements on individual gold grains from concentrates of sequential rougher flotation testwork revealed first progressively lower concentrations of the collector monomer on gold from each follow-up concentrate and secondly a direct proportionality between the surface concentration of silver and the collector monomer. Based on the available data there is no doubt that silver activitates and that collector monomers play a significant, previously not recognized, role in gold flotation.

10:30 AM
Microwave-Assisted Chalcocite Leaching in $CuCl_2$-NaCl Media Under Oxygen Pressure: *Jiann-Yang Hwang*[1]; Shangzhao Shi[1]; Xiaodi Huang[1]; [1]Michigan Technological University, Inst. of Matls. Procg., 309 Minerals & Matls. Bldg., 1400 Townsend Dr., Houghton, MI 49931-1295 USA

A study on microwave assisted chalcocite leaching was carried out with a microwave hydrothermal reactor. The leaching media is a mixed solution Of $CUC1_2$ and NaCl. The leaching temperature, oxygen pressure, stirring speed, quantities of the minerals per unit solution volume as well as the initial concentration of cupric and chloride ions were investigated as the process parameters. The results were discussed and compared with that using conventional leaching method.

Hot Deformation of Aluminum Alloys: Constitutive Analysis, Modeling and Simulation

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA; Armand J. Beaudoin, University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; Balasubramaniam Radhakrishnan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6359 USA

Tuesday AM — Room: 6E
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Julian H. Driver, Ecole des Mines de St Etienne, SMS, St. Etienne 42023 France; Mary A. Wells, University of British Columbia, Dept. of Metals & Matls. Eng., Vancouver, BC V6T 1Z4 Canada; Rustam Kaibyshev, Institute for Metals Superplasticity Problems, Ufa 450001 Russia

8:30 AM Invited
Atomistic Modeling of Hot Deformation of Disordered Al-Ni Alloys: *S. G. Srinivasan*[1]; M. I. Baskes[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

An understanding of hot deformation in Al-Ni alloys can provide insight for many thermomechanical-processing technologies. Hot deformation processes produce complex interactions between grainboundaries and dislocations, which eventually lead to unusual defect microstructures with practical implications. We systematically study the interaction of a lattice edge dislocation with a grainboundary in disordered Al-Ni alloys, with up to 10% Ni, as a function of shear. Bicrystals with a dislocation in one of the grains were crafted with various symmetric and asymmetric tilt grainboundaries. Molecular dynamics simulations using a reliable embedded atom method potential were performed on these bicrystals at close to (2/3)rd the melting temperature. Local grain and defect structures were examined using a common neighbor analysis. This information coupled with an intimate knowledge of stress-strain behavior will provide insight into deformation processes at high temperatures.

8:55 AM Invited
Damage Evolution in Hot Deformation: Nano-Micro-Mesoscale Growth: *Amit K. Ghosh*[1]; [1]University of Michigan, Matls. Sci. & Eng., 2102 H. H. Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

Engineering metals and alloys exhibit formation of voids during large plastic deformation at elevated temperature, a matter of concern in metal forming processes. Typically such void formation occurs at and near grain boundaries, since at elevated temperature grain boundary sliding, albeit small at times, provide an accommodating mechanism for deformation. Thermomechanical processing and casting often leave fine scale damage in materials, particularly at hard second phase particles and grain boundaries. These damages can grow during subsequent deformation primarily by a strain-controlled process. It is often believed that recrystallization and homogenization treatments can heal damage and rid material of all preexisting damage. Recent findings suggest that this notion is only approximately true. ìApparently healedî voids may only represent a weak interface, which may contain nanometer scale voids. As these nanovoids provide free surfaces, they experience deviatoric stresses during deformation and can grow by dislocation accumulation at their tips. Gradual and statistical opening of these nanovoids give rise to a ìcontinuous nucleationî phenomenon. In other cases, nucleation truly requires a sufficiently high local stress generated in the vicinity of phase boundaries that are incompatible. In this talk, growth of such voids and the role of healing processes on subsequent damage evolution will be discussed.

9:20 AM Invited
Application of Mesoscale Finite Element Simulations to Study the Evolution of Cube Texture During Hot Deformation of Aluminum: *Gorti B. Sarma*[1]; B. Radhakrishnan[1]; Thomas Zacharia[2]; [1]Oak Ridge National Laboratory, Compu. Sci. & Math. Div., PO Box 2008, MS-6359, Oak Ridge, TN 37831-6359 USA; [2]Oak Ridge National Laboratory, Compg. & Computatl. Sci. Direct., PO Box 2008, MS-6232, Oak Ridge, TN 37831-6232 USA

The origin and development of cube ({001}<100>) texture during hot deformation and subsequent recrystallization of aluminum alloys remains a topic of considerable interest in materials research. The application of finite element modeling at the mesoscale to study the hot deformation of microstructures containing cube oriented grains distributed among grains with S ({123}<634>) and copper ({112}<111>) orientations is described. Discretization of each grain with a large number of elements enables the model to capture the heterogeneous deformation of individual grains. The constitutive response of the material is modeled using crystal plasticity, thereby enabling the prediction of texture evolution in the microstructure. The deformation at elevated temperatures has been modeled by including slip on the non-octahedral {110}<110> systems, in addition to the usual {111}<110> systems. Microstructures with different grain sizes have been deformed in plane strain compression. The effects of the local environment, grai n size and plastic strain on the stability of the cube texture during hot deformation are examined. Research sponsored by the Office of Basic Energy Sciences, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:45 AM
Dynamic Response of AA7075 Aluminum Alloy as a Function of Temperature: *Zhe Jin*[1]; William A. Cassada[1]; Carl Cady[2]; George (Rusty) T. Gray[2]; [1]Alcoa Technical Center, Alcoa Ctr., PA 15069 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

The dynamic behavior of a commercial 7075 aluminum alloy was studied as a function of temperature. The dynamic stress-strain response of the 7075 aluminum alloy at 2100 s-1 can be characterized into two temperature regimes: (1) athermal hardening dominates at temperatures £200∞C and (2) thermally activated recovery dominates at temperatures ≥300∞C. The largest drop in the dynamic flow stress with temperature occurs at temperatures between 200∞C and 300∞C and the smallest at temperatures above 300∞C. Guiner-Preston (GP) zones and h precipitates are the major strengthening components for the dynamic loading of the 7075 aluminum alloy. Strain instability and adiabatic heating appears to have a significant impact on the dynamic response of the 7075 aluminum alloy, particularly at the lower test temperatures.

10:05 AM
Localized Deformation in Hot Working of AA 7XXX Alloys: *Henry A. Padilla*[1]; Blythe E. Gore[2]; Armand J. Beaudoin[1]; Jonathan A. Dantzig[1]; Ian M. Robertson[2]; Hasso Weiland[3]; [1]University of Illinois at Urbana-Champaign, Mechl. & Industl. Eng., 1206 W. Green St., MC-244, Urbana, IL 61801 USA; [2]University of Illinois at Urbana-Champaign, Matls. Sci. & Eng., 1304 W. Green St., MC-246, Urbana, IL 61801 USA; [3]Alcoa Technical Center, 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

In hot working of 7xxx series aluminum alloys, the thermomechanical processing window is limited by problems such as hot shortness and by property requirements such as fatigue and fracture toughness. The mechanical response of AA 7050 and AA 7055 was first characterized using routine compression test procedures. A novel test procedure was then developed to study the onset of localization due to hot shortness. Compression samples were encased in a sheath of commercial purity aluminum. The test provides for a transition from homogeneous to non-uniform deformation, as dictated by mechanical integrity of the grain boundaries. There is a concomitant progression in the controlling mechanism for mechanical anisotropy from crystallographic texture to grain morphology. The results are interpreted through complementary studies of microstructure evolution.

10:25 AM Invited
Mesoscale Simulation of Cube Texture Evolution Following Hot Deformation of Aluminum: *Bala Radhakrishnan*[1]; Gorti Sarma[1]; [1]Oak Ridge National Laboratory, Compu. Sci. & Math. Div., Bldg. 6025, MS 6359, Oak Ridge, TN 37831-6359 USA

It is well known that cube bands that survive the hot deformation process serve as nuclei for subsequent cube texture evolution in aluminum during recrystallization. The interfacial area between cube and S orientations appears to be the key microstructural feature that controls cube nucleation. The paper presents simulations of cube texture evolution at the mesoscopic length-scale with emphasis on the influence of local microstructural environment on the growth of cube islands after hot deformation. Hot deformation substructures derived from crystal plasticity based modeling are used as input to a Monte Carlo based simulation of substructure evolution. The effect of neighboring orientations such as Copper and S on the growth of cube bands is investigated as a function of plastic strain and initial grain size. The simulation results are compared with the predictions of existing analytical models of texture evolution in polycrystalline aluminum. Research sponsored by the Office of Basic Energy sciences, US Department of Energy under contract DE-AC05-00OR22725 with UT-Batelle, LLC.

10:50 AM
A Finite Element Model for Crystal Plasticity During Large Deformation and Comparison with Experimental Measurements: Rajesh Prasanna[1]; Pankaj Trivedi[1]; *Ben Q. Li*[1]; David Field[1]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Pullman, WA 99164-2920 USA

A finite element-based macro/micro model is presented for crystal plasticity during large deformation of aluminum alloys. The model development is based on the updated Lagrangian formulation for large plastic deformation with the product decomposition of the deformation gradient tensor into elastic and plastic parts. This total formulation will ensure both the strong and weak objectivity for large plastic deformations. The macroscopic stress is linked to the microstructural behavior of crystals by tracking the motion of the active slip systems underlining the plastic deformation. Our model treatment entails a multi-surface-type stress update algorithm, which ensures the plastic deformation defined by each slip system to be on the yield surface. The numerical model is first applied to a single crystal system undergoing plastic deformation and then applied to a poly-crystal system involving a few grains. The numerical formulations and algorithm implementation are presented and numerical simulations are compared with experimental measurements.

11:10 AM
Simulation of Extrudate Surface Formation by the Use of Finite Element Method: *Xinjian Duan*[1]; Terry Sheppard[1]; Xavier Velay[1]; [1]Bournemouth University, Sch. of Design, Eng. & Compu., 12 Christchurch Rd., Bournemouth, BH1 3NA England

To have a better control in surface quality of the extruded products, it would be very helpful to know which part of the as-cast billet forms the extrudate surface. The limitations of the current experimental techniques have restricted the quantitative study on the formation of outer layer of the extrudate. In this paper, a commercial FEM code, FORGE2®, is adopted to study the material flow in both direct and indirect extrusion of aluminium alloys. The validity of the FEM simulation for the study of surface formation is first verified by comparing the simulated results with those experimental observations when a flat-faced nonlubricated die is used. Then, the FEM is used to study the influence of various forming parameters (such as punch temperature, billet/container temperature differential) and the die configuration on the formation of extrudate surface in both direct and indirect extrusion.

11:30 AM
Parametric Studies of FSW Using a 2-D, Fluid Based Simulation: *Anthony Peter Reynolds*[1]; Wei Tang[1]; Tianzhong Long[1]; [1]University of South Carolina, Dept. of Mechl. Eng., 300 Main St., Rm. A224, Columbia, SC 29208 USA

Accurate simulation of the FSW process will enable rapid development of the process in particular with regard to tool design. However, this type of simulation requires a 3-D, fully coupled thermo-mechanical, solid based model with associated high computational cost. In previous work, some of the authors have demonstrated that a 2-D model using a commercial fluid dynamics code and an appropriate user defined viscosity law can accurately capture many of the trends observed in FSW. The fully thermo-mechanically coupled simulations may be run in a few hours on a PC based system. The low computational cost for this model enables rapid assessment of the effects of various material properties and the criticality of inclusion or exclusion of some property details. In this paper, the effects of varying thermal diffusivity, viscosity, strain rate sensitivity etc. on the trends in x-axis forces, required torques, and locations and magnitudes of pressure minima and maxima are examined. The model trends are compared to experimental observations wherever possible.

11:50 AM
Modeling of Friction Stir Joining as a Metalworking Process: *William J. Arbegast*[1]; [1]South Dakota School of Mines and Technology, Adv. Matls. Procg. Ctr., 501 E. St. Joseph St., Rapid City, SD 57701 USA

A ìMechanistic FSW Modelî has been developed which describes the FSW process in terms of five conventional metal working zones: a) preheat, b) initial deformation, c) extrusion, d) forging, and e) post heat/cool down. A simple approach to flow modeling using mass balance considerations reveals a relationship between pin tool geometry, operating parameters, and flow stress of the materials being joined. Comparing flow to the extrusion of material around the pin tool provides for calculation of the width of the extrusion zone, strain rates, and x-direction forces. Coupled with empirically determined relationships between processing parameters, maximum temperature, heating and cooling rates, and, material constituitive properties from Gleeble data, an understanding of the conditions necessary to produce an ìoptimumî weld emerge. Examples of this analysis approach to FSW optimization for 7050 and 6061 aluminum with comparison to experimental observations are presented.

Increasing Energy Efficiency in Aluminum

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA

Tuesday AM — Room: 5B
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Subodh K. Das, Secat Inc., Coldstream Rsrch. Campus, Lexington, KY 40511 USA; Sara Dillich, US Department of Energy, Office of Industl. Tech., Washington, DC 20585-0121 USA

8:30 AM
Aluminum Research and Development: *Thomas Robinson*[1]; [1]US Department of Energy, Aluminum Tech., Industl. Techs. Prog., Washington, DC USA

The Department of Energy - Industrial Technologies Program (DOE-OIT) is partnering with more than seventy organizations on over thirty research and development projects that reduce energy consumption and address research priorities identified in the Aluminum Industry Technology Roadmap. DOE-OIT will present an overview of its Aluminum R&D portfolio, covering technical progress, expected benefits, demonstration status, and market impacts. DOE-OITís focus is energy reduction and it uses technology roadmaps to ensure that its R&D programs address the industryís energy needs. DOE-OIT recently participated in the development of an Alumina Technology Roadmap and will publish a new Aluminum Industry Technology Roadmap by the end of 2002 that will reexamine the technology needs outlined in the 1997 Roadmap and identify new needs based on changes to the industry. DOE-OIT will also present how roadmaps influence R&D priorities.

8:45 AM
Updating the Aluminum Industry Technology Roadmap: *Richard Love*[1]; Mike Skillingberg[2]; Thomas Robinson[3]; Ross Brindle[4]; [1]Century Aluminum, Ravenswood, WV USA; [2]The Aluminum Association Inc., Tech., Washington, DC USA; [3]US Department of Energy, Aluminum Tech., Industl. Techs. Prog., Washington DC USA; [4]Energetics, Inc., Columbia, MD USA

In 1997, under the leadership of The Aluminum Association Inc. and the U.S. Department of Energy, the aluminum industry developed the Aluminum Industry Technology Roadmap, a comprehensive, long-term technology agenda for the entire industry. The Roadmap outlined quantitative performance targets, technical barriers, and research and development needed to achieve industry goals for increasing productivity, reducing costs, expanding markets, saving energy, improving worker health and safety, and minimizing environmental impact. Over the past five years, the Roadmap and its five companion ìsub-roadmapsî have stimulated over $100 million in cost-shared R&D projects with over 75 different organizations, including aluminum companies, suppliers, national laboratories, universities, and other research organizations. In November 2001, the aluminum industry published a new vision of its future. This vision, Aluminum Industry Vision: Sustainable Solutions for a Dynamic World outlines the new challenges that have emerged over the past five years and presents a bold vision of the industryís future, including a new set of industry goals. To ensure technology development remains aligned with the industryís vision, 50 representatives of aluminum companies, vendor companies, universities, national laboratories, and other researchers gathered in Pittsburgh, Pennsylvania in September 2002 to begin the process of updating the industryís Roadmap. At the workshop, participants identified technical barriers and R&D priorities in four areas: primary production; melting, solidification, and recycling; fabrication; and finished products. The Aluminum Associationís Technical Advisory Committee has taken the lead responsibility in preparing and implementing this newly revised Roadmap.

9:15 AM

Energy Efficiency Improvement Opportunities in the Aluminum Industry: *Arvind Thekdi*[1]; Sara Dillich[2]; [1]E3M, Inc., 15216 Gravenstein Way, N. Potomac, MD 20878 USA; [2]Department of Energy, Efficiency & Renewable Energy, 1000 Independence Ave., Washington, DC 20585-0121 USA

The United States is the largest single producer of primary aluminum. Total energy used by the aluminum industry (year 1995) in USA is approximately 700 trillion (10^12) Btu/year which represents approximately 2.5% of the total industrial energy use for the United States. It is one of the most energy intensive industry where energy cost represent approximately 30% of the total cost of production in USA. Although substantial improvements have been made in the energy consumption per unit of production by the industry during the last two decades, the industry can still do a better job to make further improvements. The aluminum industry plants use energy for primary metal production, primary and secondary aluminum melting and subsequent fabrication processes. The energy supplied to primary metal production is mostly in the form of electricity while the secondary aluminum melting and fabrication processes use fossil fuel (natural gas) for furnaces and electricity for rolling operations, compressors, fans and blowers, pumps, etc. Each of the energy using equipment offer an opportunity to improve their energy efficiency that can lead to substantial improvements in overall energy efficiency. This paper describes major areas of energy use and current status of energy use patterns for the primary and secondary aluminum industry and the available tools for evaluation of the current energy use and energy efficiency improvements. It outlines results of plant wide energy assessments carried out by the aluminum companies in cooperation with The Department of Energy. The paper highlights opportunities for efficiency improvement supplemented by examples of specific actions taken by the industry for energy efficiency improvement.

9:45 AM Break

10:00 AM

Improving Energy Efficiency in Secondary Aluminum Melting: *Paul E. King*[1]; Tianxiang Li[2]; Mohamed Hassan[2]; Kozo Saito[2]; Srinath Viswanathan[3]; Qingyou Han[3]; Shen-Lin Chang[4]; David Hoecke[5]; [1]US Department of Energy, Albany Rsrch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA; [2]University of Kentucky, Dept. of Mechl. Eng., Lexington, KY 40506 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Bethel Valley Rd., Oak Ridge, TN 37831-6083 USA; [4]Argonne National Laboratory, Analysis, Simulation & Modlg., 9700 S. Cass Ave., Argonne, IL 60439-4815 USA; [5]Enercon Systems, Inc., 300 Huron St., Elyria, OH 44035 USA

The objectives of ìImproving Energy Efficiency in Aluminum Meltingî project is to develop technology or to indicate operational variations that will improve the energy efficiency in aluminum melting by 25%, reduce emissions of GHG and NOx, and increase overall productivity of the aluminum industry. Estimates show that the transfer of technologies developed during this project has the potential of saving $57 million dollars/year in natural gas fuel costs (~13 trillion Btu/year) when fully implemented by the year 2015. The project utilizes a multi-company, multi-laboratory consortium to study the efficiency of the aluminum industry today and to perform the necessary research to overcome the obstacles identified as holding the efficiency of the melting technologies below a pre-defined minimum. The research team consists of three national laboratories, Secat, Inc., UK-CAT, nine participating aluminum companies and 3 industrial partners representing combustion and metal pumping/stirring technologies. This group is exceptionally strong in the aluminum melting arena with several hundred years of experience in aluminum melting technologies. The research has been divided into 4 separate but intimately coupled parts which include a) computational modeling of the combustion space for each of the furnace types, b) measurement and analysis of current steady state melting practices utilized by the member companies, c) synthesis of the measurements and the combustion space modeling into a detailed furnace model and d) experimentation. Substantial effort has gone into each of these categories with some of the general results presented in this report.

10:30 AM

Reduction of Oxidative Melt Loss: *John N. Hryn*[1]; [1]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA

This presentation will describe the efforts of a collaborative, multidisciplinary project involving Secat, Inc., seven of its member aluminum companies, the Center for Aluminum Technology at the University of Kentucky, Argonne National Laboratory, Oak Ridge National Laboratory, and Albany Research Center. The project, jointly funded by DOE/OIT Aluminum Industry of the Future program and Secat, Inc., aims to reduce melt loss during aluminum melting by 50%. To that end, the project objectives are to (1) develop an understanding of the key factors that contribute to the oxidation of molten aluminum, (2) determine the effects of major variables on the formation of dross during heating, melting, and holding of the melt at temperature, and (3) develop and evaluate technology that will reduce melt loss. Techniques used to investigate the in-situ oxidation of the molten metal surface included X-ray diffraction, X-ray fluorescence, and X-ray photoelectron spectroscopy. Microstructural analyses of industrial and lab-grown dross were also performed. Solid-state NMR was used to investigate spinel formation in dross. A small reverberatory furnace was constructed for melt trials.

11:00 AM

Modeling and Optimization of Direct Chill Casting for Reducing Ingot Cracking: *Srinath Viswanathan*[1]; Qingyou Han[1]; Adrian S. Sabau[1]; Lee Davis[2]; John A. Clark[3]; Kazunori Kuwana[4]; Mohamed I. Hassan[4]; John Hryn[5]; Greg Krumdick[5]; [1]Oak Ridge National Laboratory, Metals & Ceram., Bldg. 4508, MS 6083, Oak Ridge, TN 37831-6083 USA; [2]Wagstaff, Inc., 3910 N. Flora Rd., Spokane, WA 99216-1720 USA; [3]Albany Research Center, 1450 Queen Ave. SW, Albany, OR 97321 USA; [4]University of Kentucky, Mechl. Eng. Dept., 513 CRMS Bldg., Lexington, KY 40506-0108 USA; [5]Argonne National Laboratory, 9700 S. Cass Ave., Bldg. 362, Argonne, IL 60439-4815 USA

The goal of the project is to assist the aluminum industry to reduce the incidence of cracks from a current level of 5% down to 2%. This is important for improving product quality and consistency as well as saving resources and energy, since considerable amounts of cast metal could be saved by eliminating ingot cracking, by eliminating butt sawing. The project is a collaboration of industry, university, and national laboratory personnel through Secat, a consortium of aluminum companies. It will focus on the development of a detailed model of thermal conditions, solidification, microstructural evolution, and stress development during the initial transient in DC casting. In addition, it will develop criteria for the prediction of crack formation based on a fundamental understanding of the interaction of the as-cast microstructure, the local stress, and solidification conditions. The models developed in the project will be implemented in a commercial casting code so that they will be accessible to industry and be amenable to refinement in the future. This paper provides an overview of developments to date on experimental measurements of heat transfer at the ingot surface, inverse calculation of heat transfer coefficients, characterization of the solidification microstructure, computer models of the DC casting process for predicting the temperature and stress fields, and the determination of material properties in the mushy zone.

11:30 AM

DOE/OIT PHAST Program Application in the Aluminum Industry: *Frank L. Beichner*[1]; [1]Bloom Engineering Company, 5460 Horning Rd., Pittsburgh, PA 15236 USA

Increasing utility prices has placed an increased emphasis on reducing energy usage at many major industrial facilities. The U.S. Department of Energy (DOE) with cooperation from Industrial Heating Equipment Association (IHEA) and the major industries has developed the Process Heating Assessment and Survey Tool (PHAST) to assist in assessing and determining the major energy users for a facility, facilitating management in targeting where resources should be expended. This paper will discuss the results obtained by using the PHAST tool by Bloom Engineering Company at the Alcoa facility in Tennessee to provide an assessment of the plants energy usage. Particular emphasis will be placed on the usefulness of the information gathered and the assistance gained in determining both their overall plant efficiency and the equipment where emphasis for energy reduction should be concentrated. Also, the programs tools for assessing current designs against possible improvements will be shown.

11:45 AM
Retrofitting Regenerative Burners on Aluminum Melting Furnaces that Utilize Salt Fluxing: *Jens H. Hebestreit*[1]; *Marc D. Wolitz*[2]; [1]Bloom Engineering, Eng., Horning Rd. 5460, Pittsburgh, PA 15236-2822 USA; [2]Owens Corning Metal Systems, Eng., 1891 Reymet Rd., Richmond, VA 23237 USA

Rising fuel prices have placed an increased emphasis on reducing the cost to melt aluminum. Regenerative burner technology has been a fuel savings option for years in the aluminum industry, but maintenance, especially when using salt fluxing, has deterred many Casthouse managers from installing these systems. This paper will discuss a side well melting furnace retrofit from a cold air system to a regenerative system. The decision process detailing the conversion to regenerative rather than other technologies such as oxy/fuel is due to the production increases obtained and fuel savings achieved with the regenerative system. Also, the salt fluxing effect on media plugging and the steps taken to minimize this effect on production and maintenance will be addressed.

International Symposium on Gamma Titanium Aluminides: Fatigue and Fracture

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee
Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

Tuesday AM Room: 6F
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Robert O. Ritchie, University of California, Matls. Sci. & Eng., Berkeley, CA 94720-1760 USA; Paul Bowen, University of Birmingham, Metall. & Matls., Birmingham B15 2TT UK

8:30 AM Invited
The Influence of Microstructure on Crack Initiation & Early Growth in Gamma TiAl Alloys: *Andrew H. Rosenberger*[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLMN, 2230 Tenth St., WPAFB, OH 45433-7817 USA

The incorporation of gamma titanium aluminide alloys as rotating components in gas turbine engines will require the application of a damage tolerant design philosophy. Since gamma alloys exhibit a steep crack growth rate curve and relatively low fracture toughness design methodology will likely require damage tolerance through an assurance that a crack at a defected location will not grow to failure. In this case the mechanisms of crack initiation and early growth are critical. This study examined the mechanisms of fatigue crack initiation in lamellar gamma TiAl alloys with various microstructural scales. Crack initiation was detected using a high-resolution direct current potential difference system capable of detecting small cracks (>300 mm) in a moderately large sample. The role of microstructure was found to play a dominant role in the cracking. Significant differences in crack initiation under cyclic and monotonic loading were realized.

9:00 AM
Physics-Based Model to Predict the Fatigue Response of Gamma TiAl: *Robert G. Tryon*[1]; Animesh Dey[1]; [1]VEXTEC, 116 Wilson Pike, Ste. 230, Brentwood, TN 37027 USA

A physics-based modeling technique incorporating Monte Carlo simulation is used to predict the statistical behavior of fatigue in fully lamellar gamma TiAl. The simulation relates the random nature of the microstructure to the fatigue response of smooth round bar specimens. Microstructural characteristics such as colony size, available slip systems and critical local shear stresses are considered. The results of the research effort showed that models addressing the microstructural mechanisms that govern fatigue damage accumulation could be combined with the concept of mesomechanics to properly account for the characteristic sizes and multiple phases of fatigue. The capability developed can be used to virtually test large numbers of specimens for which actual laboratory testing would be cost prohibitive.

9:20 AM
Fatigue Properties of a Ti-48Al-2Cr-2Nb Alloy Produced by Casting and Powder Metallurgy: *Gilbert HÈnaff*[1]; Anne-Lise Gloanec[1]; Marjolaine Grange[2]; Philippe Belaygue[3]; [1]ENSMA, LMPM, 1 Ave. Clement Ader, BP 40109, Futuroscope Chasseneuil 86961 France; [2]Snecma Moteurs, Matls. & Proc. Dept., Moissy Cramayel F-77550 France; [3]Turbomeca, Bordes F-64511 France

This paper deals with an on-going study on the fatigue properties of a quaternary Ti-48Al-2Cr-2Nb alloy produced by casting (nearly-fully lamellar microstructure) and by powder metallurgy (duplex microstructure with a high fraction of gamma grains). Low cycle fatigue tests at room temperature reveal a cyclic strain hardening of the cast material which is strongly dependent on the applied strain amplitude. In particular, a pronounced hardening was observed at high strain amplitude, associated with extensive twinning, although the stabilization of the stress amplitude at lower strain amplitude can lead to the formation of a classical vein-like structure. Such a pronounced hardening was not observed in the powder metallurgy alloy. Besides the near-threshold fatigue crack growth behavior is not strongly dependent on microstructure. In the high crack growth rate regime however, the cast alloy exhibits a superior resistance.

9:40 AM Invited
Fracture of Lamellar TiAl: The Role of Lamellae Misorientation Across Colony Boundaries: *Sharvan Kumar*[1]; Ping Wang[1]; Nitin Bhate[2]; Kwai S. Chan[3]; [1]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA; [2]General Electric Global Research Center, Niskayuna, NY 12309 USA; [3]Southwest Research Institute, San Antonio, TX 78238 USA

Results of in-situ fracture experiments in the SEM of compact tension specimens of a binary TiAl alloy with a lamellar microstructure suggested that colony boundaries can provide crack growth resistance. However, the presence of sub-surface colonies in the specimen precluded unambiguous conclusions from being drawn concerning conditions under which such resistance could be expected. Furthermore, damage observed ahead of the advancing crack tip within a colony could also be influenced by the presence of sub-surface colonies. Therefore, a substantial investigation was undertaken to study crack growth in single-colony thick specimens that were however polycolony in the specimen plane. In such specimens, colony boundaries were characterized by the lamellar misorientation across them (a kink angle and a twist angle) and by the inclination of the colony boundary to the vertical plane. The results of this investigation will be presented and experimental and analytical difficulties encountered will be highlighted.

10:10 AM
Micromechanical Modeling of Damage and Fracture in Lamellar Gamma-TiAl Alloys: *Alfred Cornec*[1]; Malte Werwer[1]; [1]GKSS Research Center, Inst. for Matls. Rsrch., Max-Planck-Str. 1, Geesthacht 21502 Germany

A micromechanical multiscale finite element model is proposed for the simulation of the quasistatic damage and fracture behavior of lamellar gamma-TiAl alloys at room temperature. The model is based on a periodic unit cell for the lamellar substructure (PST-structure) of the colonies. The deformation behavior of the two phases alpha-2 and gamma is described by continuum crystal plasticity, and the clevage fracture behavior by a cohesive model. The PST-unit cell is validated by comparison with experimental data from compression and fracture tests of PST-specimens. The deformation and fracture behavior of the polycrystalline compound is simulated by multiscale finite element simulations which make use of the PST unit cells as material submodels. The results from the numerical simulations are compared with experimental data.

10:30 AM
Studies of Dislocation and Twin Activities at Crack Tips in Near-Gamma Tial Using Electron Channeling Contrast Imaging: *Boon-Chai Ng*[1]; Tom R. Bieler[1]; Martin A. Crimp[1]; [1]Michigan State University, Cheml. Eng. & Matls. Sci., 2527 Engineering, E. Lansing, MI 48824-1226 USA

Low toughness of titanium aluminides at ambient temperatures has prevented designers from using TiAl based alloys extensively in aerospace and automotive industries. Material toughness is typically associated with the ability to arrest cracks. Understanding how and why cracks propagate and arrest in these alloys will provide meaningful information for the improvement of toughness in these alloys. In this study, notched 4-point bend specimens of a near-gamma TiAl alloy were loaded in-situ in the SEM until a crack propagated from the notch root. Electron channeling contrast imaging (ECCI) was used to examine dislocation and twin activities in regions where the crack arrested or where microcracks nucleated. Observations indicate that nucleation of microcracks occurred ahead of the primary crack as a result of deformation twins impinging on grain boundaries with little or no strain transfer to the adjacent grain. Measurement of twin width and local strain generated by these twins indicates that microcracks tend t o form in conjunction with thicker twins that impose higher local strains at the boundary when there is poor compatibility of twins with deformation systems in adjacent grains. This work was supported by the Air Force Office of Scientific Research under # AFRL no. F49620-01-1-0116, monitored by Dr. Craig Hartley, and by the Michigan State University Composite Materials Structures Center.

10:50 AM Invited
The Influence of Microstructure and Surface Residual Stresses on Pre-Yield Cracking in TiAl Alloys: *Xinhua Wu*[1]; Dawei Hu[1]; Mike H. Loretto[1]; [1]University of Birmingham, IRC in Matls., Edgbaston, Birmingham B15 2TT UK

The tendency for fully lamellar samples of TiAl alloys to crack at stresses well below their macroscopic yield stress has been demonstrated in recent work and because this pre-yield cracking has been found to act as failure initiation sites during subsequent fatigue testing a major programme is underway to increase our understanding of this phenomenon. A number of TiAl-based alloys are being assessed to determine the influence of microstructure, of surface finish and of the nature and level of residual surface stresses on the extent of pre-yield cracking during tensile testing. At this stage it has been shown that both the microstructure and surface finish are important and work is underway to relate these observations to the extent of residual stresses in machined, polished and in shot-peened samples in three TiAl alloys which have very different microstructures. This work will be reported.

11:20 AM
Incorporating Damage Tolerance Considerations to Aid the Insertion of Gamma Titanium Aluminides in Fracture-Critical Applications: *James M. Larsen*[1]; Andrew H. Rosenberger[1]; Kezhong Li[2]; Reji John[1]; William J. Porter[2]; [1]Air Force Research Laboratory, AFRL/MLLMN, 2230 Tenth St., Ste. 1, WPAFB, OH 45433-7817 USA; [2]University of Dayton Research Institute, 300 College Park, Dayton, OH 45469 USA

Effective and timely transition of gamma titanium aluminides into fracture-critical components in turbine engines will require the cooperation of numerous organizations in the industry and government. This presentation explores the role of damage tolerance considerations for key fatigue-limited engine components, and discusses opportunities and impediments for material insertion. Data from the gamma TiAl alloy K5 (Ti-46.5Al-3Nb-2Cr-0.2W) are used to illustrate the role of the materialís resistance to fatigue crack initiation, small-crack growth, and fracture, with emphasis on the importance of understanding statistical variability in material properties and the relationship of this variability to microstructure. In many instances, understanding these relationships appears to hold the key to widespread acceptance of limited-ductility materials for use in fracture-critical applications.

11:40 AM
Impact Resistance of Various Cast Gamma Alloys: *Bradley A. Lerch*[1]; Susan L. Draper[2]; J. Michael Pereira[3]; Wyman Zhuang[4]; [1]NASA-GRC, 21000 Brookpark Rd., MS 49-7, Cleveland, OH 44135 USA; [2]NASA-GRC, MS 49-1, Cleveland, OH 44135 USA; [3]NASA-GRC, MS 49-8, Cleveland, OH 44135 USA; [4]Aeronautical and Maritime Research Laboratory, Airframes & Engines Div., Melbourne, Victoria 3207 Australia

This paper will present a summary of a six-year study on the impact resistance of cast gamma TiAl. Several gamma alloys (48-2-2, 47-2-2, ABB-2, ABB-23 and NCG) were ballistically impacted and variables such as impact energy, projectile hardness and impact location were related to the resulting damage. The goal was to produce damage that was similar to the domestic object damage found in low pressure turbine blades in aircraft engines. Damaged samples were subsequently tested under high cycle fatigue loading using the step-test method. The fatigue strength of each alloy was characterized as a function of initial crack size and successfully modeled using a threshold-based fracture mechanics approach. Differences among the various alloys will be discussed, particularly with respect to their tensile properties, fatigue limits and fatigue crack thresholds, and their affect on the impact damage and damage tolerance of each alloy.

12:00 PM
Fracture Processes and the R-Curves for Fatigue Crack Propagation in Gamma-TiAl: *Reinhard Pippan*[1]; Andreas Tesch[1]; Matthias H^ck[2]; Manuel Beschliesser[3]; Heinrich Kestler[4]; [1]Austrian Academy of Sciences, Erich Schmid Inst. of Matls. Sci., Jahnstrasse 12, Leoben, Styria A-8700 Austria; [2]University of Leoben, Inst. of Metal Physics 8700 Austria; [3]University of Leoben, Inst. of Physl. Metall. & Matls. Testing 8700 Austria; [4]Plansee AG, Reutte A-6600 Austria

The fracture toughness as well as the fatigue crack propagation resistance exhibits strong R-curve behavior. The effect of microstructure was investigated in this study. The fracture toughness, the fatigue crack propagation behavior as well as the crack length dependence of these properties were studied in a coarse-grained designed fully lamellar, a fine-grained near Gamma and the corresponding aged microstructures (10.000 hours at 700∞C). In order to analyse the fracture process and to visualize the crack tip shielding mechanisms in situ fracture toughness tests in the scanning electron microscope were performed. The effect of microstructure and aging will be discussed.

12:20 PM
High Temperature Assessment and Fracture of Gamma TiAl Intermetallics: *Bilal Dogan*[1]; [1]GKSS Research Centre, Max-Planck-Str. 1, Geesthacht 21502 Germany

Development of unified lifing methodology for fracture critical components will provide assurance for new innovative constructions of gas turbines in choice of materials and/or fabrication methods and assurance in operation. Safe engine operation with extended component life and enhanced decision making processes is facilitated by validated defect assessment procedures. Guidelines and methodologies for high temperature defect assessment of conventional materials and in-house expertise exist in Europe and worldwide. Introduction of new materials, such as gamma based TiAl intermetallics, offer many potential benefits to gas turbine industry, though with lower ductility and fracture toughness than those of nickel based alloys. Hence, designers and manufacturers are challenged for their use in aero-engines, and by the operators in assessment of service performance and safe operation of TiAl components. Present paper reviews the defect assessment and lifing methodologies for high temperature fracture critical components. Fracture toughness and crack growth of gamma based TiAl intermetallics, Ti48Al2Cr and Ti47Al4.3(Cr,Mn,Nb,Si,B) alloys at 700∞C are reported. Creep deformation and crack growth behaviours are studied in an attempt to pave the way for developing a life assessment methodology for components made of advanced TiAl base intermetallic alloys.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C. T. Liu: Intermetallics III–Multi-Phase Intermetallics

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Seetharama C. Deevi, Philip Morris USA, Research Center, Richmond, VA 23234 USA; Fritz Appel, GKSS Research Centre, Geesthacht D-21502 Germany; Robert W. Cahn, University of Cambridge, Materials Science and Metallurgy, Cambridge CB2 3QZ UK; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Guo Liang Chen, University of Science and Technology-Beijing, State Key Laboratory for Advanced Metals and Materials, Beijing 100083 China; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208-3108 USA; Shuji Hanada, Tohoku University, Sendai 980-8577 Japan; Linda Horton, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6132 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Dong-Liang Lin, Shangai Jiao-Tong University, Shangai 200030 China; T. G. Nieh, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; Masaharu Yamaguchi, Kyoto University, Department of Materials Science & Engineering, Kyoto 606-8501 Japan

Tuesday AM — Room: 8
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Robert W. Cahn, University of Cambridge, Dept. of Matls. Sci. & Metall., Cambridge CB2 3QZ UK; Yip-Wah Chung, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208-3108 USA

8:30 AM Invited

Microstructure and Properties of Some High-Performance Thermo-Electric Compounds: Lanting Zhang[1]; Kazuhiro Ito[1]; Haruyuki Inui[1]; *Masaharu Yamaguchi*[1]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan

The Seebeck effect was already used to generate electric energy in 19th century. However, widespread use of thermoelectric generators still requires rather drastic improvements over the properties of thermoelectric materials. In particular, a relatively low thermoelectric conversion efficiency should be improved. Recently, some intermetallic compounds with high ZT values have been found and their properties have been investigated. We prepared thin film and bulk specimens of ReSi2, Zn4Sb3 and some related materials, and measured their Seebeck coefficient, electrical resistivity and thermal conductivity in a wide temperature range. These properties vary with chemical composition, microstructure and whether specimens are in the film form or in the bulk form. The results of these investigations will be presented.

8:50 AM Invited

Lattice Parameter Dependence on Long Range Ordered Degree in the Order-Disorder Transformation: Xiaodong Ni[1]; *Guoliang Chen*[1]; Timothee Nsongo[2]; Nanxian Chen[2]; [1]University of Science & Technology-Beijing, State Key Lab. for Adv. Metals & Matls., Beijing 100083 China; [2]Tsinghua University, Dept. of Physics, Beijing 100084 China

The order-disorder transformations (ODT) of stoichimetrical Ni3Al, NiAl, and TiAl are studied by using Blagg-Williams (BW) model based on embedded atom method (EAM) atomic potential. It is a first order O-D transformation for Ni3Al while its second order O-D transformation for NiAl and TiAl alloys. The relation between the lattice parameter and the long-range order (LRO) parameter is proved to be linear for the first order transition such as Ni3Al and non-linear for the second order transition such as NiAl. The calculated linear lattice parameter dependence on LRO for Ni3Al is consistent with Cahnís experimental results. The calculated lattice parameter change proportionally corresponds to the change of the free energy of the ordered phase in the region of LRO from 0 to 1 for all alloys studied. The alternative distribution of pure Ti plane and pure Al plane in TiAl structure leads the strong directional d-bonding between nearest Ti atoms in pure Ti plane and the polarization of p-electrons at Al sites pointi ng directly along the [001] direction, resulting in an enhancement of the Ti-Al bonding. When LRO=0 the structure becomes fcc and the direction bonding is eliminated. As LRO increases, the lattice parameter a value and the volume of unit cell are reduced while the lattice parameter c value is increased.

9:10 AM Invited

Atomic Processes and Phase Transitions in Complex Intermetallics: Quasicrystals: *H.-E. Schaefer*[1]; F. Baier[1]; K. Sato[1]; W. Sprengel[1]; [1]Stuttgart University, Inst. of Theoretl. & Appl. Physics, Pfaffenwaldring 57, Stuttgart 70569 Germany

Quasicrystals are intermetallics with aperiodic structures and novel physical features. Their high temperature properties as atomic diffusion, plastic deformation etc. are controlled by atomic processes as vacancy formation and migration. As identified by specific techniques various types of vacancies (vacant atomic sites) were identified in quasicrystals: (i) Structural vacancies are available in quasicrystals and approximants; (ii) Thermal vacancies were studied at high temperatures; (iii) Vacancies can be induced by electron irradiation; where the nearest neighbour atoms can be characterized chemically. Structural phase transitions in quasicrystals could be detected for the first time on an atomic scale by positron annihilation techniques in the case of decagonal Al-Ni-Co at 1140 K. These data can be quantitatively correlated to order-disorder phenomena observed by neutron scattering.

9:30 AM

Phase Equilibria in T-Al-C and T-Al-B Systems (T: Co, Rh and Ir) and Design of $E2_1$-Co_3AlC Based Heat Resistant Alloys: *Yoshisato Kimura*[1]; Kaoru Iida[1]; Fu-Gao Wei[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Matls. Sci. & Eng., Bldg. #G3-513, 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa 226-8502 Japan

We are interested in $E2_1$-Co_3AlC as a high potential strengthener of Co-base heat resistant alloys since the $E2_1$ type ordered crystal structure is almost the same as that of the $L1_2$ type excluding that a carbon atom occupies the octahedral interstice at the body-center. To enhance the mechanical properties of the $E2_1$-Co_3AlC based alloys, we have proposed the fourth element addition; rhodium and iridium substituting for the same 9 group element cobalt, and boron for carbon. It is also expected that microstructures would be thermally stabilized through modifying the lattice mismatch between (Co) and $E2_1$ phases. Objective of the present work is to investigate the phase equilibria in the transition metal corner of T-Al-C and T-Al-B systems (T: Rh and Ir), as to establish the basis of design on Co-base heat resistant alloys strengthened by $E2_1$-Co_3AlC. Phase diagrams have been experimentally determined for related alloy systems.

9:45 AM

Phase Equilibria and Microstructures in the Fe-Si-Cr-Ti System: *Hiroshi Usuba*[1]; Keisuke Yamamoto[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa-ken 226-8502 Japan

Iron-base alloys with the two-phase (disordered A2 + ordered $D0_3$) microstructure have received considerable attention as candidates for high temperature structural materials. The volume fraction, size and morphology of $D0_3$ phase can be controlled by heat treatments in Fe-Si-Cr system. Furthermore, by the Ti addition, it becomes possible to raise the order-disorder temperature and to expand the two-phase region. Effects of Si and Ti contents on microstructures and phase equilibria have been systematically investigated by transmission electron microscopy (TEM) for the alloys with constant Cr content fixed at 10at%. The Fe-Si-Cr alloys containing 12-15at% Si have the A2+$D0_3$ two-phase microstructure below about 873K. On the other hand, the Ti addition expands this two-phase region toward lower Si content at around 5at% and there exist (A2+$D0_3$+$L2_1$) three-phase region at around relatively higher Ti content. The morphology of $D0_3$ precipitates changes from plates to cuboids accompanying the precipitation of $L2_1$.

10:00 AM

Design Strategies for Oxidation-Resistant Intermetallic and Advanced Metallic Alloys: Michael P. Brady[1]; *Peter F. Tortorelli*[1]; Ian G. Wright[1]; Bruce A. Pint[1]; [1]Oak Ridge National Laboratory, MS 6115, Oak Ridge, TN 37831-6115 USA

This paper will discuss strategies for designing intermetallic and advanced-metallic alloys to form protective oxide scales. Emphasis will be placed on novel approaches based on manipulation of oxidation phenomena unique to multi-phase structures, either designed into the initial alloy microstructure or engineered to occur in-situ during subscale alloy depletion/enrichment processes. Factors which effect the maintenance of protective oxide scale formation will also be discussed. Examples will be drawn from studies of the oxidation behavior of Ti-, Fe-, and Ni- aluminides, Mo-based silicides, and Cr-based Laves phases conducted at Oak Ridge National Laboratory over the past decade.

10:15 AM Break

10:35 AM

Nanointerfaces in High Performance Materials: *R. V. Ramanujan*[1]; [1]Nanyang Technological University, Sch. of Matls. Eng., Nanyang Ave. 639798 Singapore

Interphase interfaces play a vital role in improving the structural and functional properties of a wide class of materials. Structural properties of advanced metallic systems have been vastly improved by nanoscale engineering of the properties of such interfaces. Functional properties, such as soft magnetic properties of nanomaterials, show that the interface between the nanoprecipitate and the matrix plays a crucial role in the crystallization behavior as well as in the optimization of the magnetic properties. The author has extended to a range of alloy systems the collaborative work on nanointerfaces performed by him with Dr. C.T. Liu. Two examples of the effect of interfaces on properties will be presented. First, experimental work using TEM, EDX, STEM and SEM on the stability of nanoscale interfaces in lamellar structures in the TiAl-X system will be reported, the modeling work examining the effect of the interfacial structure and composition on the stability of such interfaces will be discussed. The seco nd example will be the development of nanoscale soft magnetic precipitates using the Herzer diagram; this requires nanosized precipitates separated by nanoscale distances. Modeling work to determine the interfacial structure, composition and energy that will produce such materials will be presented. Corresponding experimental work, using TEM, EDX, DSC and resistivity, on heat treated iron and cobalt based alloys will also be presented. These examples will demonstrate the engineering importance of nanoscale interfaces.

10:50 AM

Processing of Intermetallics for Improved Ductility, Toughness, and Superplasticity: *Shankar M.L. Sastry*[1]; [1]Washington University, Mechl. Eng., CB 1185, One Brookings Dr., St. Louis, MO 63130 USA

Intermetallics literature indicates a definite trend of the beneficial effect of grain refinement on the low temperature ductility, fracture toughness, and yield strength, particularly when the grain size is reduced to less than 1µm. However, conventional thermo-mechanical processing of bulk intermetallics has not resulted in sub-micrometer sized grains. Nanograined intermetallics produced by powder processing routes, such as mechanical alloying and vapor phase synthesis, invariably have excessive contamination and incomplete densification. Thus, improvements both in ductility and toughness have not been realized using this type of nanoparticle powder processing approaches. Recent studies of the severe plastic deformation (SPD) processing by Equal Channel Angular Extrusion (ECAE) of titanium-, nickel, and iron aluminides indicate that the SPD processing is a viable method for producing ultra fine grain structures in bulk intermetallics and holds promise for improving the room temperature ductility and toughness and high temperature superplasticity of intermetallics.

11:05 AM Invited

Formation of Point Defects in TiAl and NiAl: Yulin Hao[1]; *Rui Yang*[1]; Yan Song[2]; Yuyou Cui[1]; Dong Li[1]; Mitsuo Niinomi[3]; [1]Chinese Academy of Sciences, Inst. of Metal Rsrch., Titanium Alloy Lab., 72 Wenhua Rd., Shenyang 110016 China; [2]Queen Mary, University of London, Dept. of Matls., Mile End Rd., London E1 4NS UK; [3]Toyohashi University of Technology, Dept. of Production Sys. Eng., 1-1, Hibarigaoka, Tempaku-cho, Toyohashi 441-8580 Japan

A mean-field model is proposed to estimate the atom-atom and atom-vacancy bond energies of intermetallic compounds, with the results of first principles calculations as input. The distribution and variation of point defects with temperature and chemical composition estimated with the model agreed with experimental measurements for TiAl and NiAl alloys. In particular, off-stoichiometric effects on the concentration of point defects are considered in this treatment. Compared to previous models that neglect the off-stoichiometric effects, our theoretical study suggests lower vacancy concentrations in Al-rich alloys but higher vacancy concentrations on the Ti-rich side. As a result, the differences in vacancy concentration between Al-rich and Al-lean TiAl alloys are less than those reported in previous studies. Constitutional vacancies are present but anti-site defects are forbidden in Al-rich NiAl at 0 K. For concentrated Al-rich NiAl, the vacancy concentration has its maximum at 0 K, and gradually decreases with rising temperature until diffusion is activated.

11:25 AM

A New Ru-Rich Heusler Phase: *Q. Feng*[1]; T. K. Nandy[1]; B. Tryon[1]; T. M. Pollock[1]; [1]University of Michigan, Dept. of Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA

An ordered $L2_1$ Heusler phase based on the Ru_2AlTa composition has been identified by X-ray diffraction and electron diffraction analysis in a high Ru-containing multicomponent superalloy. Such a phase has not been previously observed in Ni-base superalloys or as a bulk intermetallic. In order to study the effect of this phase on mechanical properties of multiphase superalloys, two compositions based on this Heusler phase have been melted for the evaluation of flow behavior in compression as a function of temperature. The mechanical behavior of these Ru_2AlTa intermetallic alloys will be discussed in comparison to other Ru-containing intermetallics.

11:40 AM

Microstructural Design of a Wrought Ni-Base Superalloy for Improved Resistance Against Environmental Embrittlement: *Joachim Roesler*[1]; Dominique Del Genovese[1]; Martin Goetting[1]; Debashis Mukherji[1]; Pavel Strunz[2]; Ralf Gilles[3]; [1]Technical University Braunschweig, Inst. f¸r Werkstoffe, Langer Kamp 8, Braunschweig 38106 Germany; [2]Nuclear Physics Institute, Rez 25068 Czech Republic; [3]Technische Universit‰t Darmstadt, Petersenstrasse 23, Darmstadt 64287 Germany

The wrought Ni-Fe base superalloy Inconel 706 is of particular interest as disc material for land based gas turbines because of its balanced manufacturability and elevated temperature strength. It is also under consideration for ultra high temperature steam turbine applications with prospective steam inlet temperatures of about 700∞C. Due to the operation conditions of these advanced machines and the damage tolerant design philosophy, satisfactory creep crack growth resistance at temperatures beyond 600∞C is of paramount importance. However, Inconel 706 suffers from environmental embrittlement of the grain boundaries as other comparable alloys do, leading to fast creep crack growth and brittle intergranular fracture at the above mentioned temperature range. It will be demonstrated here that careful design of the microstructure by slight adjustment of the heat treatment cycle used today is capable of reducing the creep crack growth rate by several orders of magnitude. The reasons for this drastic effect are el ucidated by detailed microstructural analysis. ìLockingî of the grain boundaries by discontinuous precipitation of the ?-phase turns out to be the most relevant aspect. As this precipitation process happens in competition to other precipitation reactions in the grain interior (in particular: ?¥/? ¥¥-formation), it sensitively depends on the thermal history. Interrupted heating experiments and in-situ neutron scattering are used to explain these interdependencies. Based on these findings, a modified heat treatment cycle for improved resistance of Inconel 706 against environmental embrittlement is suggested. It is particularly well suited for large forgings as it employs slow cooling from the solutioning temperature.

11:55 AM

Oxidation Behavior of Aluminide Alloys in the Operating Temperatures of Solid Oxide Fuel Cells: *Weizhong Zhu*[1]; Seetharama C. Deevi[1]; [1]Philip Morris USA, R&D Ctr., 4201 Commerce Rd., Richmond, VA 23234 USA

Several different iron aluminde alloys have been investigated as potential interconnects for intermediate temperature solid oxide fuel cell (SOFC) stack, i.e., operating below 800°C. Properties of paramount importance such as oxidation resistance, contact resistance of the scale, and thermal expansion were examined. Emphasis is put on a comparative study of the oxidation behavior of aluminide alloys and stainless steels. Oxide scales of the alloys exposed to air in the temperature range of 600-800°C for up to 1000hr were identified by X-ray diffraction. The predominant scale of alloys oxidized at 800°C for 1000hr is either iron nitride or aluminum nitride with a minor amount of alumina. Oxidation behavior was investigated in an attempt to predict the long-term response of the alloys in real SOFC atmospheres. Contact resistances of iron aluminides and stainless steels were compared after extended periods of oxidation. We discuss our comparisons and discuss the potential of iron aluminides for interconnect applications.

12:10 PM Invited

Laves PhasesñStructure and Stability: *Frank Stein*[1]; Martin Palm[1]; Gerhard Sauthoff[1]; [1]Max-Planck-Institut fuer Eisenforschung, Physl. Metall., Max-Planck-Str. 1, Duesseldorf 40237 Germany

Laves phases form the largest group of intermetallic phases. Although they are well known since long, there are still unsolved problems concerning the stability of the respective crystal structures. The Laves phases crystallize with a cubic $MgCu_2$-type structure or a hexagonal $MgZn_2$-type or $MgNi_2$-type structure which differ only by the particular stacking of the same four-layered structural units. It is still not possible to predict which of the possible structure types is the stable one for a Laves phase compound AB_2. Phase transformations from a cubic low-temperature structure to a hexagonal high-temperature structure were observed as well as stress-induced transformations from the hexagonal structure to the cubic one. In addition, deviations from the stoichiometric composition were reported to result in a change of the stable structure for various systems. Existing models using geometrical and electronic factors for predicting the occurrence and the structure type of a Laves phase are reviewed. On the basis of extensive experimental results obtained for various binary and ternary alloy systems the applicability of the different models to real systems is checked and existing problems are demonstrated.

International Symposium on Structures and Properties of Nanocrystalline Materials: Mechanical Properties II

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee

Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Tuesday AM — Room: 14B
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Harriet Kung, US Department of Energy, Office of Basic Energy Scis., Germantown, MD 20874 USA; Ennio Bonetti, University of Bologna, Dept. of Physics & INFM, Bologna 40127 Italy

8:30 AM

Theoretical and Experimental Investigation of the Indentation Size Effect with Spherical and Pyramidal Indenters: J. G. Swadener[1]; *E. P. George*[2]; G. M. Pharr[3]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., PO Box 1663, Los Alamos, NM 87545

USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6093 USA; [3]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996 USA

When tested in small volumes, metals and polymers often show dramatic increases in strength compared to their macroscopic strength. Thus, for example, when hardness is measured with pyramidal indenters, an increase in hardness is observed with decreasing depth of penetration, with the effect becoming apparent at depths less than a few microns. Recently we have shown that a similar size effect exists also in spherical indentation. However, it is manifested not through the depth of indentation but rather through the radius of the indenter; significant increases in hardness are observed as the radius of the indenter is reduced. A model based on geometrically necessary dislocations was developed for spherical indentation which agrees with the experimental results for all but the smallest indent used in our study. The primary advantage of spherical indentation is that size effects and work hardening effects can be de-coupled and determined separately unlike in the case of sharp indenters. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC. Additional funding was supplied by the US Department of Energy Office of Basic Energy Science under contract W-7405-Eng-36.

9:00 AM
Characterization and Mechanical Properties of Sputter-Deposited Ni_3Al Thin Films: *Evan Andrew Sperling*[1]; Arda Genc[1]; Rajarshi Banerjee[1]; Peter M. Anderson[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Monolithic thin films of stoichiometric Ni_3Al were prepared by UHV magnetron sputtering from an alloy target. Deposition at room temperature on oxidized Si wafers produced nanocrystalline Ni_3Al oriented in the (111) direction. The size of the nanocrystalline grains was studied as a function of deposition parameters by means of X-Ray diffraction and TEM. As the Ar sputtering pressure was increased from 5 mTorr to 100 mTorr, the deposition rate and grain size decreased. Elevated temperature deposition with the substrate pre-baked and held at 400∞C produced larger grains and a chemically ordered Ni_3Al phase. The hardness and modulus of the nanocrystalline films are being measured using nanoindentation and the relationship between structure and properties will be discussed in this paper. Furthermore, the mechanical properties of the nanocrystalline films will be compared to the properties of bulk Ni_3Al alloys.

9:20 AM
Effects of Decreasing Layer Thickness on the High Temperature Mechanical Properties of Copper Based Nanoscale Multilayers: *Nathan Allan Mara*[1]; Daniel Coyle[1]; Alla Sergueeva[1]; Amit Misra[2]; Amiya Mukherjee[1]; [1]University of California-Davis, Cheml. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA

Nanometer-scale polycrystalline multilayered films (with layer thickness less than 100 nm) typically exhibit high yield strength, often approaching the theoretical strength at room temperature. However, most attempts to characterize the mechanical behavior of such thin films have been carried out using nanoindentation and scanning force microscopy or their combination and there is little data regarding their mechanical behavior at elevated temperatures. In the present investigation, the microstructure and mechanical properties of polycrystalline Cu-based multilayers (Cu/Ni and Cu/Nb) at diminishing length scales were investigated. Free-standing samples were tested in uniaxial tension at temperatures ranging from 25 to 700°C at different strain rates. The high strength of these new materials is attributed to their layered, nanoscale structure and a variety of related strengthening mechanisms. This investigation is supported by NSF, Division of Materials Research, grant NSF-DMR-9903321.

9:40 AM
Plastic Behavior of Nanocrystalline Fe Powder Formed by Mechanical MillingñA Nanoindentation Study: *Dongchan Jang*[1]; Michael Atzmon[2]; [1]University of Michigan, MSE, 3062 H. H. Dow Bldg., Ann Arbor, MI 48109-2136 USA; [2]University of Michigan, NERS & MSE, Cooley Bldg./N. Campus, Ann Arbor, MI 48109-2104 USA

Numerous authors have reported on the so-called ìinverse Hall-Petchî effect, namely the decrease of hardness with decreasing grain sizes when the latter are of the order of a few nanometers. This effect has been controversial because many synthesis methods result in porous samples, thus introducing artifacts into hardness measurements. In the present study, we report on nanoindentation hardness measurements, conducted on a scale smaller than the particle size. The grain sizes of our samples have been well characterized by the Warren-Averbach analysis. We observe a hardness increase with decreasing grain size to dimensions below those previously reported, suggesting potential artifacts in past studies. Below 18 nm, a slight hardness decrease is observed. We have also measured the strain-rate sensitivity, and found it to increase with decreasing grain size, suggesting the possibility of grain-boundary sliding at room temperature.

10:00 AM Break

10:20 AM
Superplastic Behavior of As-Equal Channel Angular Pressed 5083 Al Alloys: *Kyung-Tae Park*[1]; Dong Hyuk Shin[2]; [1]Hanbat National University, Div. of Adv. Matls.. Sci. & Eng., San 16-1, Dukmyung-Dong, Yuseong-Gu, Taejon 305-719 S. Korea; [2]Hanyang University, Dept. of Metall. & Matl. Sci., 1271, Sa 1-Dong, Ansan, Kyunggi-Do 425-791 S. Korea

A submicrometer grained structure was introduced into the two grades of the 5083 Al alloys, one containing 0.2 wt.% of Sc and the other without Sc, and their superplastic behavior was investigated. For the alloy without Sc, low temperature superplasticity was obtained while high strain rate superplasticity was possible in the alloy containing Sc. The characteristics of low temperature and/or high strain rate superplasticity of the alloys were discussed in view of microstructural evolution during equal channel angular pressing and subsequent deformation.

10:40 AM
Variation of Mechanical Properties and Texture Development in Copper and Aluminium Under ECAP: *Ralph Jˆrg Hellmig*[1]; Yuri Estrin[1]; Seung Chul Baik[2]; Min Hong Seo[3]; Hyoung Seop Kim[3]; [1]Technische Universit‰t Clausthal, Inst. f¸r Werkstoffkunde und Werkstofftechnik, Agricolastr. 6, Clausthal-Zellerfeld 38678 Germany; [2]Pohang Iron & Steel Company, Ltd., Techl. Rsrch. Labs., Pohang 790-785 Korea; [3]Chungnam National University, Dept. of Metallurgl. Eng., Daejeon 305-764 Korea

In this talk we compare the mechanical, microstructural and textural variations during the four well known routes (A, BA, BC and C) of equal channel angular pressing (ECAP) of 99.9% purity copper and technical purity aluminium. Strength and tensile ductility of the ECAP processed materials were determined as a function of the number of pressings. In addition, TEM investigations were carried out to resolve the cell structure formed as a result of ECAP. The texture evolution for all processing routes was determined by X-ray diffraction. The results obtained were used to verify a dislocation based constitutive model that combines a two-internal variable approach with crystal plasticity. The model was shown to give a very good prediction of microstructural evolution, texture development and variation of thetensile strength of the materials studied.

11:00 AM
Deformation Twins in Titanium Processed by Equal Channel Angular Pressing: *Jongryoul Kim*[1]; Inyoung Kim[1]; SooHyun Han[1]; *Yong-Seog Kim*[2]; *Dong Hyuk Shin*[1]; [1]Hanyang University, Metall. & Matls. Sci., Ansan, Kyungggi-do 425-791 Korea; [2]Hong Ik University, Matls. Sci. & Eng., Seoul 121-791 Korea

After the first pass of ECAP at 623 K, the microstructure of pure titanium was composed of {10 1} deformation twin bands, rather than dislocation slip bands. This result raises an interesting question on characteristics of the twins developed during ECAP. Maximum shear strain that can be accommodated by deformation twin in most of HCP metals is in a range from 0.13 to 0.22. This strain is much smaller than the strain imposed by ECAP, ~1.83. This indicates that the twinning modes developed in pure Ti by the ECAP might be significantly different from the conventional twinning modes. In this respect, HREM analysis was conducted to investigate the atomic structure of {10 1} twin. The analysis revealed that various twinning modes such as the b1, b2 and b3 mode occurred in the sample. These modes can accommodate much lager deformation in comparison with the conventional b4 mode.

Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging: Fundamentals, Phases, Wetting and Solidification

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Srini Chada, Motorola, Department APTC, Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Jud Ready, MicroCoating Technologies, Atlanta, GA 30341 USA; Jin Yu, KAIST, Center for Electronic Packaging Materials 305-701 Korea

Tuesday AM	Room: 15B
March 4, 2003	Location: San Diego Convention Center

Session Chairs: James P. Lucas, Michigan State University, Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824 USA; Srinivas Chada, Motorola, Dept. APTC, Plantation, FL 33322 USA

8:30 AM Invited
Ag3Sn Plate Formation in the Solidification of Near Ternary Eutectic Sn-Ag-Cu Alloys: *Sung K. Kang*[1]; Won K. Choi[1]; Da-Yuan Shih[1]; Donald W. Henderson[2]; Timothy Gosselin[2]; Amit Sarkhel[2]; Charles Goldsmith[3]; Karl J. Puttlitz[3]; [1]IBM, T. J. Watson Rsrch. Ctr., Yorktown Heights, NY 10598 USA; [2]IBM Corporation, 1701 North St., Endicott, NY 13760 USA; [3]IBM Corporation, Hopewell Junction, NY 12533 USA

Near ternary eutectic Sn-Ag-Cu alloys, such as Sn-3.8Ag-0.7Cu (wt.%), are leading candidate solders to replace the Sn-Pb eutectic solder in the transition to Pb-free soldering technology. This alloy system has three solid phases - b (Beta) Sn, Ag3Sn and Cu6Sn5. In the solidification of near eutectic Sn-Ag-Cu alloys, the equilibrium eutectic transformation is kinetically inhibited. While the Ag3Sn phase nucleates with minimal undercooling, the b-Sn phase requires a typical undercooling of 15 to 25∞C for nucleation. As a consequence of this disparity in the required undercooling for nucleation, large Ag3Sn, plate-like, structures can grow rapidly within the liquid phase, during cooling, before the final solidification of solder joints. At lower cooling rates, the large Ag3Sn plates can subtend the entire cross section of solder joints and can grossly influence the mechanical deformation behavior of such solder joints under thermomechanical fatigue conditions. It is demonstrated that the Ag3Sn plate formation can be suppressed kinetically by utilizing elevated cooling rates or suppressed thermodynamically by using reduced Ag compositions.

8:55 AM
Solidification of Eutectic and Near-Eutectic Alloys in the Ag-Cu-Sn System: Leonid Snugovsky[1]; *Doug D. Perovic*[1]; John Rutter[1]; [1]University of Toronto, Matls. Sci. & Eng., Wallberg Bldg., 184 College St., Toronto, Ontario M5S 3E4 Canada

The techniques of DSC, quenching of samples during freezing and slow unidirectional solidification were used to evaluate the ternary eutectic temperature, composition and freezing characteristics of the Ag-Cu-Sn system. The eutectic temperature was determined to be 217+/- 0.4°C at a composition of 3.5 wt% Ag, 0.9 wt% Cu , balance Sn. The ternary eutectic was found to be of the faceted (Ag3Sn) faceted (Cu6Sn5) nonfaceted (Sn) type. The Ag3Sn phase forms a broken lamellar microstructure while the Cu6Sn5 phase forms a fibrous microstructure: the fibers become hollow hexagonal prisms at very low growth rates. The volume fraction occupied by the two compound phases, taken together, was measured as 5.5+/-0.5 and calculated as 5.63 vol.%. The compound phases are very poor nucleation catalysts for Sn, an effect that can result in the formation of Sn dendrites even from a melt of eutectic composition. When fractured, both compound phases showed brittle behaviour.

9:15 AM
In Situ Observation and Simulation of Solidification Process in Soldering SOP with Sn-Ag-Cu Lead-Free Alloy: Motoharu Haga[1]; Keun-Soo Kim[1]; *Katsuaki Suganuma*[1]; [1]Osaka University, Inst. of Scientific & Industl. Rsrch., Mihogaoka 8-1, Ibaraki, Osaka 567-0047 Japan

In soldering, because cooling speed depends on a design of assemblies and material properties of components, especially lead frame alloys such as 42 alloy and Cu, the formation of defects on solidification will be strongly influenced by those factors. In the present study, solidification process of SOP joint (42 alloy and Cu lead frame) was analyzed by the in situ observation combined with solidification simulation. In situ and microstructural observations of the circuit boards with a SOP soldered with Sn-3wt%Ag-0.5wt%Cu were carried out. The β-Sn dendrite structure in the solder fillet of a SOP with 42 alloy lead frames is larger compared with that of a Cu lead frame one. This is attributed to the slower cooling speed of 42 alloy lead frame SOP than that of Cu lead frame one. The solidification for a SOP joint is not uniform and locally time dependent. The solder surface of slowly cooled region of the 42 alloy lead frame SOP exhibited solidification cracks and rough surface by coarsening the dendrite structure. According to the simulation of solidification process, the relationship between solidification process and formation characteristics of solidification defects for a SOP joint can be clarified. The experimental results explained well by the simulation of solidification process.

9:35 AM
Effects of Impurities on Phase Equilibria and Reactivity of Near Eutectic Sn-Ag-Cu Solder: *Jaeyong Park*[1]; Rajendra Kabade[1]; Choong-Un Kim[1]; Ted Carper[2]; Puligandla Viswam[3]; [1]The University of Texas at Arlington, Matls. Sci. & Eng., 500 W. 1st. St., Woolf Hall, Rm. 325, Arlington, TX 76019 USA; [2]Nokia Mobile Phones, Inc., Rsrch. & Tech. Access, 6000 Connection Dr., MS 2:3207, Irving, TX 75039 USA; [3]Nokia Mobile Phones, Inc., Rsrch. & Tech. Access, 6000 Connection Dr., MS 2-200, Irving, TX 75039 USA

Among various Pb-free solders, Sn-Ag-Cu (ACS) alloys with near eutectic composition are considered the most attractive replacement for Pb-Sn solders and are in fact used in a few selected cases. Owing to its practical importance, numerous studies have been conducted on ACS alloys in order to understand their properties as a solder, including microstructural characteristics, wettability to various UBM schemes, and mechanical reliabilities. In practical applications, however, a solder system is not free from contaminants because it is forced to be in contact with lead-frame or substrate. A small amount of contaminants introduced in the ACS system may induce a substantial change in the phase equilibria and various related properties, and, therefore its influence needs to be investigated. This study presents the influence of several elements introduced into near eutectic ACS system on its phase equilibria and reactivity with Cu substrate. The elements investigated include Au, Co, Fe, Ni, Pb and Pd.

9:55 AM
Effect of Polyethylene Glycol Residues on the Reliability of Printed Wiring Boards: Jason P. Pilon[2]; *Laura J. Turbini*[1]; [1]University of Toronto, Ctr. for Microelect. Assem. & Pkgg., 184 College St., Rm. 150B, Toronto, Ontario M5S 3E4 Canada; [2]University of Toronto, Dept. of Matls. Sci. & Eng., 184 College St., Rm. 140, Toronto, Ontario M5S 3E4 Canada

Polyethylene glycol (PEG) is a common constituent of water-soluble flux. Zado first investigated the effect of PEG on the surface insulation resistance (SIR) of comb patterns soldered with PEG-containing flux. After SIR testing he examined the boards using SEM and noted lead residues between anode and cathode. He ascribed the low SIR readings to interaction between PEG and the epoxy substrate. Later, Brous proved that PEG was present in the epoxy and developed a chemical extraction process using acetonitrile. Recently, Bent studied the effect of PEG containing fluxes on SIR using copper-metallized comb patterns. For the formulation containing PEG without activators, he observed a silvery residue between comb fingers. Although these residues were not dendritic in nature, it was shown by SEM/EDS to contain copper. This paper will characterize and identify the chemistry that leads to these residues.

10:15 AM Break

10:25 AM Invited
Horizontal and Vertical Flow of Molten Pb-Free Solders Along V-Grooves on Si: *King-Ning Tu*[1]; [1]University of California-Los Angeles, 405 Hilgard Ave., Los Angeles, CA 90095-1595 USA

V-grooves of 100 micron in width, along the [110] directions on (001) Si wafer surfaces, were etched by lithographic technique. They were coated with Cr/Cu thin films. When a bead of molten solder was dropped on a V-groove which was placed horizontally, the melt ran along the V-groove and obeyed the Washburn model with a parabolic rate of motion. The rate was measured with a CCD camera. When the V-groove was dipped vertically into a pot of molten solder, the solder climbed up the V-groove to a certain height due to the capillary effect. Combining these measurements and the measurement of wetting angles of a solder cap on a flat Cu/Cr/Si surface, we attempted to analyze the surface tension and viscosity of the molten Pb-free solder. A comparison of the wetting behaviors of Pb-free solders with eutectic SnPb solder has been made. The effect of interfacial intermetallic compound formation on the wetting behavior will be discussed.

10:50 AM
Phase Equilibria of Au-In-Sb-Sn Lead-Free Solder Alloys: Jin Zhanpeng[1]; K. Ishida[2]; Liu Huashan[1]; Wang Chong[1]; Wang RiChu[1]; *Liu Chunlei*[1]; [1]Central South University, Ctr. of Phase Diagram & Matls. Design, Sch. of Matls. & Eng., Changsha, Hunan 410083 China; [2]Tohoku University, Dept. of Matls. Sci., Grad. Sch. of Eng., Sendai 980-8579 Japan

Phase relations in the Au-In-Sb-Sn quaternary system, the most basic information necessary for the development of lead-free solder alloys, were studied using the CALPHAD (CALculation of PHAse Diagrams) technique. Gibbs energies describing the constituent phases were obtained by optimizing the obtained data on the experimental phase diagrams and thermodynamics properties. The thermodynamic database of this multi-component system provides various information on phase equilibria such as liquidus and solidus surface, isothermal and vertical sections, mole fractions of the phase constituents, etc., and thermodynamics properties such as heat of mixing, surface energy, etc., of related liquid alloys in this multi-component lead-free solder system. Typical examples for the phase diagrams and thermodynamics properties of Au-In-Sb-Sn quaternary system are calculated. Some experiments were also carried out to get some phase equilibria information of this quaternary system and verify the reasonableness of the thermodynamics database of this quaternary system and its constituent ternary and binary systems.

11:10 AM
Chemical Reactions that Lead to Conductive Anodic Filament Formation: Laura J. Turbini[1]; *Antonio Caputo*[1]; [1]University of Toronto, Ctr. for Microelect. Assem. & Pkgg., 184 College St., Rm. 150B, Toronto, ON M5S 3E4 Canada

Conductive Anodic Filament (CAF) is a failure mode in printed wiring boards (PWBs) which occurs under high humidity and high voltage gradient conditions. The filament, a copper salt, grows from anode to cathode along the epoxy-glass interface. In there recent work, Ready and associates have identified CAF to be an atacamite-like insoluble compound, $Cu_7Cl_4(OH)_{10}\sum H_2O$, with semiconductor-like properties. The natural occurring form of atacamite has been identified to be $Cu_2Cl(OH)_3$. Meeker and LuValle have proposed a general kinetic model to describe the chemical reactions, which lead to reliability failures in PWBs. Using this model, the various chemical processes that are involved in the formation of CAF in processed PWBs will be analyzed and defined, using various analytical techniques. This paper will define the chemical reactions that result in the formation of CAF.

11:30 AM
An Investigation of Addition of Copper Nanopowders on Microstructure, Microhardness and Solderability of Sn-3.5%Ag Solder Wetting on Copper Substrate: *D. C. Lin*[1]; T. M. Guo[1]; *G.-X. Wang*[1]; T. S. Srivatsan[1]; [1]The University of Akron, Dept. of Mechl. Eng., 302 E. Buchtel Mall, Akron, OH 44325-3903 USA

Eutectic Sn-3.5%Ag solder is a potentially attractive and economically viable alternative to replace lead-containing solders. In this research investigation, a series of experiments on solder/copper substrate couple were conducted with the objective of examining the influence of nano-sized copper powders on strength and performance of the eutectic Sn-3.5% Ag solders. Even addition of small percentages was found to improve strength. The strength increase is rationalized as being due to the presence and distribution of the intermetallic compound both within the solder matrix and at interfaces of the solder/copper substrate coupled with refining of the eutectic solder. Examination of the microstructure revealed that addition of copper powders refined the primary tin-rich phase in the solder matrix while concurrently refining the size of the intermetallic compound present at and along the solder substrate interface. Addition of nanopowders of copper has a positive effect on the solderability of composite solder when compared to the addition of micron sized copper powder. This presentation will highlight salient features pertaining to thermal characteristics during solidification, microstructure development, microhardness and the role of nanoparticle addition on tinósilver solder/copper substrate couple.

11:50 AM
Novel Ultrasonic Soldering Method for Lead-Free Solders: *Keitaro Kago*[1]; Kenji Ishida[2]; Toshihisa Horiuchi[2]; Kazumi Matsushige[2]; Shiomi Kikuchi[3]; Kenichiro Suetsugu[4]; Shunji Hibino[5]; Takashi Ikari[4]; [1]Kyoto University, Venture Business Lab., Yoshidahonmachi, Sakyo-ku, Kyoto 606-8501 Japan; [2]Kyoto University, Grad. Sch. of Eng., Yoshidahonmachi, Sakyo-ku, Kyoto 606-8501 Japan; [3]The University of Shiga Prefecture, Dept. of Matls. Sci., 2500 Hassaka-cho, Hikone, Shiga 522-8533 Japan; [4]Matsushita Electric Industrial Company, Ltd., Environml. Production Eng. Lab., 2-7 Matsuba-cho, Kadoma, Osaka 571-8502 Japan; [5]Matsushita FA Engineering Company, Ltd., 2-7 Matsuba-cho, Kadoma, Osaka 571-8502 Japan

The Sn-Bi solder has good prospects for the future as the low temperature type lead-free solder. But its wettability on the copper land is low and the bonding strength is weak. It is due to the segregation of Bi needle-like microcrystalline at the bonded interface. We found that the application of ultrasound improved the mechanical strength of Sn-Bi solder. It is because the ultrasound miniaturized the microcrystallines of Bi to form the interfacial layer. Moreover, we invented newly the ultrasonic soldering apparatus. By using it, we can apply the ultrasound to the printed wire board (PWB). The temperature of PWB increased above the melting point of Sn-58Bi solder and the solder paste melted only by ultrasound without other heating method. The wettability was improved. It may be because the ultrasound miniaturized the microcrystallines. The technique we invented will improve the usability and reliability of Sn-Bi solders.

12:10 PM
Surface Tension and Density Measurements of Sn-Ag-Sb Liquid Alloys, Phase Diagram Calculations and Modeling: *Z. Moser*[1]; W. Gasior[1]; Janusz Pstrus[1]; X. J. Liu[2]; I. Ohnuma[2]; K. Ishida[2]; [1]Polish Academy of Sciences, Inst. of Metall. & Matls. Sci., Reymonta St. 25, Krakow 30-059 Poland; [2]Tohoku University, Dept. of Matls. Sci., Grad. Sch. of Eng., Aoba-yama 02, Sendai 980-8579 Japan

The maximum bubble pressure method has been used to measure the surface tension of pure Sb, surface tension and density (dilatometric method) by adding to (3.8 at.% Ag-Sn) eutectic 0.03, 0.06 and 0.09 molar fractions of Sb at the temperature range 2770C to 9770C. The linear dependencies of densities and surface tensions on temperature were observed and they were described by straight-line equations. Moreover, a phase diagram calculations were performed and the resulting optimised thermodynamic parameters were used for modeling of the surface tension. In addition, a non-equilibrium solidification process using the Scheil model was simulated and compared with the equilibrium solidification behaviour in some Sn-Ag-Sb base alloys.

Magnesium Technology 2003: Magnesium Casting and Solidification and Simulations

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, LMD-Magnesium Committee, International Magnesium Association, MPMD-Solidification Committee
Program Organizers: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

Tuesday AM	Room: 2
March 4, 2003	Location: San Diego Convention Center

Session Chairs: Gerald S. Cole, Consultant USA; David Henry StJohn, University of Queensland, CAST Div. of Matls. Eng., Brisbane, QLD 4072 Australia

8:30 AM Invited
Quantitative Analysis of Cast Magnesium Alloy Microstructures and their Relationships to Processing and Properties: *Arun M. Gokhale*[1]; [1]Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Successful applications of cast magnesium alloys for structural applications require production of castings that exhibit reproducible mechanical and environmental response. Therefore, a thorough understanding of correlations between variability in the microstructure and the mechanical response, and modeling of mechanical response of the cast magnesium alloys are of interest. In this contribution, quantitative fractography and stereological techniques are applied to correlate the mechanical properties of high-pressure die-cast AZ91 and AM60 alloys with the microstructural and fractographic parameters. The data point to the control of process parameters that can reduce the variability in mechanical properties. The contribution also reports applications of the nano-indentation technique for calculation of the local constitutive behaviors of the skin and the interior regions of the cast metal through a numerical solution to the inverse problem using finite elements (FE) based simulations. The constitutive equations provide critical input to simulate micro-mechanical response of these cast microstructures.

9:00 AM Invited

Solidification of Cast Magnesium Alloys: *David Henry StJohn*[1]; Arne Kristian Dahle[1]; Trevor B. Abbott[2]; Mark Nave[3]; Ma Qian[1]; [1]University of Queensland, CAST, Matls. Eng., Sch. of Eng., Brisbane, Queensland 4072 Australia; [2]Monash University, CAST, Sch. of Physics & Matls. Eng., Melbourne, Victoria 3168 Australia; [3]Deakin University, Sch. of Eng. & Tech., Pigdons Rd., Geelong, Victoria 3217 Australia

A description of the key solidification steps in the formation of the as-cast microstructure of magnesium alloys will be presented. The focus will be on the two common magnesium alloy groups: Mg-Al alloys and Mg-Zn-rare earth alloys. The key elements to be described will be nucleation (including grain refinement), growth of the primary phase and the formation of the eutectic phases. In addition the effect of casting process (e.g. high pressure die casting, gravity die casting or sand casting) on the outcomes from solidification will be discussed. This will include consideration of the mechanisms of the formation of casting defects during solidification such as banded defects in high pressure die cast components.

9:30 AM

Characterizing Solidification by Non-Equilibrium Thermal Analysis: *Yancy W. Riddle*[1]; Makhlouf M. Makhlouf[1]; [1]Worcester Polytechnic Institute, Metal Procg. Inst., 100 Institute Rd., Worcester, MA 01609 USA

This paper presents thermal analysis data collected during the solidification of several industrially important Mg alloys. Microscope observations correlating thermal analysis with microstructure are included. These alloys are part of a larger effort at the Advanced Casting Research Center (ACRC) to catalogue the solidification behavior and resultant microstructures of established technical alloys and experimentally promising alloys. Altogether the effort is meant to aid future researchers of Mg alloys by providing a resource from which basic understanding of solidification in complicated multi-component Mg alloys may evolve. Intelligent development of new alloys with attractive mechanical properties stems from an understanding of the solidification process. Analysis of solid content accumulates as a function of both time and solidification rate, phase precipitation events, and resultant microstructures are presented for several key Mg casting alloys.

10:00 AM Break

10:15 AM

Mechanical Properties of Investment Casting AZ91 Alloy: *Zhan Zhang*[1]; Chantal Turcotte[1]; Guy Morin[3]; [1]Technologies Intermag, Inc., R&D, 357 rue Franquet, Sainte-Foy, Quebec G1P 4N7 Canada; [3]Centre IntÈgrÈ de Fonderie et de MÈtallurgie, 3247 rue Foucher, Trois-RiviÈres, Quebec G18 1M6 Canada

Shell mould investment casting process is able to produce thin wall and complex shape parts with good surface finish. This process is commonly applied to aluminium alloys, superalloys, steel, etc.. But its applications to magnesium alloys are limited due to mould-magnesium reactions. For enlarging the market of magnesium products, Technologies Intermag Inc. has developed technologies to prevent mould-magnesium reactions and successfully cast magnesium parts with this process. This paper presents the mechanical properties (ultimate tensile strength, yield strength, and elongation) of AZ91E magnesium alloy cast by shell mould investment casting process under as cast and after heat treatment. The results show that the mechanical properties of the test bars are much higher than those with plaster mould casting and close to those with sand mould casting. And the experiments indicate that shell investment casting process is a better alternative to sand mould casting than plaster mould casting for thin wall and complex shape parts. The effect of the cooling rate on mechanical properties of test bars will be described. The microstructure of the test bars under different conditions will be presented and the relationship between mechanical properties and microstructure will be discussed. In addition, the process for preparation of test bar shell moulds as well as the methods to prevent mould-magnesium reactions will be presented.

10:45 AM Invited

Influence of Physical Data and Cooling Conditions on the Solidification in Mg-Die Castings: *Konrad Weiss*[1]; Christoph Honsel[1]; [1]RWP GmbH, Am Muensterwald 11, Roetgen Germany

The simulation result have to fit not only in a quality way into the real foundry world. More and more the results of the simulation will be used to optimise the casting process. Here it is necessary to have a very precise knowledge of the phys. Data as well as the description of the geometry. In this paper we describe the development of physical data concerning the heat transfer during filling and solidification by different casting methods. The low pressure as well as the gravity die casting method is be used to get real data. Practical tests done under different test conditions are carried out to develop the right physical Data as well as the right conditions in the interface mould metal. The different surface conditions and the influence of the temperature of the die and the molten metal are important for the layout of the die casting process. The three different shapes where applied to different mould materials and casting methods. The cooling conditions where tested on specific items. The cooling shape was not restricted by the drilling conditions. The effect of the different cooling conditions are generated and measured. The results are the basics for the heat transfer conditions. The results are applied to real castings. The cellular phone housings are the real applications in low pressure. The use of the new conditions leads to a good process description.

11:15 AM

Vacuum-Sealed Molding Process for Magnesium Casting: Numerical Simulations and Design of Experiments: *Sayavur I. Bakhtiyarov*[1]; Ruel A. Overfelt[1]; [1]Auburn University, Mechl. Eng., 202 Ross Hall, Auburn, AL 36849-5341 USA

Given the rapid projected growth of magnesium casting usage in the automotive parts marketplace, there is a critical need to develop casting technologies applicable for a wide variety of part shapes, sizes, and production rates. The application of magnesium components will allow reductions in vehicle weight, improvement of parts-consolidation and minimizing of noise/vibration characteristics. Magnesium is considered the fastest-growing metal in cars. Many of the earliest applications are instrument panel components, valve covers, etc. These are produced by a reasonably mature die-casting technology. Many potential structural components for automobiles could be converted to magnesium if a high volume, cost effective manufacturing process were developed. In this paper we propose a modified vacuum-sealed molding process for magnesium casting. Some results of computer modeling and design of experimental research are presented.

11:45 AM

Numerical Simulation of Natural Convection in Magnesium Alloy Squeeze Casting: *Alfred Yu*[1]; *Naiyi Li*[2]; *Henry Hu*[1]; [1]University of Windsor, Mechl., Matls. & Auto., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ford Motor Company, Mfg. Sys. Dept., Ford Rsrch. Lab., Rm. 2349, MD3135, 2101 Village Rd., Dearborn, MI 48124 USA

Natural convection has received less attention than conduction in metal solidification processes. It may play an important role in solidification due to its potential influence on solutes distribution in melts and grain structure. A coupled mathematical model has been developed to simulate the solidification phenomena of squeeze cast magnesium alloy (AM50). An analysis based on a Control-Volume Finite Difference approach and an Enthalpy Transforming Model was employed to understand the heat transfer and fluid flow in a cylindrical casting during solidification. The effect of natural convection on the total solidification process of squeeze casting AM50 was examined under various processing parameters, and comparison results are presented and analyzed.

Materials Lifetime Science and Engineering - I

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; D. Gary Harlow, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015-3085 USA; Dwaine L. Klarstrom, Haynes International, Inc., Kokomo, IN 46904-9013 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Robert P. Wei, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015 USA

Tuesday AM — Room: 18
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Peter K. Liaw, University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; Stephen Dale Antolovich, Washington State University, Mechl. & Matls. Eng., Pullman, WA 99164-2920 USA

TUESDAY AM

8:30 AM Invited

Linkage Between Safe-Life and Crack Growth Approaches for Fatigue Life Prediction: *D. Gary Harlow*[1]; Robert P. Wei[1]; [1]Lehigh University, Mechl. Eng. & Mech., 19 Memorial Dr. W., Bethlehem, PA 18015-3085 USA

Current methods for predicting fatigue lives are based upon the safe-life and/or the crack growth approaches, both of which are empirically based. They do not adequately reflect long-term operating conditions, or identify the sources and extent of their contributions to variability. A linkage between these two approaches is established and demonstrated herein. The S-N response and its variability in fatigue life are related to key internal variables, both deterministic and random, that can be clearly identified in crack growth models. The identification and understanding of the role of these variables are paramount for predicting fatigue crack growth and subsequent damage evolution. The effectiveness of this approach is shown through analysis of an extensive set of S-N data for 2024-T4 aluminum alloy from the literature. Variability associated with manufacturing and material variables are considered. To put life prediction on sound scientific and probabilistic bases, this demonstrated linkage should be adopted.

9:00 AM Invited

A Mechanistic Based Study of Fatigue Crack Propagation in the Single Crystal Nickel Base Superalloy CMSX-2: Stephen Dale Antolovich[1]; Bruce Fergus Antolovich[2]; [1]Washington State University, Mechl. & Matls. Eng., 247 Dana Hall, Pullman, WA 99164-2920 USA; [2]Special Metals Corporation, Numeric Modlg., New Hartford, NY 13413 USA

FCP testing was done on CMSX-2 at 298 & 973K in air and vacuum for two crystallographic orientations. Two fracture surface morphologies were observed; precipitate shearing and precipitate avoidance. TEM revealed two different deformation mechanisms; precipitate shearing and bypass. A finite element analysis (FEA) was performed to predict the stresses and strains ahead of the crack tip for each tested condition. FEA, TEM and SEM observations suggest that crack growth is non-self similar and fracture surface geometry is controlled by the state of stress calling into question use of LEFM. High resolved shear stresses in the direction of the Burgers vector along with low normal stresses to the precipitates produced crystallographic crack growth while low resolved shear stresses and high normal stresses led to precipitate avoidance. This fundamental understanding of the crack growth mechanisms will assist in more accurately modelling FCP rates in single crystal Ni-base superalloys.

9:30 AM

Temperature Evolution During Fatigue of HASTELLOYÆ C-2000Æ Alloy: *Tarik A. Saleh*[1]; Bing Yang[1]; Peter K. Liaw[1]; Raymond A. Buchanan[1]; Dwaine L. Klarstrom[2]; [1]University of Tennessee-Knoxville, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

Infrared (IR) thermography was employed as a nondestructive evaluation technique to investigate the thermal behavior of HASTELLOYÆ C-2000Æ alloy during fatigue testing. Stress-strain and S-N Curves at R ratios of 0.1 and -1 (Ûmin/Ûmax, where Ûmin and Ûmax are the applied minimum and maximum stresses, respectively) were generated for the alloy. Four stages of temperature evolution were observed: (1) an initial increase in temperature followed by a temperature decrease to (2) an equilibrium (steady-state) temperature, which held until (3) a rapid temperature increase and (4) a temperature drop after specimen failure. Additionally, the relationship between the fatigue lifetime and the steady-state temperature during fatigue testing was explored experimentally and theoretically. This research is supported by the National Science Foundation Integrative Graduate Education and Research Training (IGERT) program with Drs. W. Jennings and L. Goldberg as contract monitors, and Haynes International, Inc.

9:50 AM

Elevated-Temperature Crack Growth Behavior of Nickel-Base HASTELLOYÆ X Alloy: Y. L. Lu[1]; L. J. Chen[1]; *P. K. Liaw*[1]; G. Y. Wang[1]; S. A. Thompson[2]; J. W. Blust[2]; P. F. Browning[2]; A. K. Bhattacharya[2]; J. M. Aurrecoechea[2]; D. L. Klarstrom[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Solar Turbines, Inc., 2200 Pacific Hwy., PO Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The crack-growth behavior of a nickel-base HASTELLOYÆ X superalloy was investigated under fatigue, creep, and hold-time test conditions at 816°C and 927∞C. The hold-time tests were conducted with 2-minute and 1-hour holds. It was found that the fatigue crack-growth rate could be correlated with the stress intensity factor range, DK, and the creep crack growth rate can be correlated with the stress intensity factor, K. The crack grew faster at a higher temperature. It was also noted that the introduction of a hold time at the maximum load led to an increase in the cyclic crack-propagation rate. The longer hold time gave the greater crack-growth rate. The crack-growth rates in the hold-time tests were predicted from the crack growth rates obtained from both the fatigue and the creep crack growth tests, using a semi-empirical linear summation model. Crack growth rate predictions reproduce most of the characteristics observed experimentally.

10:10 AM

Development of a Generic Creep-Fatigue Life Prediction Model: *Tarun Goswami*[1]; [1]Arkansas Tech University, Mechl. Eng., 1815 Coliseum Dr., Russellville, AR 72801 USA

Creep-fatigue life prediction of high temperature materials continues to be in the developmental stage due to the number of variables encountered and their influence in altering the cyclic lives. A generic life prediction method has been developed in this paper using the data from the following alloy groups; a) pure metals, b) solder alloys, c) copper alloys d) titanium alloys, e) low alloy steels, f) stainless steels, g) tantalum alloys and h) superalloys. Nearly 2200 data points were compiled from the published sources on the above alloy groups and independent parameters were identified within the following four test parameters, namely; 1) strain range, 2) strain rate, 3) dwell time and 4) temperature. A change in the above four parameters altered the cyclic fatigue life under the creep-fatigue conditions. Statistical Analysis Software (SAS) was used to analyze the data. The independent parameters were combined with each other up to a sixth order to produce a multi-variate, best-fit, equation. The coefficient of correlation obtained in this case was 15%. Therefore, transformation functions were used in logarithmic terms and four independent parameters were evolved transforming strain rate, strain rate, dwell time and temperature, parameters. The data were analyzed with SAS and residuals were plotted for each combination of test parameters from the equation. This equation produced a coefficient of correlation of 54% and the residual plots, plotting the residuals with observed life showed a very close match in the normal probability distribution plot. These results are summarized and the generic life prediction equation presented in this paper.

10:30 AM

Microstructural Simulation and Life Prediction for Advanced Gas Turbine Coatings: *Liang Jiang*[1]; Suchismita Sanyal[1]; Sundar Amancherla[1]; Ji-Cheng Zhao[1]; Melvin R. Jackson[1]; K. Anand[1]; P. R. Subramanian[1]; Kaisheng Wu[2]; Yunzhi Wang[2]; [1]General Electric Company, One Research Cir., Niskayuna, NY 12309 USA; [2]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

In the severe operation environment of gas turbines, advanced metallic coatings are used as environmental coatings or as bond coats between superalloy substrate and ceramic thermal barrier coating for protecting substrate superalloy from oxidation and hot corrosion. Both oxidation and interdiffusion between the metallic coatings and substrate alloys result in complex and progressive degradation of the metallic coating, especially the reduction of aluminum concentration in the coating. Coupled with thermodynamic and kinetic databases, a simplified model with consideration of Ni-Cr-Al multicomponent diffusion in single phase was developed for the calculation of interdiffusion profiles and lifing of metallic coatings. A more sophisticated model with consideration of Ni-Cr-Al multicomponent diffusion in multiphase was developed for the microstructural evolution in coatings. The models †help understand the failure mechanisms of metallic coatings for gas turbines and can capture the detrimental effects resulting from oxidation and interdiffusion.

10:50 AM

Effect of the Microstructure on the Very High Cycle Fatigue of Titanium Alloys at Room and Cryogenic Temperatures: *Claude Bathias*[1]; [1]CNAM/ITMA, 2 rue ContÈ, Paris 75003 France

In several industries, the required design lifetime of many components often exceeds 10^8 cycles. This requirement is applicable to aircraft turbine but also for space engine such as cryogenic pump. Although a large amount of fatigue data has been published in the form of S-N curves, the data in the literature have been limited to 10^7 cycles. Time and cost constraints rule out the use of conventional fatigue tests of more 10^7 cycles to check structural materials. A possibility of accelerated testing of specimens is now considered by using a piezoelectric fatigue machine working at high frequency. A short description of the machine is given. It is found that there is no asymptote after 10^7 cycles and that the SN curve is always decreasing up to 10*9 cycles for TA4V6 or 6246 Ti alloy at room and at cryogenic temperature. The effect of forging process is very important in the gigacycle fatigue, inducing sometime a shift of more than 100 MPa between the SN curves. After 10^7 cycles the initiation mechanism is always at the interior of the specimen and related to the size of the alpha platelets. The mechanism is discussed. A special device has been built in our laboratory in order to test specimens in liquid hydrogen, and to determine the S-N curve up to 10^8 cycles. A short description of the device is given. A comparison between those results and data in gas helium is made and discussed. It is confirmed that the fatigue strength at 20 Kelvin is much higher than at room temperature, but affected by hydrogen or helium.

11:10 AM Invited

Contact Fatigue of Dental Multilayers: *M. Huang*[1]; X. Niu[1]; Z. Suo[1]; V. Thompson[2]; D. Rekow[2]; W. O. Soboyejo[1]; [1]Princeton University, Princeton Matls. Inst. & Dept. of Mechl. & Aeros. Eng., Princeton, NJ 08544 USA; [2]New York University, Coll. of Dentistry, New York, NY 10034 USA

This paper presents the results of a combined experimental and computational study of contact fatigue in multilayered structures that are relevant to dental restorations. These include top layers of crown materials (zirconia, alumina and Empress II) that are bonded to dentin or materials with equivalent elastic properties to dentin. Cyclic contact-induced deformation is shown to result in viscoelastic/viscoplastic deformation and ratcheting phenomena that give rise ultimately to sub-surface pop-in. The local conditions for pop-in are also determined from hydraulic fracture experiments that utilize fluid pressure in the loading of cracks/notches within dental materials. The measured pop-in conditions are then used as critical conditions in finite element models of the effects of viscous flow on the pressure loading within cracks/notches. Finally, the physical insights and finite element models are integrated into a mechanism-based framework for the prediction of fatigue damage in multilayered structures that are relevant to dental restorations.

11:40 AM Invited

Physically-Based Models for the Prediction of Fatigue and Dwell Fatigue Crack Growth in Ti-6242: Comparison of Models with Experiments: F. McBagonluri[2]; Chris Mercer[1]; E. Akpan[1]; W. Shen[1]; *W. O. Soboyejo*[1]; [1]Princeton University, Princeton Matls. Inst., D404 E. Quad., Princeton, NJ 08544 USA; [2]University of Dayton, Dept. of Matls. Eng., Dayton, OH 45390 USA

This paper presents the results of a combined experimental and theoretical/computational study of fatigue and dwell fatigue crack growth in Ti-6242 with three different microstructures (equiaxed, elongated and colony a/b structures). The micromechanisms of short and long fatigue growth are studied using interrupted fatigue tests and beachmarking techniques. The insights developed during these experiments are then used to guide the development of physically-based fracture mechanics models for the prediction of crack growth during fatigue or dwell fatigue loading. The predictions from the models are compared with experimental measurements of sub-surface and surface crack growth. The implications of the results are then discussed for the modeling of fatigue and dwell fatigue in engineering structures and components.

Materials Processing Fundamentals: Powder Synthesis and Processing

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee, EPD-Process Fundamentals Committee

Program Organizers: Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA; Princewill N. Anyalebechi, Grand Valley State University, L. V. Eberhard Center, Grand Rapids, MI 49504-6495 USA

Tuesday AM	Room: 1A
March 4, 2003	Location: San Diego Convention Center

Session Chair: Princewill N. Anyalebechi, Grand Valley State University, L. V. Eberhard Center, Grand Rapids, MI 49504-6495 USA

8:30 AM

Extremely Rapid Sintering of Bronzes,Steels and Refractory Metals (W, Re) and Hard Metals (WC, Tin, Tib2, Etc.) In2.45 Ghz Multi- and Single- Mode Microwave Fields: Dinesh Agr[1]; Jiping Cheng[1]; Rustum Roy[1]; *A. Upadhyay*[1]; [1]The Pennsylvania State University, Matls. Rsrch. Inst., University Park, PA 16802 USA

To the surprise of nearly everyone including the authors, it has been shown that all common metal powders can be sintered routinely in common microwave (2.45, 0.915 GHz) apparatus in 10-30 minutes. The sintering times for all are reduced by more than one order of magnitude. Pre-alloying mixtures react even faster than alloyed powders. Continuous compositional gradation from steels to ìhard metalsî are easily achieved. Magnetic metals display unexpected behaviors in H fields. Microstructure differences (SEM level) will be used to suggest that the nature of the porosity may provide the explanation for some of the improved properties. In two-phase systems the unique ìanisothermalî conditions attained in a microwave field also play a major role.

9:00 AM

Processing Ti3Al/Al2O3 Composites Using Mechanical Milling: *Gorgees Adam*[1]; [1]University of Waikato, Waikato Ctr. for Adv. Matls., PB 3105, Hamilton 2001 New Zealand

A Ti3Al(O)/Al2O3 metal matrix composite material was produced by sintering an Al/TiO2 composite powder produced using high mechanical milling. The composite typically contains 50-60vol.%Al2O3. We have recently developed a low cost process which allows us to reduce (or increase) the volume fraction of the Al2O3 phase in the composite material. This process involves the use of several physical and chemical separation techniques. The detailed process itself is confidential at the time of writing this abstract, and a provisional patent application has been filed. By using this process, we have successfully reduced the volume fraction of the Al2O3 phase in the Ti3Al/Al2O3 composite from 50-60% to 20-30%. This favourable outcome shows that the process facilitates the freedom of controlling the volume fraction of the Al2O3 particles in the composite to achieve a variety of different mechanical properties desirable for different applications. This paper will introduce and discuss this process and the mechanical properties of the composites produced.

9:20 AM

Effect of Surface Oxidation on the Sintering Activity of Al Powders: *Fei Tang*[1]; Stacy Scott[1]; Fran Laabs[1]; Iver Eric Anderson[1]; [1]Iowa State University, Ames Lab., Metal & Ceram. Scis. Prog., Rm. 223, Metals Dvlp. Bldg., Ames, IA 50011 USA

In order to promote the sintering behavior and eventually improve the performance of Al parts made by powder metallurgy, very high purity Al powders (99.99%) with a thin, passive surface oxide were produced by a gas atomization reaction synthesis (GARS) technique. Vacuum sintering at 525°C for 24, 48, 72 and 100 h of fine (dia.<10µm), tap densified commercial purity (CP) and GARS Al powders produced extensive necking in the GARS samples, but essentially no detectable sintering in the CP samples. BET measurements of the surface area of both kinds of Al samples at 500, 525, and 550°C quantified the relative sintering kinetics. The enhancement of GARS Al sintering activity may be attributed to the very thin oxide (3-5nm) layer on the GARS powder compared with the 10-20nm oxide layer on the CP Al powder. Funding of this project is from DOE Basic Energy Sciences under contract number W-7405-Eng-82.

9:40 AM

Inductively Coupled Plasma Deposition of Ceramic Thin Films: A Review: Patrick R. Taylor[1]; *Edgar E. Vidal*[1]; [1]Colorado School of Mines, Metallurgl. & Matls. Eng., 1500 Illinois St., Hill Hall, Golden, CO 80401-1887 USA

The deposition of ceramic materials onto substrates has always been of special interest in the development of surface related technologies. Special interest has been placed on using thermal plasma as the deposition method, specifically inductively coupled plasmas. Many different modifications to the same basic principle of deposition have been performed through out the materials research community. Such technique has many advantages over more conventional techniques, such as high deposition rates, high surface area coverage, clean energy source, no contamination from electrodes and near atmospheric pressures. Materials deposited vary from simple oxide systems like TiO2 to more complex structures like ternary oxides and nitrides. This paper reviews recent developments relative to the inductively coupled plasma deposition of thin films.

10:00 AM Break

10:20 AM

Analysis of Gas Atomization Process Physics: *Iver Eric Anderson*[1]; Robert Lee Terpstra[1]; Sebastian Rau[2]; Boris Rauscher[2]; Richard S. Figliola[3]; [1]Iowa State University, Ames Lab. (USDOE), 222 Metals Dvlp. Bldg., Ames, IA 50011 USA; [2]University of Bremen, Bremen

28331 Germany; [3]Clemson University, Dept. of Mechl. Eng., Clemson, SC 29631 USA

The development of a discrete jet, close-coupled gas atomization nozzle which operates over a wide pressure range in an open wake condition has enabled the study of the effect of gas velocity, from Mach 2.5 to 3.5, on the size distribution of the resulting powder. Achievement of a sufficiently narrow geometric standard deviation, 1.95 or less, of the powder size results permitted a meaningful comparison with the mean size predictions of two atomization models based on capillary and acceleration wave formation. High-speed cinematography permitted observation of near-field (primary) and downstream (secondary) melt break-up processes. Correlations with gas-only measurements and schlieren imaging detected changes in wake recirculation related to melt stream splitting as a function of atomization gas pressure (for N2 and Ar) and nozzle design. Analysis of these results suggests further work to increase understanding of this complex process. Support by USDOE-BES and the Process Science Initiative under contract no. W-7405-Eng-82.

10:40 AM

Barium-Strontium Titanate Powder Synthesis in a Thermal Plasma Reactor: Patrick R. Taylor[1]; *Edgar E. Vidal*[1]; Ana M. Vera-Arcetti[2]; [1]Colorado School of Mines, Dept. of Metallurgl. & Matls. Eng., 1500 Illinois St., Hill Hall, Golden, CO 80401-1887 USA; [2]Indinvest, S.p.A. , Production Dept., Biassono, Milan Italy

The synthesis of high-purity ultra-fine barium strontium titanate powders (BST: Ba0.6Sr0.4TiO3) has been performed in a non-transferred arc D.C. thermal plasma reactor. Slurries of barium and strontium nitrates and titanium dioxide were used as precursors. Oxygen and argon were used as carrier gas, and pure argon and combinations of argon and nitrogen were used as plasma gases. The plasma torch power was varied, as well as the oxygen flow rate and the total molar concentration of the precursors. A graphite plate was designed to protect the torch electrode and lower the level of impurities in the product. The powders collected from the different sections of the reactor were characterized using Transmission and Scanning Electron Microscopy, X-Ray Diffraction, and chemical analysis. The powders produced showed total conversion to BST, ultra-fine particle size (< 30 nm) and high purity, which makes this a potential ceramic material for electronic devices.

11:00 AM

Preparation of Acicular Nickel Powder by a Modified Polyol Process: *Hun S. Chung*[1]; Dong J. Kim[1]; Kening Yu[2]; [1]Korea Institute of Geoscience and Mineral Resources, Minls. & Matls. Procg., 30 Kajung-dong, Yoosung-ku, Daejeon 305-350 Korea; [2]Chinese Academy of Sciences, Inst. of Process Eng., PO Box 353, Beijing 100080 China

Morphology of fine metal powders becomes important depending on their industrial applications. In the present study, a combined process of chemical reduction and polyol processes was attempted to prepare acicular nickel powders. Experiments were carried out under the various conditions of reactant concentration, reduction time, and reaction temperature. The nickel powders of 1~10 micrometers in length and 0.1~0.6 micrometers in diameter were synthesized with nickel hydrooxide slurry in ethylene glycol which was obtained from nickel sulfate and sodium hydroxide, and hydrazine hydrate was used as a reducing agent. The reduction was completed within 60 minutes at the temperature of 80 to 95 Celsius, and the produced powder was finely dispersed without agglomeration.

11:20 AM

The Mechanical Properties of Al Foam by Batch Process: *Bo Young Hur*[1]; *Sang Youl Kim*[1]; *Soo Han Park*[1]; *Duck Kyu Ahn*[1]; *Chol Kyu Kwon*[1]; [1]Gyeongsang National University, ULSFoM-NRL, ReCAPT, Div. of Matl. Eng., 900, Kajoa-Dong, Chinju 660-701 S. Korea

Metallic foam which has light-weight and thermal resistance structure was expected synergy effect on mechanical, thermal absorption properness from energy and environmental problem it was composed of solid and pore as composite material was controlled of pore shaper, cell size, cell size distribution and porosity by manufacturing process. Porous Aluminum was manufactured by down pot stirring furnace with used Aluminum alloy. The measurement of porosity were calculated by image microscope, and so. The uniaxial compression test was performed with a universal testing machine. The Compressive stress-strain curve of cellular solids can be divided into three regions: the linear elastic, the plateau, and the densification. The absorption energy per unit volume of Al foam was evaluated. The absorption energy can be evaluated by integrating the area under the stress-strain curve. The average value of absorption energy per unit volume of Al foam at a strain of 50% is 3MJ/m3.

11:40 AM

Effects of Heat Treatment on Foam Struts and their Measurement: *Jikou Zhou*[1]; [1]Princeton University, Dept. of Mechl. & Aeros. Eng., Princeton, NJ 08544 USA

This paper examines the effects of heat treatment on open-cell aluminum foam struts. First, the strut microstructures are characterized via SEM, FIB and TEM. The stress-strain behaviors of struts extracted from the as-received and aged foams are determined using micro-tensile testing techniques. The strut microstructures and mechanical properties are then compared with those of the bulk materials from which the foams are fabricated. Finally, the effects of heat treatment of foam struts are discussed.

Materials Processing Under the Influence of Electrical and Magnetic Fields - III

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Joanna R. Groza, University of California-Davis, Chemical Engineering Material Science Department, Davis, CA 95616 USA; George S. Dulikravich, The University of Texas at Arlington, Multidisciplinary Analysis, Inverse Design, and Optimization (MAIDO) Program, Department of Mechanical and Aerospace Engineering, Arlington, TX 76019 USA; Nagy H. El-Kaddah, University of Alabama, Department of Metallurgical & Materials Engineering, Tuscaloosa, AL 35487-0202 USA; James W. Evans, University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA; Zuhair Munir, University of California, College of Engineering, Davis, CA 95616-5294 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Tuesday AM Room: 14A
March 4, 2003 Location: San Diego Convention Center

Session Chair: George S. Dulikravich, University of Texas at Arlington, Dept. of Mech. & Aero. Eng. Dept. MAIDO Prog., Arlington, TX 76019 USA

8:30 AM Opening Remarks

8:35 AM

Fluid Flow Motion and Solidification Under Combined Action of Magnetic Fields and Microgravity: *Ben Q. Li*[1]; Kai Li[1]; Yan Shu[1]; Henry C. de Groh[2]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Pullman, WA 99164 USA; [2]NASA Glenn Center, Cleveland, OH 44135 USA

Mathematical models are developed to represent g-jitter induced fluid flows and their effects on solidification under combined action of magnetic fields and microgravity. The numerical model development is based on the finite element solution of governing equations describing the g-jitter driven fluid flows, heat transfer and solutal transport with and without an applied magnetic field in space vehicles. The numerical model is applied to study an upcoming shuttle flight experiment system that involves the growth of crystals from melts. To validate the model predictions, an g-jitter simulator is developed using the oscillating wall temperatures where timely oscillating fluid flows are measured using a laser PIV system. The measurements are compared well with computed results.

8:55 AM Invited

Using the Magnetic Body Force to Eliminate g-Jitter in Microgravity Experiments: C. D. Seybert[2]; *James W. Evans*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA

Although orbiting laboratories provide environments which can minimize density-based convection, residual accelerations, or g-jitter, still exist. This complicates experiments, for example on solidification at the diffusional limit. Low-frequency or steady (ìDCî) accelerations are difficult or impossible to eliminate by damping. This paper details the use of magnetic fields to counter these accelerations. Using accelerometers and a three-axis magnet system, residual acceleration could be effectively eliminated. Absolute control of the conditions during, say, crystal growth could be achieved. We have designed a magnet with a uniform magnetic field gradient for possible use in space experiments. Modeling of the flow induced by random accelerations will be presented, along with comparisons to the flow when the g-jitter is countered by the magnet system.

9:25 AM Invited
Optimization of Intensities and Orientations of Magnets Controlling Melt Flow During Solidification: *Brian H. Dennis*[1]; George S. Dulikravich[2]; Marcelo Colaco[2]; [1]University of Tokyo, Grad. Sch. of Frontier Sci., 7-3-1 Hongo, Bunko-ku, Inst. of Environml. Studies, Tokyo 113-8656 Japan; [2]University of Texas at Arlington, Mech. & Aeros. Eng. Dept., Multidisciplinary Analy., Inverse Design & Optimization (MAIDO) Prog., UTA Box 19018, Arlington, TX 76019 USA

When growing a large single crystal, such as a semiconductor crystal, it is desirable to remove the thermally induced convection effects entirely, leading to heat transfer by pure conduction. If the melt speed is low, it is less likely that small particles of the crucible wall will be deposited in the mushy region and consequently in the crystal. It is also desirable to achieve a distribution of the dopant in the crystal that is as uniform as possible. A least-squares spectral finite element method was used to develop an accurate computer code for prediction of solidification from a melt under the influence of an externally applied magnetic field. A micro-genetic optimizer was used with this solidification analysis to determine the distributions of the magnets and the shape of the crucible that will minimize the convective flow throughout the melt or in desired regions of the melt only.

9:55 AM Break

10:10 AM
Melt Flow Instability and Turbulence in Electromagnetically Levitated Droplets: Xin Ai[1]; *Ben Q. Li*[1]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Pullman, WA 99164 USA

Magnetically levitated droplets have found a wide range of applications in both industrial and materials processing in microgravity environment. One of the critical issues concerning the applications of these droplets is the internal melt flows induced by the applied electromagnetic fields. This paper addresses these issues through the numerical analysis of fluid flow instabilities and flow transition to turbulence chaotic motions and direct numerical simulations of turbulence in magnetically levitated droplets. Numerical implementation and computed results are presented for flow instability and turbulence flows in magnetically levitated droplets under terrestrial and micro gravity conditions.

10:30 AM
A Contactless Inductive Velocity Reconstruction Method for Metallic and Semiconducting Melts: *Frank Stefani*[1]; Gunter Gerbeth[1]; [1]Forschungszentrum Rossendorf, MHD Dept., PO Box 510119, Dresden D-01314 Germany

The knowledge of the flow in metallic and semiconducting melts is important for a number of industrial problems. Optical methods cannot be applied due to the opaqueness of those fluids. Ultrasonic methods have problems with wetting and thermal stability when applied in hot metallic melts. Evidently, contactless methods for velocity determination would be highly desirable. In some applications even a coarse knowledge of the flow topology and the direction of the main eddies would be of high value. We study the possibility of velocity reconstruction in electrically conducting fluids from external measurements of different induced magnetic fields. It is shown that for a reliable velocity reconstruction the effect of the electric potential at the fluid boundary on the induced magnetic field should be taken into account.

11:00 AM
Fluid Velocity Measurements in Electro-Vortical Flows: *A. Cramer*[1]; P. Terhoeven[2]; A. Kraetzschmar[2]; G. Gerbeth[1]; [1]Forschungszentrum Rossendorf, MHD Dept., PO Box 510119, Dresden D-01314 Germany; [2]Moeller, Ltd., Hein-Moeller-Str. 7-11, Bonn D-53115 Germany

This class of flows is characterized by the feeding of strong electrical currents into a volume of liquid metal. The interaction of these currents with their own magnetic field drives flows which exhibit phenomena like formation of jets and pinch-effect. Further increasing the current may lead to disruption of the melt column accompanied with a discharge. This can be used for a liquid metal current limiter with self-healing properties. The paper will give results from a systematic study of the flow structure performed by means of our mechano-optical velocity probe. The measurements have been done in a liquid metal column carrying an axial current of several 100 Amps. The column was separated by an insulating plate with a small hole drilled in the center thus restricting the current path. The measured flow structure of the evolving jet was found to be in good agreement with our numerical simulation.

11:20 AM
Droplet Deformation and 2-D/3-D Marangoni Flow Phenomena in Electric Fields: Yunlong Huo[1]; *Ben Q. Li*[1]; Song Suping[2]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Sloan 207, Pullman, WA 99164 USA; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Pullman, WA 99164 USA; [2]Washington State University, Sch. of Mechl. & Matls. Eng., Pullman, WA 99164 USA

An integrated numerical model is presented for free surface phenomena and Marangoni fluid flows in electrically levitated droplets under both terrestrial and microgravity conditions. The model development is based on the boundary element solution of the Maxwell equations simplified for electrostatic levitation applications and the free surface deformation that is primarily caused from the surface Maxwell stresses resulting from the applied electric fields. The electric and free surface model is further integrated with a finite element model for the surface tension induced fluid flows in the levitated droplets. Both 2-D and 3-D fluid flow structures may be developed in the electrically levitated droplets depending on the applied laser heating sources. The integrated model is applied to study the electric field distribution, free surface deformation and 2-D and 3-D (both transient and steady state) internal fluid flow structures in normal and micro gravity for both symmetric and tetrahedral heating arrangement.

11:40 AM
Natural Convection of Liquid Gallium Under a Magnetic Field with Application of Electric Current: *Masayuki Kaneda*[1]; Toshio Tagawa[1]; Hiroyuki Ozoe[1]; [1]Kyushu University, IAMS, 6-1 Kasuga Koen, Kasuga, Fukuoka 816-8580 Japan

The heat transfer rate of natural convection of liquid gallium in a cubic enclosure was measured under the influence of magnetic field with or without external electric current applied from outside. The one vertical wall of the enclosure was heated with constant heat flux and an opposing wall was cooled isothermally by electro-conductive walls and four other walls were thermally and electrically insulated. Magnetic field horizontal and parallel to the hot and cold walls was applied. Electrodes were placed at the center of both hot and cold walls and electric current flows from the cold wall to the hot wall, which presumes the thermoelectric current. Resulted Nusselt number on the hot wall decreased by applying a magnetic field only, but both magnetic field and electric current change the average Nusselt number. Combination of magnetic filed and electric current may control the natural convection by induced Lorentz force.

Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction: Global & Local Interrogation

Sponsored by: Structural Materials Division,
Program Organizers: James M. Larsen, US Air Force, Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA; Leo Christodoulou, Defense Advanced Research Agency, Arlington, VA 22203-1714 USA; William J. Hardman, Naval Air Systems Command, Propulsion and Power Directorate (AIR 4.4.2), Patuxent River, MD 20670-1534 USA; Andrew J. Hess, Naval Air Systems Command, Propulsion and Power Directorate, Patuxent River, MD 20670-1534 USA; J. Wayne Jones, University of Michigan, College of Engineering, Department of Materials, Science and Engineering, Ann Arbor, MI 48109 USA; Stephan M. Russ, Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA

Tuesday AM   Room: 16A
March 4, 2003   Location: San Diego Convention Center

Session Chairs: Stephan M. Russ, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA; Michael L. Dent, Air Force Research Laboratory - Anteon Corp., Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA

8:30 AM Invited
Overview of Technologies to Support Risk-Based Prognostic Decision System: *Thomas A. Cruse*[1]; [1]Consultant, 398 Shadow Pl., Pagosa Springs, CO 81147 USA

The presentation covers a recently-completed AF-sponsored study to explore and assess technologies that could be used to support a risk-based decision for continued flight operations of individual legacy turbine engines. The focus of the study was to reduce the uncertainties in the prognostic evaluation of remaining life through innovative material state sensing and new thermal history recording sensors. The decision system is based on a Bayesian network life model and a predictive confidence interval strategy tied to the quality of the input data. Material state modeling is the most urgent need given that the material is the source of greatest uncertainty in making a risk-based decision for continued operations. The presentation will discuss each element of the proposed decision system while emphasizing the required innovations for state awareness, microstructural modeling, and network modeling that must be addressed in future prognostic systems.

9:00 AM Invited

Enhanced Prognosis Through Probabilistic Damage Assessment: *Stephen J. Hudak*[1]; Michael P. Enright[1]; Harry R. Millwater[2]; R. Craig McClung[1]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., San Antonio, TX 78238-5166 USA; [2]University of Texas at San Antonio, San Antonio, TX 78249 USA

This paper examines the potential benefits of integrating fully probabilistic life prediction methods into the prognostics process. This was accomplished by using an existing probabilistic life predictions code (DARWIN) as a demonstration platform. DARWIN was modified to accept actual aircraft engine (F-16/F100) data from flight data recorders. Modules were also added to DARWIN to transform engine speed (RPMs) to stress, filter the stress to enhance computational efficiency, add crack initiation (to the existing crack propagation capability), and expand the frequency of inspection capability to simulate continuous onboard monitoring. Example analyses were performed for fatigue critical locations on a generic bladed compressor disc. Extensive usage data were analyzed and used to characterize the influence of inter- and intra-mission usage variability on disk fatigue life and probability of failure. Analyses were also performed to demonstrate the significant reliability benefits of continuously monitoring damage with onboard sensors, even when detection sensitivities are appreciably less than those commonly employed in depot inspections.

9:25 AM Invited

Damage Indicators for Advanced Life Prediction Systems: *Eva L. Suarez*[1]; Yoshika Omori[1]; Michael J. Duffy[1]; [1]Pratt & Whitney, Fatigue & Fracture Mech., 400 Main St., MS 163-07, E. Hartford, CT 06108 USA

New techniques for tracking engine component life usage have been devised to improve current engine life tracking processes, providing the customer with a safer, more reliable, affordable, and comprehensive damage prognosis tool. The new technique consists of damage accumulated at real time by quantitative algorithms, relating aircraft and engine gas path continuously measured or calculated performance data to the assessment of total remaining capability for the engine componentís critical life limiting locations. Two methodologies have been developed, evaluated, and compared against its classical life-tracking counterpart. One is a statistical approach where the uncertainty is evaluated by levels of confidence on the results. The second is a first principle physical relation governing damage mechanisms and the outcome was compared to Finite Element Methods thermal and structural results. Material behavior algorithms have been developed for various failure modes. Life indicator prediction results have shown very high correlation to design analysis as indicated by the high statistical coefficients of determination.

9:50 AM Break

10:20 AM Invited

eSTORM: Enhanced Self Tuning On-Board Real-Time Engine Model: *Thomas Brotherton*[1]; Al Volponi[2]; Rob Luppold[3]; Donald L. Simon[4]; [1]Intelligent Automation Corporation, 13029 Danielson St., Ste. 200, Poway, CA 92064 USA; [2]Pratt & Whitney, E. Hartford, CT 06108 USA; [3]Luppold & Associates, 164 N. Water St., W. Newton, PA 15089 USA; [4]US Army Research Laboratory, NASA Glenn, Cleveland, OH 44135 USA

A key to producing reliable engine diagnostics resides in the fusion of processing algorithms. Fusion of techniques has been shown to improve diagnostic performance while simultaneously reducing false alarms. Presented here is an approach that fuses a physical model called STORM (Self Tuning On-board, Real-time engine Model), with an empirical neural net model to provide a unique hybrid model called enhanced STORM (eSTORM) for engine diagnostics. STORM is a piecewise linear approximation of the engine cycle Deck. STORM provides significant improvement over existing real-time engine model methods however, there are several effects that impact engine performance that STORM does not capture. Integrating an empirical model with STORM accommodates the modeling errors. This paper describes the development of eSTORM for a Pratt & Whitney F117 turbofan engine. Comparative results of using STORM and eSTORM on simulated engine data are presented. eSTORM is shown to work extremely well in reducing STORM modeling errors and biases for the conditions considered.

10:45 AM Invited

Vibration-Based Crack Detection in Rotating Engine Components: *HÈctor M. RodrÌguez*[1]; Huageng Luo[1]; Dennis M. Corbly[2]; [1]GE, Global Rsrch., 1 Research Cir., K1-2A72, Niskayuna, NY 12309 USA; [2]GE, Aircraft Engines, 1 Neumann Way, A413, Cincinnati, OH 45215 USA

A physics-based approach for the detection of cracks in rotating disks is presented. The approach takes advantage of the distinct behavior in the vibration response of cracked rotating disks. In particular, radial-axial cracks induce a unique vibration response as they open due to tensile hoop stresses caused by centrifugal loading. The crack opening forces a redistribution of the disk mass. This mass redistribution yields an additional unbalance that is proportional to the square value of the speed, and hence the resulting crack-induced unbalance force is proportional to the fourth power of the speed. This unique unbalance response characteristic brings the opportunity to implement a monitoring system to detect early stage disk cracks by measuring the vibration response of the rotating assembly at non-invasive locations (e.g., bearings). The present work shows an experimental study of the proposed crack detection approach. In the test, an aircraft engine disk in a spin pit is used to investigate the change in the rotor vibration response during the growth of an induced crack in a highly stressed location. The results from the test provide a further insight into the technique and its potential implementation as a prognostic tool for engine health management.

11:10 AM Invited

USN Rotor Spin Facility Experience with Disk Crack Detection Sensing Technology: *Greg C. Muschlitz*[1]; [1]NAVAIR, AIR 4.4.7.1, B106, Unit 4, 22195 Elmer Rd., Patuxent River, MD 20670-1534 USA

The US Navy has been actively pursuing and developing advanced disk crack detection sensing systems from various vendors. The Rotor Spin Facility has been actively testing these systems to determine maturity and capability of these systems. Spin testing capability will be discussed including the results of several disk crack detection efforts recently concluded. Future plans will also be discussed.

11:35 AM Invited

Updating Turbomachinery Fatigue Life Remaining by Use of Blade-Tip Sensors: *Andreas H. von Flotow*[1]; [1]Hood Technology Corporation, 1750 Country Club Rd., Hood River, OR 97031 USA

Blade tip sensors in turbomachinery can monitor aspects of the stress state of blades and can detect accumulating damage of blades and their host disks. A system employing these sensors can operate continuously, whenever the machine operates. The challenge is to develop techniques for using this information for prognosis. Current practice for quantifying fatigue life of rotating components of turbomachinery specifies life in units of operating time, or ìequivalent engine cycles.î No account is taken of the operating conditons. Nor is an attempt made to detect and track damage accumulating during operation. The new capability with blade-tip sensors promises to deliver improvements over this current operating procedure. This presentation will outline the prognostics capabilites promised by use of blade tip sensors, showing data from seeded fault tests. The presentation will outline the prognostics techniques in development. Finally, the presentation will illuminate the need for tighter linkage between this development work and existing expertise in fatigue life quantification.

Measurement and Interpretation of Internal/ Residual Stresses: Welding

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Shaping and Forming Committee

Program Organizers: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Bimal K. Kad, University of California, Ames Laboratory, La Jolla, CA 92093-0085 USA

Tuesday AM	Room: 17B
March 4, 2003	Location: San Diego Convention Center

Session Chairs: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Thomas Gnaupel-Herold, NIST Center for Neutron Research, Gaithersburg, MD 20899-8562 USA

8:30 AM Invited
Application of X-Ray and Neutron Residual Stress Mapping to Understand Cracking of Kraft Recovery Boiler Tubes and Air Ports: *Camden R. Hubbard*[1]; James R. Keiser[1]; Gorti B. Sarma[1]; Kimberly A. Choudhury[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., MS 6064, Bldg. 4515, Oak Ridge, TN 37830-6064 USA

Cracking of 304L stainless steel/carbon steel co-extruded tubes that form floors and walls in most recovery boilers is being observed with increasing frequency in North American boilers. The relationship among stresses in the floor, wall tubes and air port tubes to operating conditions and crack advance into the carbon steel is being investigated. Both X-ray and neutron diffraction and finite element modeling are being used to characterize the surface and through thickness residual stresses throughout boiler operation. The measured residual stresses have been used both as initial conditions for finite element models and for improving and validating the models. Other studies have evaluated the cracking resistance of several alloys that are alternatives to the conventionally used 304L stainless steel. Understanding of the air port forming process and design is underway to see if alternate materials and alternate forming methods can reduce the probability of cracking.

9:00 AM Invited
Phase-Resolved Residual Stresses Around a Weld in the Single Crystal Superalloy CMSX-4: *David Dye*[1]; Ronald B. Rogge[1]; Kelly T. Conlon[1]; Roger C. Reed[2]; [1]National Research Council of Canada, Neutron Prog. for Matls. Rsrch., Chalk River Labs., Chalk River, Ontario K0J 1J0 Canada; [2]University of British Columbia, Dept. Matls. & Metals Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

The repair welding of single crystal nickel-base Superalloy gas turbine blades is of great interest because it allows remediation of defects in large castings that would otherwise be scrapped. Sealing welds and blade tip repair are also being explored. Measurement of the full residual stress tensor in both the gamma and gamma-prime phases around an autogenous weld in a coupon of CMSX-4 is demonstrated, with rationalization of the results using a finite element model of the welding process. The measurements were performed using constant-wavelength neutron diffraction, and demonstrate that in the weld and heat-affected zone the principal tensile residual stress is in the radial direction, that the hoop stresses are compressive and that the shear stresses appear to be small. Cracking, when it occurs, occurs on {110}-type planes. Finally, the weld region appears to retain the orientation of the parent material.

9:30 AM
Neutron Diffraction Residual Stress Measurements on Single Crystal Superalloy Weld Joints: Ganjiang Feng[1]; [1]GE, 1 River Rd., 55-127, Schenectady, NY 12345 USA

Single crystal nickel-base superalloys are widely used in aircraft and advanced industry gas turbine components, which are often made by investment casting and exposed to high temperatures and cyclic loadings over extended periods of time. Due to the complexity of the turbine components, it is inevitable to employ joining processes during fabrication and repair. However, the single crystal alloys containing greater than 10% refractory elements are generally viewed as unweldable. Due to the high strength and the strong precipitation reaction of the single crystal superalloys, the weld joints are subjected to high residual stresses. These residual stresses often cause distortion and cracking of the weldments. To obtain a crack-free joint, the adverse effect of residual stresses and distortion must be kept within acceptable limits. Therefore, a thorough understanding of residual stress distribution is critical to develop successful joining processes for single crystal superalloys. In the past, a large portion of the work carried out on the residual stress distribution was accomplished by removing thin layers from the surface by chemical, electrolytic or mechanical methods. This will cause re-distribution of the stresses in the weldments. The purpose of the present investigation is to determine, by the neutron diffraction method, the internal residual stresses distribution throughout single crystal weld joint without removing surface layer. In addition, most of the research work conducted in the past has been concerned with equiaxial alloys. There is little work in the area of single crystal superalloys.

9:50 AM
Spatially Resolved Residual Stress Measurements in a Welded Specimen Using Neutron Diffraction: *Cecilia Larsson*[1]; T. M. Holden[2]; M. A.M. Bourke[2]; M. Stout[2]; L.-E. Lindgren[3]; [1]Linkoping University, Dept. of Mechl. Eng., Linkoping 581 83 Sweden; [2]Los Alamos National Laboratory, LANSCE/MST-8, Los Alamos, NM 87545 USA; [3]Lulea University of Technology and Dalarna University, Div. Compu. Aided Design, Lulea 97187 Sweden

Residual stress measurements were made on a tubular container, fabricated from a Co-based Haynes 25 alloy by welding two hemispherical end caps to either end of a cylinder. The measurements were motivated by concerns following long term exposure to heat of the container in a satellite application. Spatially resolved measurements were made using the recently commissioned Spectrometer for Materials Research at Temperature and Stress (SMARTS) at Los Alamos National Laboratory. By varying the orientation of the sample in the path of the incident neutron beam, residual strains were determined in the axial, hoop and radial directions as a function of distance from the centerline of the weld. Strains were determined by fitting single peaks in diffraction spectra as well as using entire spectra in Rietveld refinements. The maximum effective stress was determined to be around 200 MPa, well below the yield strength of the material (475 MPa). The results were compared with finite-element (FE) simulations and conclusions drawn on the effects of the welding process on the residual stress state.

10:10 AM Break

10:25 AM
How Type-2, Intergranular, Strains Affect the Interpretation of Measured Lattice Strains in Terms of Stress Fields in Engineering Components: *Thomas M. Holden*[1]; [1]Northern Stress Technologies, 5 Spring St., Deep River, Ontario K0J 1P0 Canada

Early neutron diffraction measurements[1] on bent steam generator tubes revealed a puzzling paradox. The apparent axial stress at the top of the bend was negative as measured with the aid of the (111) reflection of the f.c.c. structure but positive as measured with the (002) reflection at the same location. Further work with different materials and components showed such anomalies to be the rule rather than the exception. The origin of the anomalies lies in the superposition of the stresses on the length scale of the component, the type-1 stress fields, and those on the scale of the grain size, the intergranular effects. This superposition is also a contributory reason for the oscillatory anomalies seen in X-ray diffraction when strain is plotted as a function of the square of the sine of the angle of the scattering vector with respect to the surface normal. Strategies for correcting the measured lattice strains for intergranular effects are discussed for components fabricated from Inconel-600, zirconium alloys and beryllium. [1]T.M. Holden, R.A. Holt, G. Dolling, B.M. Powell and J.E Winegar, Met. Trans. A 19A (1988) 2207-14.

10:55 AM
Residual Stress Measurement with Focused Acoustic Waves and Direct Comparison with X-Ray Diffraction Stress Measurements: *Shamachary Sathish*[1]; Richard W. Martin[1]; Richard Reibel[1]; Thomas J. Moran[2]; [1]University of Dayton Research Institute, Structl. Integrity, 300 College Park, Dayton, OH 45469-0127 USA; [2]Metals, Ceramics and Nondestructive Evaluation Division, Matls. & Mfg. Direct., AFRL, WPAFB, Dayton, OH 45433 USA

The technique of measuring small changes in acoustic wave velocity due to external or internal stress have been used for quantitative determination of residual stress in materials during the last decade. Application of similar methodology with focused acoustic waves leads to residual stress measurement with spatial resolution of a few millimeters to a few microns in diameter. The high spatial resolution residual stress measurement requires development of new methodologies in both design of acoustic lenses and instrumentation for acoustic wave velocity determination. This paper presents two new methodologies developed for measurement of residual stress with spatial resolution of a few millimeters. The design of new type of acoustic lens for achieving higher spatial resolution in residual stress measurement is introduced. Development of instrumentation for high precision local surface wave velocity measurement will be presented. Residual stresses measured around a crack tip in a sample of Ti-6Al-4V; using focused beam will be compared with x-ray diffraction measurements performed on the same region of the sample. Results of residual stress measurements along a direction perpendicular to the electron beam weld in a sample of Ti-6Al-4V, determined using focused acoustic waves and x-ray diffraction technique are also presented. The spatial resolution and penetration depth of x-rays and focused acoustic beam with reference to residual stress measurements are discussed.

11:25 AM
X-Ray Residual Stress Measurements in 7020 Alloy Welded Joins: *Marzena Lech-Grega*[1]; [1]Institute of Non-Ferrous Metals, Light Metals Div., Pilsudskiego 19, Skawina 32-050 Poland

The study presents analyse of residual stresses in the around weld region of plates welded by TIG process using different welding currents in Al-Zn-Mg (7020) alloy. The residual stress by x-ray method have been measured. Dependence of welding process parameters and stress

TUESDAY AM

distribution was observed. The most changes in stress value in 25-40 mm zone from welded join axis have been observed and they correspond to the structure changes in this area.

11:45 AM
Analysis of the Residual Stress Distribution in API X65 Steel Welded Joint Using Advanced Indentation Technique: *Yun-Hee Lee*[1]; Dongil Son[1]; Yeol Choi[1]; Won-jae Ji[1]; Jang-Bog Ju[1]; Dongil Kwon[1]; [1]Seoul National University, Sch. of Matls. Sci. & Eng., Shinrim-Dong, Kwanak-Gu, Seoul 151-742 S. Korea

Residual stress in steel welded joint reduces the remnant lifetime of the in-field structure by degrading the fatigue and fracture properties. However, the stress evaluation is very difficult due to severe gradient of complex microstructures and mechanical properties in the welded region. Therefore, a nondestructive and local mechanical testing technique is needed. The advanced indentation technique, which has been developed for testing in local region, was used for the characterization of the welded joint. The indentation load-depth curve as the result of the test was very sensitive to the signal and the strength of the residual stress. Furthermore, the interaction between indentation stress and residual stress can be theoretically modeled. Finally, a stress-analysis equation based on the curve shift and the stress interaction parameter was proposed. The advanced indentation technique was applied to the characterization of a API X65 steel welded joint for the natural gas transmission pipeline. The evaluated stress distribution was compared with that from the saw cutting method.

Microstructural Processes in Irradiated Materials: Multiscale Modeling I

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

Tuesday AM — Room: 11A
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Robert Odette, University of California-Santa Barbara, Dept. of Mechl. Eng. & Environl. Eng., Santa Barbara, CA 93106 USA; StÈphanie Jumel, ElectricitÈ de France, MMC, Moret sur Loing 77818 France

8:30 AM
Multiscale Modeling of Irradiation Embrittlment: *G. Robert Odette*[1]; [1]University of California-Santa Barbara, Dept. of Mechl. & Environl. Eng., Santa Barbara, CA 93106 USA

Development of a comprehensive-integrated multiscale-multiphysics model of irradiation embrittlement is reviewed. Key model components include: defect production including local migration and interactions of cascade species leading to generation of cluster complexes and migrating defects - the products of long-term cascade aging; long-range diffusion of defects and solutes including trapping and in the presence of an evolving microstructure; defects and solutes annihilation and clustering; between dislocations interactions with defect complex and precipitates; hardening superposition of the irradiation-mediated and pre-existing dislocation obstacles; shifts in the toughness temperature master curve. The model provides quantitative predictions of the combined effects of irradiation (flux, fluence, temperature) and material (composition, microstructure, starting properties) variables. A detailed description of how the components of a hierarchical model are linked and the computational techniques are given. Finally, verification and calibration of the model with the enormous Irradiation Variables experiment data base are outlined.

9:15 AM
Interaction of Interstitial Clusters with Impurities and a Dynamical Phase Diagram of Microstructural Evolution of Irradiated Materials: Toby S. Hudson[1]; Maria-Jose J. Caturla[2]; Sergei L. Dudarev[3]; Adrian P. Sutton[1]; [1]Oxford University, Matls. Dept., Parks Rd., Oxford, Oxfordshire OX1 3PH UK; [2]Lawrence Livermore National Laboratory, L-353, Livermore, CA 94550 USA; [3]EURATOM/UKAEA Fusion Association, Theory & Modlg. Dept., Culham Sci. Ctr., Abingdon, Oxfordshire OX14 3DB UK

We discuss the role impurities play in the microstructural evolution of irradiated materials.† By including elastic interactions in a kinetic Monte Carlo simulation, we show trapping of one dimensionally gliding interstitial clusters both near single impurities, and in between the fields of two impurities.†Our model also allows for infrequent changes in the Burgers vector of these clusters, sometimes allowing them to escape such trapping and giving rise to three-dimensional diffusional transport. A dynamical phase diagram is constructed in order to determine under what conditions and how this trapping affects the overall microstructural evolution.†The difference between the microstructural evolution of a system with impurities with positive and negative relaxation volumes is discussed.

9:35 AM
Dynamic Properties of Edge Dislocations Decorated by Interstitial Loops in a-Fe: *Yuri N. Osetsky*[1]; David J. Bacon[1]; Bachu N. Singh[2]; [1]The University of Liverpool, Dept. of Eng., Brownlow Hill, Liverpool L69 3GH UK; [2]Risoe National Laboratory, Matls. Dept., PO Box 49, Roskilde DK-4000 Denmark

Fast thermally-activated one-dimensional glide of clusters of self-interstitial atoms (SIAs) has been observed by atomistic simulations in a variety of bcc, fcc and hcp metals. In bcc iron, for example, all SIA clusters produced in high-energy displacement cascades are glissile. This phenomenon may contribute directly to the microstructure evolution through creation of a high vacancy supersaturation due to absorption of glissile SIA clusters at sinks such as grain boundaries and dislocations, and may be responsible for the formation of specific microstructures, e.g. microvoids, void lattice, rafts of dislocation loops and decoration of dislocations by clusters and dislocation loops. The dislocation decoration is believed to play an important role in localisation of plastic deformation in irradiated materials. Possible effects have been studied recently within the framework of cascade-induced source hardening model using the isotropic elasticity theory. However, some features of the cluster-dislocation interactions cannot be described in terms of elasticity theory. It is, on the other hand, important to understand the details of these interactions since they might affect the behaviour of decorated dislocations. In this paper we present results of atomistic simulation of interactions between a moving dislocation and decoration formed by small interstitial loops with the same Burgers vector b. We have simulated an edge dislocation with b =Ω<111> gliding in {110} plane and sets of SIA clusters with size in the range 0.5-2.5nm distributed below the extra half-plane, using a many-body interatomic potential for bcc iron. The results obtained and the pinning effect of the decoration are discussed from the point of view of the cascade-induced source hardening model.

9:55 AM Break

10:25 AM
Kinetic Monte Carlo Simulations of Precipitation Under Irradiation: *FrÈdÈric Soisson*[1]; [1]CEA Saclay, DMN/SRMP, Gif-sur-Yvette 91191 France

The phase transformation kinetics in alloys under irradiation are now widely studied using various Monte Carlo techniques. The simulations we use are based on an atomistic description of the diffusion events. In previous studies (e.g. in the case of copper precipitation in iron) we have shown how both the details of these mechanisms and the corresponding migration barriers control the kinetic pathway of phase transformations during thermal ageing. Here we present some results concerning precipitation under irradiation, with simulations including vacancy-atom exchanges, dumbbell migration and ballistic replacement due to displacement cascades.

10:45 AM
A Self-Consistent Mean-Field Theory for the Kinetics of Segregation: From Equilibrium to Irradiation-Induced Steady States: *Maylise Nastar*[1]; [1]CEA/Saclay, DEN/DMN/SRMP, Bat. 520, Gif-sur-Yvette 91191 France

Segregation under irradiation is due to a permanent flow of vacancies and interstitials towards point defect sinks like surfaces and interfaces. A model based on a mean field lattice rate theory has already been proposed to simulate segregation in irradiated austenitic steels[1]. We complement this model with a self-consistent treatment of the mean field kinetic equations which reproduces in a natural way the up to now neglected correlation effects inherent to the vacancy diffusion mechanism[2]. Then both kinetics and thermodynamics are treated in a mutually consistent way. In particular the equilibrium properties are taken into account and we show how they contribute to the details of irradiation induced segregation profiles, e.g. the W shaped profiles. The role of the correlation effects on the kinetics and on the steady state of radiation induced segregation is discussed. [1]M. Nastar, P. Bellon, G. Martin, and J. Ruste, Phase Transformations and Systems Far from

Equilibrium, Materials Research Society Symposium Proceedings, Vol. 481, edited by E. Ma, P. Bellon, M. Atzmon and R. Trivedi (Pittsburgh, Pennsylvania Materials Research Society) p. 383, (1997). [2]M. Nastar, V. Yu Dobretsov, and G. Martin, Phil. Mag. A, Vol. 80, p.155, (2000).

11:05 AM
Modeling Cascade Aging and the Formation of Vacancy-Solute Clusters in Fe-Based Alloys: *Brian D. Wirth*[1]; Jaime Marian[1]; Eduardo M. Bringa[1]; G. Robert Odette[2]; [1]Lawrence Livermore National Laboratory, MSTD/CMS, PO Box 808, L-353, Livermore, CA 94550 USA; [2]University of California-Santa Barbara, Mechl. Eng., Santa Barbara, CA 93106 USA

Ferritic steels represent a technologically important class of materials that are widely used in current nuclear fission reactors and proposed as candidate materials for plasma-facing structures in future fusion energy facilities. Predicting their in-service performance requires a detailed understanding of the mechanisms of defect accumulation and microstructure evolution in harsh radiation environments. The physical processes involved in radiation damage are inherently multiscale, spanning more than 15 orders of magnitude in length and 24 orders of magnitude in time. In this talk, we describe multiscale modeling to predict the aging of primary defects produced in displacement cascades. The results illustrate the mechanisms responsible for the formation of i) vacancy-solute clusters which are the so-called matrix defect features in reactor pressure vessel steels, ii) <100> dislocation loops observed in ferritic steels irradiated to high dose and iii) vacancy-He clusters which serve as Helium bubble and void nuclei in fusion reactor materials.

11:25 AM
Migration and Directional Change of Interstitial Clusters in a-Fe: Searching for Transition States by the Dimer Method: *Fei Gao*[1]; Heinisch Howard[1]; Kurtz J. Richard[1]; Yuri N. Osetsky[2]; William J. Weber[1]; Richard G. Hoagland[1]; [1]Pacific Northwest National Laboratory, PO Box 999, MSIN K8-93, Richland, WA 99352 USA; [2]The University of Liverpool, Matls. Sci. & Eng., Dept. of Eng., Liverpool L69 3GH UK

Interstitial clusters produced by displacement cascades in metals exhibit thermally activated, one-dimensional glide. Only small interstitial clusters are observed to change their glide direction during the period of MD simulations. In order to overcome the ìtime barrierî in MD simulation, the dimer method is employed to search for possible transition states of interstitial clusters in a-Fe. The lowest energy barriers correspond to defect migration along <111> directions. Small clusters change their direction by a <110> dumbbell mechanism, whereas the directional change for larger clusters is a two-step process consisting of translation along a <100> direction and rotation into an equivalent <111> direction. These mechanisms are also investigated using long-time-scale MD simulations. This paper also addresses whether migrating interstitial clusters can change their directions by thermal activation under stress. The results are discussed in terms of modeling the long-time-scale dynamics of defect evolution under irradiation.

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - III

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Alloy Phases Committee
Program Organizers: Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Michael R. Notis, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18015 USA; Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA

Tuesday AM — Room: 12
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: C. Robert Kao, National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 32054 Taiwan; Suzanne E. Mohney, Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA

8:30 AM Invited
Electromigration Induced Phase Change in Solder Joints and Lines: *K. N. Tu*[1]; Gu Xu[1]; Hua Gan[1]; W. J. Choi[1]; [1]University of California-Los Angeles, Dept. of Matls. Sci. & Eng., Los Angeles, CA 90095-1595 USA

Electromigration induced phase change in solder joints is unique due to eutectic alloy composition. In a eutectic alloy below the eutectic temperature, there is no chemical potential gradient as a function of composition at constant temperature and pressure. For example, there is no interdiffusion or homogenization between 70Pb30Sn and 30Pb70Sn in a diffusion couple annealed below the eutectic temperature, except ripening. Hence, electromigration can induce a very large compositional gradient in the eutectic SnPb alloy by driving Sn (near room temperature) or Pb (near 150∞C) from cathode to anode. In the up-hill diffusion, there is no reverse atomic flux to oppose electromigration due to the large concentration gradient induced by electromigration. It is different from the classic Soret effect. This eutectic effect may explain why a large amount of Cu or Ni can be dissolved from the UBM at cathode and driven to anode to from intermetallic compounds. This is because Sn-Cu and Sn-Ni also form eutectic structures of Sn and intermetallic compounds. In this talk, experimental observations of the eutectic effect will be presented.

8:50 AM Invited
Electromigration Effects upon the Low-Temperature Sn/Ni Interfacial Reactions: *Sinn-wen Chen*[1]; Chih-ming Chen[1]; [1]National Tsing-Hua University, Dept. of Cheml. Eng., Hsin-Chu 300 Taiwan

The Sn/Ni interfacial reactions at 100°C with and without the influence of passing through electric currents were studied. The intermetallic products include plate-like $NiSn_3$ and continuous Ni_3Sn_4 layer. The formation site of the $NiSn_3$ phase at the Sn/Ni interface is random, and its growth rate is far faster than that of the Ni_3Sn_4 phase. The growth of the $NiSn_3$ phase is controlled by the diffusion of the constituent Ni, and grain boundary diffusion plays an important role. Electromigration caused by an electric current of 4◊10^{3}A/cm^2 density has a significant effect on the $NiSn_3$ phase growth.

9:10 AM Invited
Electromigration Studies of Sn96.5/Ag3.5 Flip Chip Solder Bumps: *I. H. Chen*[1]; T. L. Shao[1]; Chih Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Eng., 1001 Ta Hsueh Rd., Hsinchu 30050 Taiwan

With environmental concerns increasing, the microelectronics industry is paying much more attention on lead-free solder alternatives. One of the most popular alternatives is Sn96.5/Ag3.5 for flip chip package. Electromigration damage of Sn96.5/Ag3.5 solder bumps was studied. Lead-free Sn96.5/Ag3.5 solder bumps were formed on a chip after printing and reflowing process. The under-bump metallization (UBM) on the chip side was Cr/Cr-Cu/Cu, and the metallurgy layer on the substrate side was Ni/Au. The chip was mounted on a BT substrate and then filled with underfill. The bumps were stressed at the current density of 2 * 10E4 A/cm^2 at 100°C. Whiskers and hillocks were found on the anode side, while voids were observed on the cathode side. Redistribution of atoms was examined, and composition change of intermetallic compounds was monitored. The failure mechanism of the bump after current stressing was discussed.

9:30 AM
TEM Study on the Interfacial Reaction Between Electroless Plated Ni-P/Au UBM and Sn-3.5Ag Solder for Flip Chip Package: *Eun-Jeong Kwon*[1]; Chul-Woong Yang[1]; Seung-Boo Jung[1]; Jun-Seok Ha[2]; Hee-Kwang Kang[1]; Jong-Kwon Choi[1]; [1]SungKyunKwan University, Dept. of the Adv. Matls. Eng., 300 Chonchondong, Jangan, Suwon, Kyunggi-do 440-746 S. Korea; [2]LG Electronics, Inc., 16, Woomyeong, Seoul, Seocho 137-724 Korea

Electroless Ni-P/Au deposition has attracted attention in chip package because of its simplicity of process, low cost and many good properties. But, it has not been still understood the mechanism of interfacial reaction during reflow and the information of interfacial compounds which is formed at solder joint with these two IMCs, although it seems that various Ni-Sn IMCs and Ni-P IMCs exist at the interface after soldering. Therefore, we studied on the interfacial reaction between electroless plated Ni-P/Au UBM and eutectic Sn-3.5Ag solder by using XRD, SEM, and TEM. Especially, chemical and crystallographic analysis using TEM provided us very consequential informations about microstructure of the interface. In this study, UBM is prepared by the electroless plating of Au/Ni-15at%P on Cu substrate, and then it is reacted with Sn-3.5Ag eutectic solder at 260°… for various times to examine different sequential stage of the interfacial reaction.

9:45 AM
Intermetallic Compound Growth on Cu and Electroless Ni-P/Cu Substrates with Sn-5Bi-3.5Ag Solder: *Jeong-Won Yoon*[1]; Chang-Bae Lee[2]; Seung-Boo Jung[1]; [1]SungKyunKwan University, Adv. Matls. & Process Rsrch. Ctr. for IT, 300 Chunchun-dong, Changan-gu, Suwon, Gyounggi-do 440-746 Korea; [2]Osaka University, Grad. Sch. of Eng., Suita, Osaka 565-0871 Japan

The growth kinetics of intermetallic compound layers formed between Sn-5Bi-3.5Ag solder and (Cu, electroless Ni-P/Cu) substrates were investigated at temperature between 70° and 200° for 0 to 60 days. The duplex structure of both Cu6Sn5 and Cu3Sn intermetallics existed in the couple of the Sn-5Bi-3.5Ag/Cu system, whereas the only normal Ni3Sn4 intermetallic was found on the electroless Ni-P/Cu substrate. The layer growth of intermetallic compounds satisfied the parabolic law at given temperature range. As a whole, because the values of time exponent(n) have approximately 0.5, the layer growth of the intermetallic compounds was mainly controlled by diffusion mechanism. The layer growth rate of the Ni3Sn4 intermetallic was much slower than that of the Cu-Sn intermetallic. The Ni-P deposit was a very good diffusion barrier for the Sn-5Bi-3.5Ag solder.

10:00 AM Break

10:25 AM Invited
Solid-State Aging Reactions Between Ni and SnCu Lead-Free Solders with Different Cu Concentrations: W. T. Chen[1]; R. Y. Tsai[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 32054 Taiwan

The eutectic Sn-0.7Cu solder is considered a very promising lead-free replacement for the Sn-37Pb solder, especially for wave soldering applications. For industrial uses, a 0.2% uncertainty in composition is generally considered acceptable. In other words, for the Sn-0.7Cu solder, the Cu concentration can range from 0.5 to 0.9 weight percentage. However, our recent study revealed that, during soldering, the Cu concentration had a very strong effect on the reactions between Sn-Cu solders and Ni. With increasing Cu concentration in solder, the reaction product at the interface after reflow changed from a Ni3Sn4-based compound to a Cu6Sn5-based compound. In other words, during soldering, the Cu concentration must be strictly controlled in order to obtain consistent results. In this study, we would like to extend our earlier study to investigate whether this strong concentration dependency also occurs during the solid-state aging of the solder joints. We aged solder joints at the solid-state at several different temperatures for time as long as 1000 hours. The solder compositions studied include Sn-0.2Cu, Sn-0.3Cu, Sn-0.4Cu, Sn-0.5Cu, Sn-0.6Cu, Sn-0.7Cu, and Sn-1.0Cu. Analysis techniques used include optical microscope, SEM, EPMA, and XRD. It turned out that after solid-state aging, the dependence in Cu concentration disappeared. Detailed mechanism will be presented for this interesting phenomenon.

10:45 AM Invited
Intermetallic Formation Between Lead Free Solders and Ag-Pd Metallizations: Gaurav Sharma[1]; Chad Eichfeld[1]; *Suzanne E. Mohney*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA

We will describe the intermetallic formation between Ag-Sn and Ag-Sn-Cu solders and Ag-Pd metallizations (including pure Pd). The Ag-Pd metallization is of interest since it is used in low temperature co-fired ceramic packaging. Prolonged use of such packages at elevated temperature will likely affect the solder joints; therefore, we have studied solid state diffusion couples in these systems. Intermetallic growth was more rapid for solders on Pd compared to Ag-Pd metallizations. For the Ag-Pd metallizations, the resulting intermetallic layers were found to grow with the square root of time between 1 and 16 days, and the rate of intermetallic growth increased rapidly with temperature. For example, 59 microns of reaction product were observed after 16 days at 176∞C, whereas only 6 microns were found after 18 days at 125∞C. Rate constants and activation energies as well as the identity of the intermetallic phases will be reported.

11:05 AM Invited
Stress Evolution Related with Phase Transformation During Solid State Reaction of SnAg: J. Y. Song[1]; *Jin Yu*[1]; [1]Korea Advanced Institute of Science and Technology, Ctr. for Elect. Pkgg. Matls., 373-1 Guseong-dong Yuseong-gu, Daejon 305-701 S. Korea

In microelectronics packaging, recent trends to eliminate Pb from solders triggered intensive research on the processing method, reliability and economic issues of the Pb-free interconnection. Electroless Ni plating has been widely used as an under-bump-metallization(UBM) layer for a long time, however there has been little studies on the stress evolutions in the UBM layer during the solder reflow process. In this study, stresses in the electroless Ni UBM/electroplated SnAg solder layer due to the amorphous-to-crystalline phase transformation and the intermetallic formation were measured in-situ using laser curvature method during the solid state reaction at 200°C. The tensile stress developed and increased with time as Ni3Sn4 and Ag3Sn formed at the UBM/solder interfaces. Similarly, kinetics of phase transformation were analyzed from the stressYu evolutions during the solid state reaction, and compared with those of previous works. Finally, effects of Sn and Sn-Ag solder on the stress evolution and phase transformation of the underlying Ni layer were investigated.

11:25 AM
Studies of Interfacial Reaction Between Cu Particles Reinforced SnAg Solder and Ni Thin Film UBM: *S. J. Wang*[1]; C. Y. Liu[2]; [1]National Central University, Cheml. & Matls. Eng., No. 300, Jung-da Rd., Jung-li City, Taoyuan 320 Taiwan; [2]National Central University, Cheml. & Matls. Eng., No. 300, Jung-da Rd., Chung-Li, TaoYuang 320 Taiwan

It has been pointed out that the Cu particles reinforced SnAg composite solder has excellent mechanical properties. To use this composite solder as C4 (Controlled Collapse Chip Connections) solder bumps, we have to understand the interfacial reaction between composite solder and C4 UBM (Under Bump Metallization). Currently, we notice that Ni thin film is one of most common UBM for C4 solder joint. In this paper, we have studied the wetting reaction between Cu particles reinforced SnAg Pb-free solder and Ni thin film UBM. We will report the compound formation and kinetics of interfacial compound growth with varying thickness of Ni thin film, which are 500≈, 1000 ≈, and 2000 ≈. The preliminary results showed that Cu-Sn compound formed on the surface of Ni thin UBM, which can retard the reaction between Ni UBM and SnAg solder. No spalling was found in all Ni thin UBM with different Ni film thickness. The possible mechanism of spalling prevention will be proposed during this talk.

11:40 AM
Electromigration in SnAg3.8Cu0.7 Solder Joints for Flip Chip Technology: *Ying-Chao Hsu*[1]; T. L. Shao[1]; Chih Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Eng., 1001 Ta Hsueh Rd., Hsinchu 300 Taiwan

Lead-free SnAg3.8Cu0.7 solder will be widely used in microelectronic packaging industry due to good fatigue resistance and high joint strength. Electromigration in SnAg3.8Cu0.7 solder was investigated for flip chip solder bump structure. An under-bump metallization (UBM) of Cr-Cu/Cu/Au tri-layer was deposited on the chip side and electroless Ni/Au pad was deposited on the BT board side. Electromigration damage was observed under the current density of 2*10^4 A/cm2 at 100°C. Voids were found at cathode side and crack was observed at solder/thin film UBM interface after current stressing. Copper and nickel atoms were found to move in the direction of electron flow. Intermetallic compounds of Cu-Sn and Ni-Cu-Sn were also observed to spread into the solder bump due to current stressing.

Science and Technology of Magnetic and Electronic Nanostructures: Bionanoelectronics

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Ramamoorthy Ramesh, University of Maryland, Department of Materials and Nuclear Engineering, College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Tuesday AM Room: 15A
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Ramamoorthy Ramesh, University of Maryland, Dept. of Matls. & Nucl. Eng., College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin, Dept. of Matls. Sci. & Eng., Madison, WI 53706 USA

8:30 AM
Molecular NanoElectronics: *R. Stanley Williams*[1]; [1]Hewlett-Packard Laboratories, 1501 Page Mill Rd., MS 1123, Palo Alto, CA 94304 USA

Economic and general physics considerations indicate that the rapid improvements we have come to expect in silicon integrated circuits may saturate around the year 2010. However, fundamental physical laws indicate that it should be possible to compute with a power efficiency that is at least one billion times better than present silicon electronics. The most straightforward way currently known to achieve such efficiencies are to fabricate circuits very much smaller than they are at present. Thus, there is a tremendous business incentive to invent new electronic devices and circuits that will have dimensions of the order of nanometers. In addition, new fabrication techniques will be required that can inexpensively produce and connect these devices in vast quantities. In order to satisfy both requirements simultaneously, we have assembled a trans-disciplinary team of chemists, physicists, engineers and computer scientists at HP Labs to explore the use of molecules as active electronic devices.

9:15 AM

Nanoscale Molecular Electronic Circuits: *Yong Chen*[1]; [1]Hewlett-Packard Laboratories, 1501 Page Mill Rd., MS 1123, Palo Alto, CA 94304 USA

Within the next two decades the miniaturization of Si-based microelectronic circuits will gradually approach its scientific, technical, and economic limits. Meanwhile recent advances in molecular electronics has led to considerable promise of scaling circuits further down at nanometer scale due to the inherently small nature of molecule. In our lab, we have used molecules with bistable electronic properties to fabricate high-density memory circuits. The metal electrodes were fabricated by imprinting lithography at sub-50 nm scale. Langmuir-Blodgett molecular thin films with switchable electronic properties were sandwiched between the nanoscale metal-electrodes. Bistable electronic statues with high on-off ratio and reversible switching properties have been observed in these devices. With the nanoscale devices, fully functional high-density memory circuits were fabricated and demonstrated. The design of molecules, devices, and their circuit applications will be discussed.

10:00 AM

Molecule Cascades for Computation: *Andreas J. Heinrich*[1]; Christopher P. Lutz[1]; Jay A. Gupta[1]; Donald M. Eigler[1]; [1]IBM Almaden Research Center, 650 Harry Rd., D1, San Jose, CA 95120 USA

Molecule cascades provide new opportunities to study and utilize the controlled motion of adsorbates on surfaces. The motion of one CO molecule on a Cu (111) surface causes a nearby molecule to hop to a new site, which in turn moves another molecule, and so on in a cascade of motion similar to a row of toppling dominoes. By combining STM atom-manipulation and in-situ isotope selection we study the hopping mechanism of such molecule cascades. We find the surprising result that entire CO molecules hop by quantum tunneling at temperatures between 0.5 K and 6 K! An interesting application of molecule cascades is computation on the nanometer length scale. We show that molecule cascades can provide all of the devices and interconnects required for the one-time computation of an arbitrary logic function.

10:45 AM

Biomolecular Approaches to Nanostructure Assembly: *Anand Jagota*[1]; [1]DuPont, CR&D, E356/317A, Experimental Sta., Wilmington, DE 19880-0356 USA

This talk will describe work in our group on novel uses of biological molecules for assembly of nanostructures with application to nanoelectronic devices. Specifically, we will describe a family of peptides that bind to carbon nanotubes. These have been discovered by application of the phage display technique; binding has been verified independently. This finding offers a way by which carbon nanotubes can be functionalized by biological molecules, opening the possibilities of separation, handling, and placement. We will also describe the construction of DNA-nanoparticle assemblies where we can demonstrate remarkable control on the structure by prior control on nanoparticle size, non-specific binding, and ligand number. We will also report on nanoparticle assemblies that make use of specific protein-DNA interactions. Finally, we show how microtubules can be used as scaffolds and templates to construct conducting rods. Microtubules, which are composed of repeating units of alpha and beta tubulin, form a high aspect ratio tube about 25 nm in diameter and up to tens of microns long. We will describe techniques for metallization of the microtubule before or after immobilization on a substrate, resulting in conducting nanorods or wires.

Surface Engineering in Materials Science - II: Surface Engineering and Modifaction

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Surface Engineering Committee

Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA; A. Agarwal, Plasma Processes, Inc., Huntsville, AL 25811-1558 USA; Narendra B. Dahotre, University of Tennessee-Knoxville, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; C. Suryanarayana, University of Central Florida, Mechanical, Materials & Aerospace Engineering, Orlando, FL 32816 USA

Tuesday AM — Room: 7A
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: N. B. Dahotre, University of Tennessee-Knoxville, Dept. of Matls. Sci. & Eng., Knoxville, TN 37932 USA; J. Majumdar, University of Michigan, Dept. of Mechl. Eng. & Matls. Sci. & Eng., Ann Arbor, MI 48109-2125 USA

8:30 AM Invited

Processing, Microstructure and Wear Properties of Ti-Base Alloys Strengthened: *Jeff Th.M. de Hosson*[1]; [1]University of Groningen, Dept. of Appl. Physics, Matls. Sci. Ctr. & the Netherlands Inst. for Metals Rsrch., Nijenborgh 4, Groningen 9747 AG The Netherlands

In this study the Laser Melt Injection (LMI) process is explored to create a Metal Matrix Composite (MMC) consisting of 80 µm sized WC particles embedded in the top layer of a Ti-6Al-4V alloy. In particular the influences of the principal process parameters, e.g. power density, scanning speed and power flow rate, on the dimensions of the laser track and microstructural features are examined. An important finding is that the particle distribution is homogeneous and that the particles are injected over the whole depth and whole width of the melt pool. The microstructure was investigated by advanced transmission electron microscopy including energy filtering techniques and scanning electron microscopy with an integrated Electron Back-Scatter Diffraction/Orientation Imaging Microscopy (OIM). In the resolidified Ti-alloy melt pool TiC dendrites and W grains are found. Occasionally a crystal orientation relation between WC, W2C and TiC is observed, depending on the WC interface.

9:00 AM

Silicon Carbide Coating on Ti-6Al-4V Alloy Using Laser Surface Modification: *Abhijeet Prakash Joshi*[1]; Mario F. Arenas[1]; Ramana G. Reddy[1]; [1]University of Alabama, Metall. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

Ti-6Al-4V is an alloy with a wide range of applications in the aviation and space industries due to its excellent properties such as low density, high melting point, high tensile strength, good corrosion resistance and formability. However, it suffers from poor wear resistance and has a high friction coefficient, limiting its industrial widespread use. Laser surface modification provides an excellent approach for improving wear resistance. In this study, experiments were carried out using pulse Nd-YAG laser to induce melting and reaction of a powder SiC placed on the surface of the Ti-6Al-4V alloy. The experiments were performed in an inert atmosphere of argon gas. A dispersively reinforced SiC coating was formed on the surface of Ti-6Al-4V alloy. Optical microscopy, X-ray diffractometery (XRD), scanning electron microscopy (SEM), and hardness testing were used to characterize the coating formed. Results showed an improvement in wear resistance.

9:20 AM

The Effects of Phosphate Treatment on Surface Friction and Coating Delamination of Galvannealed Steel: *Doojin Paik*[1]; Gary M. Michal[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., 10900 Euclid Ave., Cleveland, OH 44106-7204 USA

Phosphate coatings on the surface of a metal during a forming operation are known to reduce surface friction. Recently phosphate has been applied to the surface of galvanneal coatings on highly formable steel sheet substrates. Such phosphate treatments have been found to both decrease surface friction and increase the amount of galvanneal coating delamination that occurs during a forming operation. To explore a possible relationship among coating delamination, surface friction and the presence of phosphate the deformation states of drawing, plane strain and biaxial stretching were applied to candidate samples.

Coating delamination was found to occur only under conditions of compressive strain. The presence of cracks in the galvanneal coatings prior to, and due to, deformation was investigated using SEM. Finally, a model was developed that takes into account the role of phosphate, preexisting cracks and how reduced surface friction can promote galvanneal coating delamination by a wedging mechanism.

9:40 AM

Structures Induced by Carbon Implantation and Annealing of Tetrahedral Carbon: *Chun Wing Lam*[1]; Igor Bello[2]; Yeshayahu Lifshitz[2]; O. Kutsay[2]; W. Y. Luk[2]; S. T. Lee[2]; X. Meng[2]; [1]City University of Hong Kong, Ctr. of Super-Diamond & Adv. Films (COSDAF) & Dept. of Physics & Matl. Sci., 83 Tat Chee Ave., Kowloon, Hong Kong China; [2]City University of Hong Kong, Hong Kong China

100 nm thick tetrahedral amorphous carbon (ta-C) films were deposited by filtered cathodic vacuum arc (FCVA). The ta-C films were then implanted with 50 keV carbon ion beam to doses ranging from 3◊10^{13} to 3◊10^{17} cm^{-2} using metal vapor vacuum arc (MEVVA) ion source. The implanted films were annealed at various temperatures in order to form novel nanostructures being investigated upon the ion exposure with reference to the films as deposited and annealed. Considerable structural reconstruction occurred above a dose threshold of 3◊10^{15} cm^{-2} as indicated by Raman. Transmission electron diffraction (TED) patterns collected from the samples exposed to higher doses (above 3◊10^{16} cm^{-2}) indicate a significant degree of preferred orientation. In correlation, electron energy loss spectroscopy (EELS) revealed the localized increase in sp^2 carbon bonding over the film cross-section while high resolution electron transmission spectroscopy (HRTEM) disclosed characteristic formation of graphitic basal planes preferably along the ion tracks.

10:00 AM Break

10:20 AM

Pulsed Laser Deposited Coatings for Hydrogen and Hydrogen Isotope Permeation Resistance: *Thad M. Adams*[1]; James Fitz-Gerald[2]; [1]Westinghouse Savannah River Company, Savannah River Tech Ctr., Bldg. 773-41A/151, Aiken, SC 29808 USA; [2]University of Virginia, Matls. Sci. & Eng., 116 Engineerís Way, Charlottesville, VA 22904 USA

The permeation of hydrogen and hydrogen isotopes is a critical issue in the operation of tritium extraction facilities and fusion reactors. To date several coating materials and materials systems have been investigate to reduce the permeation of hydrogen and hydrogen isotopes through common structural materials such as stainless steels. These materials include oxide ceramics, thermally grown oxides, metals, intermetallics, and amorphous systems. Permeation response for these coatings system has also been shown to be highly dependent on application techniques-thermal spray, CVD, EBPVD- and resulting microstructural features. The current research is focused on three coatings systems applied using pulsed laser deposition combined with laser surface modification and alloying. 304L and 316L stainless steel substrates have been coated with tungsten, aluminum oxide, chromium oxide, and Al-Fe-Ni-Nd amorphous alloy using PLD. Laser modification of the substrate has been performed in an attempt to enhance coating adhesion. Following characterization of the coating-substrate system using a field emission gun SEM, X-ray diffraction, and Auger electron spectroscopy, hydrogen-charging experiments have been carried out at 350∞C for 14 days. Additional testing has been conducted to evaluate coating behavior under thermal cycling and also under a high temperature (500∞C) humid environment.

10:40 AM

Surface Modification of Graphitic Foam: *Sharmila M. Mukhopadhyay*[1]; Erik Ripberger[2]; Ajit K. Roy[2]; Indy Roca[3]; R. Pulikollu[3]; [1]Wright State University, Mechl. & Matls. Eng., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, WPAFB, OH 45433 USA; [3]Wright State University, Dayton, OH 45435 USA

Possible surface modification methods to improve the functionality of micro-cellular graphitic foams have been investigated. This solid has a complex morphology made up of graphitic planes, edges, and nanometer size structures. Since it has over 80% open interconnected porosity, the surface area per unit volume is high. This makes surface engineering the best route for targeted property modification. This paper will discuss the influence of liquid solutions and plasma coatings on foam surfaces. Surface spectroscopy studies indicate that surface oxygen fraction can be enhanced by some of these treatments, which can result in enhanced infiltration of matrix materials such as epoxy resins. Water uptake of the foam is increased by such treatments, as is the strength of foam-epoxy composites. Other treatments can result in the opposite effect, i.e. induce surface inertness, which is useful for durability of stand-alone foam structures. These results have been analyzed in light of the current state of knowledge in surface modified graphitic fibers.

11:00 AM

Single-Crystal Laser Cladding of Superalloys: Influence of Preheating on the Microstructure: *Cyrille BezenÁon*[1]; Matthias Hoebel[2]; Jean-Daniel WagniËre[1]; Wilfried Kurz[1]; [1]EPFL, CTML, CP 110, Lausanne CH-1015 Switzerland; [2]ALSTOM Power Technology, Baden CH-5401 Switzerland

The oxidation resistance of a single-crystalline Ni-base superalloy can be improved by the application of a protective layer of NiCrAlY alloy. However, current processes such as plasma spraying, produce a polycrystalline layer leading to a reduced thermo-mechanical fatigue strength of the component, due to the difference in E-modulus between clad and substrate. Therefore, a new processing allowing the deposition of a single-crystal oxidation resistant coating has been developed. In this process, the alloy powder is melted by a high intensity laser beam and solidified epitaxially onto the single crystal substrate. For a successful single-crystal deposition, nucleation and growth of misorientated grains such as stray grains must be avoided. In this paper, the influence of the substrate preheating temperature on the formation of stray crystals is experimentally determined using Electron Back-Scattering Diffraction (EBSD). It is shown that with a preheating temperature of 750∞C, nucleation of misoriented grains o ccurs in the remelted zone of the substrate and develops, into a columnar mode in the NiCrAlY layer. Processing without preheating avoids nucleation of new grains and leads to single-crystal deposition. The experimental observations are discussed with the aid of an analytical model of the columnar-to-equiaxed transition (CET).

11:20 AM

Laser Surface Modification and Cell Adhesion onto Titanium Surfaces: Steve Mwenifumbo[1]; N. Morris[2]; M. Li[3]; A. Rodriguez[2]; Seyed Allameh[1]; *W. O. Soboyejo*[1]; [1]Princeton University, Princeton Matls. Inst., D404 E. Quad., Princeton, NJ 08544 USA; [2]Princeton University, Electl. Eng., Princeton, NJ 08544 USA; [3]Spectra Physics Lasers, 1305 Tera-Bella Ave., Mountainview, CA 94043 USA

This paper presents the results of a study of cell adhesion onto Excimer laser micro-grooved surfaces. Following a brief review of the laser processing parameters required for optimum micro-groove processing, the paper explores the interactions between human osteosarcoma (HOS) cells and laser-grooved Ti-6Al-4V surfaces during the initial stages of cell spreading and adhesion. In particular, the paper examines the effects of laser groove spacing on cell/surface interactions and adhesion, which is measured with a simple cell lift-off test. The implications of the results are then discussed for the design of improved cell/surface integrity between Ti biomedical implants and the human body.

Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties: Metallic Multilayers and Composites & Radiation Damage

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Amit Misra, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Harriet Kung, Los Alamos National Laboratory, Materials Science & Technology Division, Los Alamos, NM 87545 USA; Stuart A. Maloy, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Michael Nastasi, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Ricardo Schwarz, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Tuesday AM — Room: 10
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Harriet Kung, US Department of Energy, Div. of Matls. Scis. & Eng. USA; Mike Nastasi, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

8:30 AM Invited

Origins of Strength in Multilayer Composites: *Richard G. Hoagland*[1]; Amit Misra[1]; C. H. Henager[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]Pacific Northwest National Laboratory, Richland, WA 99352 USA

At very thin layer thickness in multilayer composites, strength ceases to obey a Hall-Petch relation, a behavior that may be, at least in metallic systems, a consequence of a transition in the nature of critical events from those involving groups of dislocations, like pile-ups, to interface crossings by single dislocations. In this paper we focus on the nonlinear atomic scale mechanisms responsible for the resistance encountered by single dislocation on crossing interfaces in fcc/fcc and fcc/bcc metallic composites. In fcc/fcc composites with small lattice mismatch, such as Cu/Ni, the limits to strength are found to be approximately equal to the coherency stress, even for semicoherent interfaces. A number of additional factors play a role, however, and include nonlinear effects on modulus, hardening due to steps, and the effect of stress on the core structures of interfacial misfit dislocations. In addition, the misfit dislocation core structures are found to be very different in Cu/Ag than Cu/Ni. In an fcc/bcc system the interface is found to contain two noncoplanar layers of misfit dislocations each with different Burgers vector. Such interfaces appear to be capable of acting both as sources and sinks for glide dislocations. This work was supported by the Office of Basic Energy Sciences, US Dept. of Energy.

9:00 AM Invited

Observation of the Diffusion Processes in Annealed Cr/Cu/Ni/Au Thin-Films by Quantitative X-Ray Mapping in Analytical Electron Microscopy: M. I. Danylenko[2]; M. Watanabe[1]; C. Li[1]; A. V. Krajnikov[2]; M. A. Vasiliev[3]; *D. B. Williams*[1]; [1]Lehigh University, Dept. of Matls. Sci. & Eng., 5 E. Packer Ave., Bethlehem, PA 18015-3085 USA; [2]Institute for Problems of Materials Science, 3 Krzhizhanivsky St., Kiev 03142 Ukraine; [3]Institute for Metal Physics, 36 Vernadsky Blvd., Kiev 03142 Ukraine

In microelectronics devices based on metallic multilayer thin-films, the thermal stability of the films in terms of the microstructure and the microchemistry is one of the most important properties. However, the thermal stability of multilayer systems is usually rather low. In a Cr/Cu/Ni/Au multilayer thin-film system, the Ni layer is usually inserted as a diffusion barrier to prevent any interactions between the Cu and Au layers since any interdiffusion between the Cu and Au layers degrades film properties. For industrial applications, control of the diffusion processes around the Ni layer are the key to improving the thermal stability in the Cr/Cu/Ni/Au multilayer system. To observe the diffusion behaviour in the annealed multilayer films, quantitative X-ray mapping has been applied using a 300keV field-emission gun analytical electron microscope. From quantified X-ray maps, the diffusion paths of Cu and Au in the Ni layer have been identified.

9:30 AM Invited

The Correlation of the Deformed and Recrystallized States in Directionally Solidified Cu-Cr: *C. W. Sinclair*[1]; J. D. Embury[2]; [1]The University of British Columbia, Dept. of Metals & Matls., Vancouver, BC V6T 1Z4 Canada; [2]McMaster University, Dept. of Matls. Sci. & Eng., Hamilton, ON L8S 4L7 Canada

A detailed study has been undertaken on the deformation of directionally solidified Cu-Cr containing 1.6at% Cr fibres that are initially dislocation free. The work hardening behaviour is dominated both by the need to load the Cr fibres until they yield at local stresses of the order of 4 GPa and the complex accommodation of large plastic strain gradients in the matrix and elastic strains in the Cr with continued plastic flow. Recrystallization of these materials provides evidence for the occurrence of ìoriented nucleationî such that similar orientations are produced at all the fibres in the array. Detailed experimental evidence and models for this process will be presented in this paper.

10:00 AM Break

10:30 AM Invited

Effects of Simultaneous Displacive and Ionizing Radiations and Electric Field on Radiation Damage in Ionic and Covalent Crystals: *Chiken Kinoshita*[1]; Syo Matsumura[1]; Kazuhiro Yasuda[1]; [1]Kyushu University, Appl. Quantum Physcs & Nucl. Eng., Hakozaki 6-10-1, Fukuoka 812-8581 Japan

A review is given on the formation process of radiation-induced defects and their stability under simultaneous displacive and ionizing radiations and electric field for ionic and covalent crystals. In ionic crystals such as MgO-Al2O3, retardation of dislocation loop formation, preferential formation of bubbles and retardation of amorphization are observed under simultaneous ionizing and displacive radiations. Furthermore, simultaneous irradiation with ions and electrons in the covalent semiconductors retards the accumulation of amorphous zones. The electric field, on the other hand, suppresses the formation of dislocation loops and enhances their growth in thinner specimens of ionic crystals. Those results are discussed in terms of the instability of defect clusters under electronic exciation and displacements and of the interaction between charged defects and electric fields.

11:00 AM Invited

Radiation Damage Effects in Oxides: *Kurt E. Sickafus*[1]; Robin W. Grimes[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS-G755, Los Alamos, NM 87545 USA; [2]Imperial College, Dept. of Matls., Prince Consort Rd., London SW7 2BP UK

We have used atomistic computer simulations and ion beam irradiations to examine radiation damage accumulation in multi-component oxides. We have developed contour energy maps via computer simulations to predict the effects of oxide structure and chemical composition on radiation-induced atomic disorder, defect migration, and swelling. Ion irradiation damage experiments have been performed on fluorite, pyrochlore, and perovskite-structured oxide ceramics to test the predictions from computer models. This presentation will examine theoretical predictions of radiation damage behavior and the results of experimental tests using ions.

11:30 AM Invited

Irradiation-Induced Solute Segregation and Precipitation: Current Status: *Alan J. Ardell*[1]; [1]University of California, Dept. of Matls. Sci. & Eng., Los Angeles, CA 90024-1595 USA

Irradiation-induced segregation of atoms in solid solution is an important phenomenon in commercial alloys used in nuclear reactors. Grain boundaries in neutron-irradiated austenitic stainless steels eventually become depleted of Cr and enriched in Ni, with the deleterious consequence of promoting the susceptibility of the steel to stress-corrosion cracking. It is therefore not surprising that irradiation-induced solute segregation (IISS) is currently a lively topic of research in the nuclear materials community. IISS was first observed in the mid-seventies, and it was realized essentially immediately that the coupled diffusion of solute atoms and vacancies and/or interstitials to point defect sinks must play an important role in the process. Shortly after the first observations of IISS, the related phenomenon of irradiation-induced precipitation (IIP) in under-saturated alloys was discovered. This was exemplified by the unexpected heterogeneous nucleation of precipitates at dislocation loops, as well as at grain boundaries and free surfaces. Irradiation-induced homogeneous precipitation in the interior of defect-free grains of under-saturated Ni-Si alloys was also observed. As he did in so many different fields of research, Terry Mitchell made important contributions in this area as well. In this presentation the subjects of IISS and IIP will be reviewed, starting with its early history and ending with the current state-of-the art.

The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research: Hexagonal Materials, Intermetallics, & Ceramics

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Japan Institute of Metals, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Julia R. Weertman, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Katherine T. Faber, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Morris E. Fine, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Wayne King, Lawrence Livermore National Laboratory, San Ramon, CA 94583-2496 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Ben Mori, Tokyo 168-0081 Japan

Tuesday AM — Room: 9
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: T. Mori, Rutherford Appleton Laboratory, ISIS Fac., Chilton, Didcot OX11 0QX UK; P. K. Liaw, University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37966-2200 USA

8:30 AM Invited

Making Golf Easier with Titanium and Materials Science: *E. Y. Chen*[1]; C. C. Chen[1]; F. H. Froes[2]; [1]TiTech International, Inc., 4000 W. Valley Blvd., Pomona, CA 91769-3060 USA; [2]University of Idaho, Inst. of Matls. & Adv. Processes, Moscow, ID 83844-3026 USA

The metal of the Titans is now extensively used in golf club heads allowing the weight to be strategically placed by the designer, making a user-friendly head with an oversized ìsweet spot.î As a tribute to Professor Mike Meshii for his contributions to the field of Materials Science, and to help ease him into what expects to be one of many pursuits in his well-deserved retirement, this paper discusses the application of titanium and materials science to fabricating the modern golf club head. Specific topics covered include the design and manufacture of hitting faces, often with judicious weighing using dense elements such as tungsten, processes for producing the clubs, and the evolution of materials used in golf balls. With very thin, high strength faces a major ìtrampolineî effect can be built into the head adding distance to the golferís device. Can the average golfer benefit from these advances? - Yes, although it may cost you.

9:00 AM
Crystallographic and Morphological Aspects of a Laths in α-β Ti Alloys: *Dhriti Bhattacharyya*[1]; Gopal B. Viswanathan[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

In alpha/beta Ti alloys, the crystallographic orientation of α laths and their corresponding geometrical orientations are closely linked due to the nature of the interphase boundary between the α (hcp) and the β (bcc) phases and the Burgers orientation relationship (OR) between the phases. In this work, it is demonstrated that different colonies of α laths growing within the same β grain could have distinctly different geometrical growth directions but could still maintain a close crystallographic relationship. It is seen that different variants of the α phase share a common (0001) basal plane, but are rotated only by ~10°. Similar phenomenon is also observed across two neighboring β grains where a preference for the a colonies to have their common (0001) planes parallel to common or closely oriented {110} planes in the β phase. TEM samples were extracted from these special boundaries using Focussed Ion Beam (FIB) for detailed analysis. Explanations for the observed phenomena are discussed based on the results obtained from Orientation Microscopy (OM), SEM and detailed TEM investigations.

9:20 AM
Change in Lamellar Boundary Structure and Saturation of Yield Stress in Nano Lamellar TiAl Alloy: *Kouichi Maruyama*[1]; Gou Suzuki[1]; Hee Y. Kim[2]; [1]Tohoku University, Dept. of Matls. Sci., Aoba-yama 02, Aoba-ku, Sendai 980-8579 Japan; [2]University of Tsukuba, Inst. of Matls. Sci., 1-1-1, Ten-nodai, Tsukuba 305-8573 Japan

Mechanical properties and lamellar boundary structure of a Ti-39mol%Al alloy with fully lamellar structure were studied at room temperature paying special attention to effects of lamellar spacing. Lamellar spacing ranging from 20nm to 600nm was made by isothermal aging at various temperatures, and most of lamellar boundaries were gamma/alpha2 boundary in the specimens. Yield stress of the alloy increases with decreasing lamellar spacing, and the Hall-Petch relation holds over the range of lamellar spacing greater than 100nm. The yield stress saturates at a value of about 1GPa below the critical lamellar spacing. TEM analysis of the lamellar boundaries revealed that misfit dislocations are introduced to the boundary when the lamella is thicker than 50nm. The saturation of yield stress will be discussed on the basis of the TEM observation of lamellar microstructures.

9:40 AM
Parent-Product Interphase Boundaries and their Implications on the α to γ_M Massive Transformation in Ti-Al Alloys: Ping Wang[2]; Veer Dhandapani[3]; Mukul Kumar[4]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA; [3]Motorola, Inc., Digital DNA Labs., 3501 Ed Bluestein Blvd., MD K-10, Austin, TX 78251 USA; [4]Lawrence Livermore National Laboratory, 7000 East Ave., MS L-370, Livermore, CA 94550 USA

The massive-matrix interphase interfaces associated with the α to γ_M massive transformation in Ti-(46-48)Al alloys were studied. Special experiments were performed to arrest the transformation at an early stage. Nucleation and growth kinetics were determined and the former compared with calculations for various nuclei shapes using classical nucleation theory. Orientation relations between the γ_M and parent α (retained α_2) phases were determined using EBSD in an SEM and by electron diffraction, and the interphase interfaces and defect structures in the γ_M phase characterized by two-beam bright-field/weak-beam dark-field TEM and HRTEM. The results reveal that the γ_M nucleates at grain boundaries with a low-index orientation relation and coherent interface with one parent grain, but grows into the adjacent grain with a high-index/irrational orientation relation. The growth interfaces between the two phases are generally free of misfit dislocations and consist of curved parts as well as planar facets whose macroscopic habit varies from high-index/irrational to low-index orientation. On an atomic scale the growth interfaces are occasionally found to be faceted along {111} planes with steps, but are incoherent with respect to the parent grain into which growth occurs. The implications of these results on the nucleation and growth mechanisms associated with the α to γ_M massive transformation will be discussed. The authors are grateful for support of this research by the National Science Foundation under grants DMR-9224473 and 9731349, Dr. Bruce MacDonald, Program Monitor.

10:00 AM
Scanning Electron Microscopy of Natural Ice: *I. Baker*[1]; D. Cullen[1]; D. Iliescu[1]; R. Obbard[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

The microstructural location of impurities in natural polycrystalline ice has been of interest for over a century. Recently, we have developed a technique to determine the microstructural location of impurities in ice that involves controlled preferential sublimation of ice in a low-vacuum scanning electron microscope equipped with a nitrogen gas-cooled cold stage. The sublimation leaves behind concentrated impurities, which can be identified using x-ray microanalysis. In this presentation, observations on specimens from Greenland, Antarctica, and pond and river ice will be presented. The polar ice cores showed impurities such as Cl, S and less-commonly Na, Ca and Mg throughout the material. In the ice from Greenland, filaments consisting chiefly of NaCl were observed in the grain boundaries (GBs). These are thought to form by coalescence of the impurities that were located in the GB plane. The grain interiors of the lake and river ice also contained impurities. However, in river ice, most of the impurities were located in inclusions of ≤ 20 µm diameter in the GBs. In contrast, the pond ice contained many unusually-shaped, but often facted, hexagonal-symmetry features from 200-1000 µm in diameter that were usually located in or near the GBs. These features appear to simply be small ice crystals that have nucleated on impurities. This research was supported by US National Science Foundation grant OPP-9980379 and Army Research Office grant DAAD 19-00-1-0444.

10:20 AM
Grain Size Hardening and Softening in Intermetallic Compounds at Low Homologous Temperatures: *Hans Conrad*[1]; Jay Narayan[1]; [1]North Carolina State University, Matls. Sci. & Eng., Raleigh, NC 27695-7907 USA

Results by the authors on the effect of grain size on the hardness of laser-deposition WC and TiN films with grain sized in the range of 6-46 nm are presented and compared with data in the literature for these materials and other intermetallic compounds. Three grain size regimes were identified: Regime I (d=10^-6-10^-3m), Regime II (d=10^-8-10^-6m) and Regime III (d<10^-8m). Regimes I and II are characterized by grain size hardening (Hall-Petch effect) governed by intragranular dislocation activity, Regime III by grain size softening (inverse Hall-Petch effect) characterized by the absence of intragranular dislocation activity and resulting from grain boundary shear. The mechanisms governing each of the regimes are discussed.

10:40 AM Cancelled
Comparison Between Microstructures of Conventional and New Raney Catalysts: *Jose G. Cabanas-Moreno*[1]; Beatriz H. Zeifert[1]; Hector A. Calderon[1]; J. B. Salmones[2]; [1]ESFM-IPN, Ciencia de Materiales, Apdo. Postal 75-707, Mexico DF 07300 Mexico; [2]Instituto Mexicano del Petroleo, Eje Central Lazaro Cardenas 152, Mexico, DF 07730 Mexico

11:00 AM
Influence of Si Content and Sputtering Condition on the Microstructure and Mechanical Properties of r.f.-Sputtered Transition Metal Nitride Films: *Masateru Nose*[1]; Wen-An Chiou[2]; Yutaka Deguchi[3]; Takehiko Mae[4]; Kiyoshi Nogi[5]; Mike Meshii[6]; [1]Takaoka National College, Dept. of Industl. Arts & Crafts, 180 Futagami-machi, Takaoka, Toyama 933-8588 Japan; [2]University of California-Irvine, Dept. of Cheml. Eng. & Matls. Sci., 636E Engineering Tower, Irvine, CA 92697-2575 USA; [3]Toyama University, Grad. Sch. of Sci. & Eng., 5190 Gofuku, Toyama 930-8555 Japan; [4]Toyama National College of Technology, Dept. of Environl. Matls., 13 Hongo-machi, Toyama 939-8630 Japan; [5]Osaka University, Joining & Welding Rsrch. Inst., 11-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan; [6]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

The influence of Si content and sputtering condition on the microstructure and mechanical properties (hardness and Youngís modulus) of transition metal nitride films such as ZrSiN and TiSiN were investigated using XRD, TEM and nanoindentor. While the substrate was heated from room temperature to 673 K during deposition, some films were applied with d.c.-bias voltage. Without substrate heating and bias application, the hardness of the films increased for 5-10 GPa over binary system such as ZrN or TiN with a small amount (3-5 at%) of Si;

it then decreased to a hardness lower than those of binary systems when Si was more than 10 at%. The tendency to grow columnar grains was strongest at a ~ 3-5 at% of Si, but it became equiaxial when Si was > 5 at%. However, nano-crystal and/or amorphous like structure were formed if Si was > 10 at%. The increase of hardness with small additional amount of Si to metal nitride films is probably attributed to the atomic strain caused by Si atoms at nonequilibrium sites. These results also indicate that the grain size can be of minor contributions to the hardening. The influences of the sputtering conditions will also be discussed.

11:20 AM

HREM Analysis of γí Particles in Late Stages of Coarsening in Ni Alloys: Lorenzo Calzado-Lopez[1]; T. Mori[2]; Christian Kisielowski[3]; *Hector A. Calderon*[1]; [1]ESFM-IPN, Ciencia de Materiales, Apdo Postal 75-707, UPALM Ed 9, Mexico, DF 07300 Mexico; [2]Rutherford Appleton Laboratory, ISIS Fac., Chilton, Didcot OX11 0QX UK; [3]Lawrence Berkeley National Laboratory-National Center for Electron Microscopy, Berkeley, CA 94720 USA

Coarsening of coherent particles in solid matrices apparently leads to particle splitting after a given critical size. Experimentally symmetrical particle arrangements have been used to demonstrate the occurrence of splitting but little has been done regarding kinetics or the development of particle size distributions (PSD) after splitting. This investigation deals with determination of order domains in γí particles by high resolution electron microscopy (HREM). Several Ni base alloys have been used (model and commercial) in the late stages of coarsening where a critical size for splitting is expected. Nevertheless the present results show that most particles (around 85%) in a variety of arrangements and sizes, have different order domains. This suggests that splitting has not taken place since otherwise particles in a given group would have an identical order domain. On the other hand, both kinetics and PSD show an smooth development without any indication of splitting.

11:40 AM

Dislocation Activity During Superplastic Flow in the Zn-22% Al Alloy: *Yuwei Xun*[1]; Wen-An Chiou[1]; Farghalli A. Mohamed[1]; [1]University of California-Irvine, Dept. of Cheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

An investigation was conduced on the superplastic Zn-22% Al alloy that contained nanometer-scale dispersion particles, which were introduced into the microstructure by powder metallurgy. In the investigation, specimens were crept at a strain rate near the center of the superplastic region, which represents region II (the intermediate-stress region), in the sigmoidal plot between stress and strain rate that was reported for the alloy. Preliminary transmission electron microscopy (TEM) observations reveal evidence of lattice dislocation activity. This evidence is demonstrated by the presence of particle-dislocation interactions in the interiors of the grains of the alloy. The results are discussed with reference to various deformation mechanisms proposed for micrograin superplasticity.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Metals and Sulfide Systems

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday AM — Room: Solana
March 4, 2003 — Location: San Diego Marriott Hotel

Session Chairs: R. Hurman Eric, University of the Witwatersrand, Sch. of Process & Matls. Eng., Wits 2050 S. Africa; Seshadri Seetharaman, Royal Institute of Technology, Div. of Metall., Stockholm SE-100 44 Sweden

8:30 AM Keynote

Solute Interactions with Dissolved Oxygen in Molten Copper Systems: *Seshadri Seetharaman*[1]; Patrik Fredriksson[1]; Ragnhild Aune[1]; [1]Royal Institute of Technology, Div. of Metall., Stockholm SE-100 44 Sweden

In fire refining of copper, the affinities of various impurity elements on dissolved oxygen is of great significance. Further, in the design of oxygen probes in liquid copper, the influence of solute interactions on the activity of oxygen need to be considered in order to evaluate the oxygen concentration in molten copper. The present paper presents a series of measurements of the activities of oxygen in liquid Cu-M-O alloys (where M stands for Mn, Zn, As, Se or Te) carried out by the present group employing the solid electrolyte galvanic cell technique. Extreme care was taken to control the oxygen potentials in the inert gas atmosphere, and to keep the oxygen levels very low in the molten metal. The oxygen content was determined by analysis of the samples taken out from the melt. The results obtained showed that Mn has a strong negative influence on the activity coefficient of oxygen in liquid copper. A similar, but less prominent effect was observed in the case of Zn. Arsenic had no influence on the activity of oxygen in Cu (liq.). Se and Te had slight negative effect on oxygen activities in copper at $X_O 10^{-5}$, while at lower oxygen levels the interaction coefficients were found to be positive. The results obtained were compared with empirical models for the calculation of interaction coefficients available in literature.

9:05 AM

Desulfurization Kinetics of Molten Copper Matte by Gas Bubbling: H. Sang Sohn[2]; Yasuhiro Fukunaka[1]; Toshio Oishi[3]; [1]Kyoto University, Dept. of Energy Sci. & Tech., Sakyo-ku, Kyoto 606-8501 Japan; [2]Kyungpook National University, Dept. of Matls., Daegu 702-701 Korea; [3]Kansai University, Dept. of Matls. Eng., Suita 564-8180 Japan

The kinetic study on desulfurization of copper matte by gas bubbling was designed under the presumptions of lower FeO activity and of no fayalite slag formation. Copper matte with 59% Cu and 22.6% S in weight ratio was oxidized at 1523K by bubbling of Ar+O2 gas through a submerged nozzle. The effects of oxygen partial pressure and flow rate of gas on the oxidation rate of matte were discussed. The desulfurization rate and evolution rate of SO2 gas of copper matte were influenced by the oxygen partial pressure. However, they were confounded each other. The desulfurization rate and evolution rate of SO2 gas of copper matte were described by the mass transfer rate through the gas film around the rising gas bubbles. The calculated results reasonably describe the observed desulfurization rate and evolution rates of SO2 gas at a constant oxygen partial pressure of 0.2 atm.

9:30 AM

Phase Equilibria and Thermodynamics in the Ag-Pb-S Ternary System: *R. Hurman Eric*[1]; Hakan Ozok[1]; [1]University of the Witwatersrand, Sch. of Proc. & Matls. Eng., PB 3, Wits, Johannesburg, Gauteng 2050 S. Africa

Within the Ag-Pb-S ternary system, the boundaries of the immiscibility region together with the tie-line distributions were re-established at 1198K by the equilibration-quenching technique where samples were kept in evacuated and sealed silica capsules. Activities of Pb were measured by the dew-point method along the Ag-Pb binary at 1198, 1373 and 1473K. Positive deviations were observed and the results were modelled by the Krupkowski formalism. Activities of Ag, Pb, and S, along the ternary miscibility gap were calculated at 1198K by utilizing the bounding binary thermodynamics, phase equilibria and tie-lines. Activities of Pb were also measured along the metal-rich boundary of the miscibility gap to confirm the calculated Pb activities at 1198K.

9:55 AM

Activities of the Cu_2S-FeS System Calculated from the Data of the Cu_2S-FeS-Sb_2S_3 System: *Grigore Matei*[1]; Yasushi Takasaki[2]; Kimio Itagaki[3]; Kazuo Koike[2]; [1]University ìPolitehnicaî of Bucharest, Matls. Sci. & Eng. Fac., Spl. Independentei 313, Bucharest Romania; [2]Akita University, 1-1 Gakuen-Cho, Tegata, Akita 010-8502 Japan; [3]Tohoku University, 2-1-1 Katahira, Aoba-Ku, Sendai 980-8577 Japan

The thermodynamic property of the Cu_2S-FeS system is important not only in the case of copper smelting but also in the case of general pyro-metallurgy. However, there are not many reported activity data. In this study, activities of Cu_2S and FeS in the Cu_2S-FeS system were calculated using the Darkenís method from the activity data of the Cu_2S- FeS-Sb_2S_3 system measured by the transportation method at 1050°C. The activities of Cu_2S and FeS in the Cu_2S-FeS system showed negative deviation from the ideal behavior and satisfied the relation of the Gibbs-Duhem equation.

10:20 AM Break

10:40 AM Invited
High Temperature Thermodynamic Studies on Sulfide Systems Using the Dew Point Technique: *R. Hurman Eric*[1]; [1]University of the Witwatersrand, Sch. of Process & Matls. Eng., PB 3, Wits 2050 S. Africa

The dew-point technique of activity measurements take advantage of the volatility of a component in solution. The pressure of the volatile component over a sample is determined from observations of the temperature at which condensation of the vapor occurs. The method is applicable best when the vapor pressure of the components would differ by about two to three orders of magnitude. Experimentally, this is a closed system involving the use of evacuated and sealed transparent quartz sample probes. Furthermore reliable vapor pressure data of the volatile component is essential along with a specialized high temperature furnace capable of giving reproducible results. In this context, the vertical Dew-Point Furnace (DPF) was designed and built after elaborate trials until enough proficiency was gained for reproducible results. Over the years the DPF was modified and improved. The DPF developed was versatile and could easily be used for activity measurements in the isopiestic mode (another method of measuring activities of a volatile component in a solution). The necessary ancillary systems; namely evacuation-flushing-evacuation followed by sealing of quartz sample probes under vacuum were also perfected over the years. As and when necessary the dew-point technique was supplemented by classical equilibration-quenching method employed on sealed quartz sample probes to reveal necessary phase equilibrium information. The following were studied utilizing the above technique: (i)Activities in Cu_2S-PbS and FeS-PbS binary and Cu_2S-FeS-PbS ternary at 1200°C, (ii)Phase equilibria and thermodynamics in the Pb-PbS system, (iii)Phase equilibria and thermodynamics in the Cu-Pb-S and Fe-Sn-S systems, (iv)Activities in FeS-SnS and Ag_2S-SnS binary and FeS-Cu_2S-SnS ternary at 1200°C,(v)Phase equilibria and thermodynamics in Ag-Pb, Fe-Pb-S, Ag-Sn-S and Ag-Pb-S systems. Most recently the following systems have been tackled: Phase equilibria and thermodynamics in the Sn-Pb-S system and activities in Ag_2S-PbS binary, Ag_2S-Cu_2S-PbS and Ag_2S-FeS-PbS ternaries and Ag_2S-Cu_2S-FeS-PbS quaternary at 1200°C.

11:10 AM
Thermodynamic Properties of Copper-Nickel Mattes: *L. Sh. Tsemekhman*[1]; A. V. Tarasov[2]; V. M. Paretsky[2]; [1]AO ìGipronickelî, 1 Grazhdansky Pr., Saint-Petersburg 195220 Russia; [2]"GINTSVETMETî, 13 Acad. Korolyov St., Moscow 129515 Russia

Studies into thermodynamic properties of Cu-Ni and Cu-Ni-S systems and mattes have been conducted with the aid of a MC-1301 mass-spectrometer designed for investigation of processes of vapor formation of difficult-to-volatilize substances. The instrument constitutes a combination of an evaporator of Knudsen chamber type and a mass-spectrometric analyzer of vapor phase. Effusion Knudsen chambers were preliminarily calibrated based on the silver and gold vapor pressure recommended as IUPAC pressure standards. Partial pressures of vapor components were determined, depending on the objective set, by the method of complete isothermal evaporation or the ion current comparison method. Measurements of temperature relationship of the ion current intensity of Cu+ and Ni+, as well as atomic copper vapor pressure above molten copper and atomic nickel vapor pressure above solid nickel metal using the method of complete isothermal evaporation made it possible to define equations of the effect of temperature within a range of 1400K to 1800K on copper and nickel vapor pressures. The process of copper-nickel system evaporation within the entire range of compositions had a clear-cut incongruent character and the intensity of ion current of Cu+ during the test period decreased, while that of Ni+ increased. When studying the Cu-Ni-S system, the sulfur partial pressure was measured by the complete isothermal evaporation method and the copper and nickel partial pressure was measured by the ion current comparison method. The values of partial pressures of components above the Cu-Ni-S melt were used for calculation of activities of these components in the melt. The copper and nickel activity data were used for assessment of the value of soluble losses of these metals in slag of autogenous smelting. Studies of evaporation processes of commercial-grade mattes were carried out at a temperature of 1500K. As a result of processing of experimental data, relationships of partial pressures of copper, iron and sulfur were obtained, which are valid for the following ranges of compositions, % at.: 0.3-15.4 Cu, 35.2-80.6 Fe and 10.4-30.7 Ni. <p>PCu = 1.35 - 0.034[Cu] - 0.013[Fe] - 0.014[Ni] (1) P = 1.52 - 0.039[Cu] - 0.015[Fe] - 0.010[Ni] (2) PFe◊10-2 = 1.10 - 0.024[Cu] + 0.004[Fe] - 0.039[Ni] (3) <p> Activity values and activity coefficients of nickel have been calculated on the basis of the data obtained.

11:35 AM
Determination of the Degree of Oxidation of Molten Copper Using an Electrochemical Cell: *JosÈ Alberto V·zquez-Monroy*[1]; JosÈ Antonio Romero-Serrano[1]; Mario Alberto GarcÌa-GarcÌa[1]; Samuel Ganz·lez LÛpez[1]; [1]E.S.I.Q.I.E-IPN, Metallurgl. Eng., Unidad Profesional Adolfo Lopez Mateos, Lab. Pesados de Metalurgia, MÈxico D.F. 07738 MÈxico

Copper is the third most widely used metal in terms of tonnage per year throughout the world, after iron and aluminum. Copper is an excellent conductor of electricity and heat, only slightly exceeded by silver. The electrical conductivity of copper explains its the sharp increase with the arrival of the age of electricity. At the present, almost 75% of copper consumption goes into this type of usage. The high temperature galvanic cells have come into prominence on account of their numerous applications in both fundamental and applied measurements. Since the inception of the oxygen-ion conducting electrolytes like ZrO2-CaO, the oxygen concentration cells have been extensively used in laboratories as well as in industries. In metal production technology, these cells have been used to monitor the oxygen potentials in metals. The pioneer among the various industrial users of such cells is the copper industry. Today, high temperature solid electrolyte cells involving oxygen ion conducting electrolytes have become an integral part of copper pyro-refining and continuous casting, because it is possible to control the oxygen level and to predict the amount of other alloy elements. For these reasons, in the present paper the degree of molten copper oxidation was determined in terms of the amount of Cu3P used as deoxidant, the particle size of Cu3P and temperature. The oxygen sensor was prepared with Zirconia Stabilized with Itria (ZEI), using Ni-NiO as reference electrode. The first part of this study consisted of developing an electrochemical cell to measure the oxygen activity in molten copper. The second part consisted of a thermodynamic analysis, which was carried out with a commercial software FACT (Facility for the Analysis of Chemical Thermodynamics). This software has databases of pure substances and systems in solution and can be used to study complex process like the Copper production. The thermodynamic study allowed to relate the oxygen activity and its concentration. Finally it was developed a model of dissolution of Cu3P in the bath in terms of the reaction time and temperature, which allows to estimate the dissolution rate of Cu3P particles in the melt.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Aqueous and Electrochemical Processing II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday AM Room: Pacific
March 4, 2003 Location: San Diego Marriott Hotel

Session Chairs: Yoshiaki Umetsu, Tohoku University, Inst. of Multi. Rsrch. for Adv. Matls., Sendai 980-8577 Japan; Rafael Padilla, University of ConcepciÛn, Metallurgl. Eng., ConcepciÛn Chile

8:30 AM Keynote
Environmentally Friendly Route to Copper Production: Sulfidation and Leaching of Chalcopyrite Concentrates: *Rafael Padilla*[1]; [1]University of ConcepciÛn, Metallurgl. Eng., Edmundo Larenas 270, ConcepciÛn Chile

In the production of copper from chalcopyrite (CuFeS2) concentrates by the smelting and converting technology, the ambient pollution with gaseous SO2 and toxic metals such as arsenic compounds is still a serious problem, especially in the converting step due to the batch nature of the conventional Peirce Smith process. Non-SO2 emitting alternatives are leaching processes. However, few of them have had a limited industrial application and they are not very effective in discarding safely the toxic impurities. A combination of pyro-hydrometallurgical methods: the sulfidation of chalcopyrite and subsequent leaching of the sulfidized material is a non-polluting alternative to treat not only clean chalcopyrite concentrates but also complex chalcopyrite-enargite concentrates. In this article, this alternative is discussed concerning the thermodynamics and kinetics of the sulfidation of chalcopyrite with gaseous sulfur. The kinetics of the leaching of the sulfidized concentrate in an H2SO4-NaCl-O2 system is also discussed.

9:05 AM

Construction of Electrical Conductivity of Copper Sulfate Electrolytes Acidified with Sulfuric Acid for Copper Electrorefining and Electrowinning Processes: *Kazuteru Tozawa*[1]; Yoshiaki Umetsu[2]; Quing-Quan Su[3]; Zi-Qiang Li[4]; [1]Tohoku University, Sendai 980-0845 Japan; [2]Tohoku University, Inst. of Multi. Rsrch. for Adv. Matls., Sendai 980-8577 Japan; [3]Ebara Corporation, Tokyo 144-8510 Japan; [4]Chandu Science and Technical University, Chandu China

The electrical conductivity of electrolytes for copper electrorefining and electrowinning processes has been reported by many researchers since 1911. The experimental results show that the conductivity increases with an increase in sulfuric acid concentration, but decreases with an increase in copper concentration. In pure copper sulfate solution, however, the conductivity increases with an increase in copper concentration. After confirming the conductivity of pure and acidified copper sulfate solutions experimentally, we found that the conductivity of acidified copper sulfate electrolytes is mainly controlled by concentrations of hydrogen ion and free (not hydrated) water. It can be explained on the basis of the finding that the conductivity of copper sulfate electrolytes acidified with sulfuric acid decreases with an increase in copper concentration.

9:30 AM

Separation of Copper and Arsenic from Copper-Arsenic-Sulfuric Acid Electrolytes by Using Electrodialysis: *J. P. Ibanez*[1]; C. Gutierrez[1]; L. Cifuentes[2]; [1]Arturo Prat University, Dept. of Metall., Av. Arturo Prat 2120, Iquique Chile; [2]Universidad de Chile, Dept. of Mining Eng., Av. Tupper 2069, Santiago Chile

The separation of arsenic and copper from Cu-As-H2SO4 electrolytes by using electrodialysis was investigated at ambient temperature in a laboratory batch cell. The effect of current density and pH were studied. A solution of copper free of arsenic was obtained by this technique at all the conditions investigated. The efficiency for the transport of Cu2+ was found to reach values higher than 98% in 3 hours. The transport of arsenic and copper through the ion exchange membranes was favored by increasing the pH and by increasing the current density. The pH of the system was found to be a key parameter for the process, since it controls the speciation of the ions to be transported and the formation of unwanted precipitates that reduced the global efficiency of the electrodialysis process. The main conclusion of the work is that the electrodialysis is a promising technique to be used in separating and/or concentrating ions of interest in electrometallurgical plants of copper.

9:55 AM

Effect of Fe(II), Co(II) on the Formation of Lead and Manganese Oxides During Copper Electrowinning: *A. Pagliero*[1]; F. Vergara[1]; J. Ipinza[2]; J. L. Delplancke[3]; [1]University of ConcepciÛn, Dept. of Metallurgl. Eng., Edmundo Larenas 270, PO Box 53-C, ConcepciÛn 00187 Chile; [2]University Arturo Prat, Dept. of Metallurgl. Eng., Arturo Prat 2120, PO Box 121, Iquique Chile; [3]University Free of Brussels, Fac. of Appl. Scis., 50 Av. F.D. Roosevelt, CP 194/03, Brussels B-1050 Belgium

Small manganese concentration and other impurities (Fe^{2+},Al^{3+}) in the high acid electrolyte are transferred to copper electrowinning. Experimental test of electrolysis were conducted a 50∫C in 180 g/L H_2SO_4 to investigate the behavior of manganese and iron in solution on the film stability of the alloy PbCaSn. The effects of ions such as Fe^{2+}, Co^{2+} in acidic electrolytes with manganous ions on the anode corrosion were investigated. These impurities were found to affect the anode PbCaSn corrosion. The presence of 4g/L Fe^{2+} in the electrolyte and different manganese concentration in the rank 0.07 to 4 g/L, inhibit the formation of slimes formed by β-MnO_2 tetragonal (disproportionation reaction). However, the layer of MnO_2 amorphous formation on the lead oxides is uninterrupted but its thickness is very thin. This condition aid the dissolution of the PbO_2. The corrosion of PbCaSn anode is much less when it is compared to media containing only cobalt and manganese.

10:20 AM Break

10:35 AM Invited

Surface Tension, Density and Viscosity Coefficient of Acidic Copper Sulfate Solution Simulating Electrolyte Solution for Electrolytic Copper Production With/Without Addition of Gelatin: Kazuteru Tozawa[1]; Quing-quan Su[2]; *Yoshiaki Umetsu*[3]; [1]Tohoku University, Sendai 980-0845 Japan; [2]Ebara Corporation, Tokyo 144-8510 Japan; [3]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 1-1 Katahira 2-chome, Aoba-ku, Sendai 980-8577 Japan

Surface tension, density and viscosity coefficient of the acidic copper sulfate solutions simulating electrolyte solution for copper electrorefining has been measured and are summarized as a function of the solution composition and temperature. Nickel sulfate is taken to be a representative coexisting sulfate affecting the physical properties of the electrolyte solution. The sulfates in the solutions including sulfuric acid increase the values of physical properties under consideration. Gelatin, representing organic additives for electrolyte solution, was found to sensitively affect the surface tension of the solution at low concentration less than 200mg/L. The surface tension was markedly lowered by addition of gelatin and the higher concentration of gelatin led to more pronounced decrease in the measured values. The surface tension gradually increased with time after addition of gelatin and the increment was enhanced by higher acid concentration and higher temperature. The observed change in the surface tension is considered to reflect preferential adsorption of gelatin to the solution surface and decomposition of gelatin due to hydrolysis to form various molecules having smaller molecular weights.

11:05 AM Invited

The Mechanism of Sphalerite Dissolution in Ferric Sulphate-Sulphuric Acid Media: *A. Pratt*[1]; J. E. Dutrizac[1]; T. T. Chen[1]; [1]CANMET, 555 Booth St., Ottawa, Ontario K1A OG1 Canada

Fracture exposed surfaces of sphalerite samples having five different iron contents ranging from approximately 0 wt% to 14.8 wt% Fe were leached from 15 to 120 seconds at idd0c in 0.3 M $Fe(SO4)_{1.5}$-0.3 M H_2SO_4 media. The reacted samples were examined using X-ray photoelectron spectroscopy (XPS) which provides chemical state information on the first few nanometers of the solid surface. Surface compositions obtained from reference and leached sphalerites show that the leaching rate increases with increasing iron content. Detailed evaluation of high resolution S2p spectra show that the leaching progresses via the formation of polysulphide species. Disulphide species were not detected in any of the experiments. Examination of the Zn2p and Fe2p spectra show little change in the chemical states over the duration of these experiments. Longer leaching times resulted in the generation of elemental sulphur. The ratios of elemental sulphur to dissolved ferrous ions and zinc suggest that less than 5% of the sulphide is oxidized to sulphate, for zinc extractions ranging from 8 to 100%. Morphological studies showed that the elemental sulphur initially formed at a few isolated sites, such as the grain boundaries Furthermore, faceted euhedral sulphur crystals were often identified. These observations suggest that at least part of the elemental sulphur forms via dissolved sulphide species which are oxidized in solution by the ferric sulphate.

11:35 AM

Technological Flowsheet of Sulfide Copper Concentrates Treatment as Non-Ferrous Metals Production By-Products: *Y. M. Shneerson*[1]; A. Y. Lapin[1]; T. Y. Kositskaya[1]; K. A. Muravin[1]; L. V. Chugaev[1]; [1]Norilsk Nickel RJS, Gipronickel Institute JS, Saint-Petersburg Russia

At Gipronickel Institute the technological flowsheet of pressure leaching of copper from rich copper concentrates (i.e. containing: Cu -59-75%; Ni -0.5-4.64%; Fe -0.2-1.3%; S -21.8-26.9%) was developed. As a result, the possibility of deep decomposition of copper-containing minerals with extraction of elemental sulphur (up to 90-95%) was established. The obtained sulphur concentrates contain 80-90% of elemental sulphur, which can be separated from sulphide residue through autoclave melting and settling. As a rule, sulphide concentrates remaining after extraction of sulphur contain up to 1-2% of noble metals. The sulphide concentrate can be purified to the level of rich concentrates or pure metals using the well-known methods as in the case of tankhouse slimes. The presented research specifies the parameters of main operations: pressure leaching and sulphur extraction. It was established that 98% of copper could be transferred into solution at temperatures of 108-110∞C and oxygen partial pressure 0.4-0.6 MPa. The above process may be realised as a one- or two-stage operation. Applying the two-stage operation will reduce the total residence time of the material in an autoclave and thus, the total number of required autoclaves. Experiments were carried out in autoclaves of various volumes using the products of nickel matte processing.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Recycling, Waste Treatment and Environmental Issues II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday AM Room: Leucadia
March 4, 2003 Location: San Diego Marriott Hotel

Session Chairs: Jes´s Contreras, Atlantic Copper S.A., Huelva Spain; Florian Kongoli, FLOGEN Technologies Inc., Matls. Tech. Dept., Montreal, Quebec H3S 2C3 Canada

8:30 AM Keynote
Metal Recycling and Waste Treatment: *Yoshihiko Maeda*[1]; [1]Dowa Mining Company, Ltd., 1-8-2 Marunouchi, Chiyoda-Ku, Tokyo 100-8282 Japan

It is said that the 21st century will be an era to remedy global environment. To reserve natural resources for the future and to avoid dispersion of heavy metals, after usage, not to cause pollution problem, it is extremely important to recycle heavy metals as much as possible by reasonable recovery procedures. Nonferrous metal industry is expected to play a great role in recycling of heavy metals that are originally their products. From the depressed metal price experience in 1980th in Japan due to the sudden change in current exchange rate between US dollar and Japanese yen, one of major nonferrous metal company, Dowa Mining, has diversified its business area not only to so-called down stream, but also to environmental business including metal recycling, industrial waste treatment and soil remediation by utilizing technology and facilities once used or still used for mining and smelting of copper, zinc, lead and precious metals. In this paper, some of these applications will be presented.

9:05 AM
The Decomposition Mechanism of Precursor 2,4,5-Trichlrorophenol for 2,3,7,8-TCDD Using Ab Initio Molecular Orbital Method Calculation: *Takashi Araki*[1]; Mitsuhito Hirota[1]; Akio Fuwa[1]; [1]Waseda University, Grad. Sch. of Sci. & Eng., Ookubo 3-4-1 Soudai-rikou 60-110, Sinjuku-ku, Tokyo 169-8555 Japan

Dioxins (a generic term for PCDDs and PCDFs) have been the most serious air pollutants, because these compounds have highly acute and chronic toxicity. At present, removal and inhibition techniques of dioxins emission have been proposed by several workers, and catalytic decomposition method has been believed to be one of the most useful methods. However, the reaction mechanism on this method has not yet been clarified. In this work, we have paid attention to behavior of 2,4,5-trichlrorophenol, since this is the most generative precursor of 2,3,7,8-TCDD of the highest toxicity among PCDDs congeners. Such a study that clarifies the decomposition reaction mechanism of 2,4,5-trichlrorophenol may give us important knowledge for advancing the dioxin emission control. Thus, we have studied the decomposition mechanism of 2,4,5-trichlrorophenol using ab initio molecular orbital method calculation.

9:30 AM
Scrap Combination for Recycling Valuable Metals: Direct Extraction and Recovery of Neodymium Metal from Magnet Scraps: *Toru H. Okabe*[1]; Osamu Takeda[2]; Kazuhiro Fukuda[3]; Yoshiaki Umetsu[3]; [1]The University of Tokyo, Inst. of Industl. Sci., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8505 Japan; [2]Santoku Company, Ltd., 4-14-34, Fukae-Kitamachi, Higashi-nada-ku, Kobe 658-0013 Japan; [3]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls. (IMRAM), 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

A fundamental study was conducted with the purpose of establishing an environmentally sound recovery process for recycling valuable metals by combining scraps containing valuable metals. As an example, we investigated direct extraction and recovery of neodymium (Nd) in magnet scraps by utilizing liquid metal as an extraction agent. It was found that liquid magnesium (Mg) or silver (Ag) are suitable extraction agents, and can extract Nd out of iron alloys with high efficiency at around 1100 K. The newly developed extraction apparatus for Nd is a mechanically simple, static device without moving parts, where the Mg extraction medium circulates due to temperature difference inside the reaction vessel. This developed device can simultaneously accomplish continuous extraction of metal Nd from scraps, re-extraction of Mg from Mg-Nd alloy, and eventual recovery of pure Mg. As a result, metal Nd with 98% purity was directly recovered from magnet scraps under certain conditions. It was also shown that the extraction agent Mg could be reused. The results of this investigation show the possibility of establishing a procedure for directly extracting metal Nd in pure metal form without oxidization.

9:55 AM
Distribution Behaviors in Recycling of Copper from the Waste: *Junzo Hino*[1]; [1]Toho Tinanium Company, Ltd., Titanium Div., 3-3-5 Chigasaki, Chigasaki, Kanagawa-Pref. 253-8510 Japan

Most of the waste that contains a small portion of copper is landfilled. The recycling process of copper from the waste, such as shredder residue, should be developed in order to save mineral resources and prevent environmental pollution. Shredder residue generated from disposed automobiles and electric appliances consists of many types of plastics, glasses and metals. The combustion heat of plastics is efficiently used for melting in the recycling process, which consists of melting and reduction. The knowledge of distribution behaviors of main components is available to develop the process, because the waste contains a lot of different kind of components. Distribution behaviors in the recycling process are evaluated by using the thermodynamic calculation program, HSC Chemistry. The calculation results are compared with the practical operation data of the recycling process and discussed.

10:20 AM Break

10:35 AM Invited
Environmental Policy and Continuous Improvement in Atlantic Copper: *Jes´s Contreras*[1]; Adelino Alonso[1]; Pedro Hidalgo[1]; Miguel Palacios[1]; [1]Atlantic Copper S.A., Huelva 21001 Spain

Since the commencement of its activities in Huelva in 1970, Atlantic Copper has maintained a course of action that has been more demanding than the successive environmental legislations current at different times. As its primary objective it has sought compatibility between the protection of the environment and both its industrial activity and the social and economic development of the area in which its Metallurgical Complex is located. For this reason, since the seventies it has maintained a continuous programme of investments which have allowed it to attain its present environmental situation. As a consequence of the Environmental Policy of Atlantic Copper, in April 1998 it obtained certification for its Integrated System of Environmental Management (SIGMA) according to international standard ISO-14001, and under European Community Regulation 1836/93 for the Eco-Management and Eco-Audit System (EMAS). The SIGMA formalises the environmental management practices of Atlantic Copper and engages it to have available a system that can be fully audited. Atlantic Copper was the first European company in the non-ferrous metallurgical sector to obtain both the UNE-EN-ISO 14001 certification and the EMAS registration. The present paper reviews the actions by Atlantic Copper in environmental matters that have allowed it to achieve its present position. It also describes the coming stages of improvement planned and the modifications to be carried out in the near future, in particular the criteria for the selection of technologies on the basis of the concept of BAT (Best Available Techniques) introduced by the European Union, and always guided by the final objective of converting Atlantic Copper into a reference within the sector, comparable to the best designed and operated installations in the world.

11:05 AM Invited
Phytoremediation Potential of Several Plants for Nickel Contaminated Soils: *A. Cullaj*[1]; A. Hasko[2]; Florian Kongoli[3]; [1]University of Tirana, Dept. of Chem., Tirana Albania; [2]Agricultural University of Tirana, Dept. of Agronomy, Tirana Albania; [3]FLOGEN Technologies, Inc., 5757 Decelles Ave., Ste. 511, Montreal, Quebec H3S 2C3 Canada

Several industrial sites suffer from the contamination of soils from heavy metals, which are emitted among others by anthropogenic mining and metallurgical activities. Effective and economic physicochemical technologies for remediation of these sites remain elusive and costly. A new alternative remediation technique is the so-called phytoremediation. This is based on the ability of some plants to accumulate very high concentrations of metals from soils and thus providing the basis for a remediation of the contaminated sites. This technique, is an emerging branch of natural biotechnology and has several advantages compared to the sophisticated physicochemical techniques of soil remediation. It is not only environmentally friendly and pleasing to the eye but also its costs are quite low since it is solar driven, and, in some cases, plants can accumulate metals to such levels that economic mineral recovery maybe feasible even in conventional Ni refinery or smelting operations. In this work, the potential of many plants to accumulate nickel has been investigated in order to identify the species which offer the best phytoremedial potential for nickel contaminated soils. Field surveys have been made in five nickel-containing sites in order to identify the nickel tolerant species that have spontaneously grown in contaminated soils. Atomic Absorption Spectrometry measurements were carried out on 145 different plants collected. 16 of them were identified as having an hyper ability to accumulate nickel since they contained more than 10 000 mg Ni per kg (DW). Seven taxa are of Alyssum genus and one of Bornmuellera genus of Cruciferae. The highest accumulation of nickel was present in aerial parts of Alyssum murale var. chlorocarpum Hausskn (25 500 mg/kg) and Alyssum markgrafii O.E. Schulz (23 700 mg/kg). The seeds germinated are more evidenced at A.m.var. chlorocarpum, about 63%. These plants are suggested as the most promising species to be used for phytoremediation purposes in nickel contaminated soils.

11:35 AM
Development of Roasting Process for Fly Ash from Municipal Incinerators: *Mototsugu Matsuno*[1]; Katsuhiro Tomoda[1]; Junnichi Takahashi[1]; [1]Sumitomo Metal Mining Company, Ltd., Energy & Environ. Business Div., 5-11-3 Shinbashi Minato, Tokyo 105-8716 Japan

Several types of process have been developed for treating fly ash generated from municipal incinerators. The incinerator fly ash contains not only toxic metals such as lead and cadmium, but also dioxins. A new process was examined in the present study. Fly ash is formed into green pellets with some additives, and the pellets are then roasted to eliminate or stabilize the toxic metals, and to decompose dioxins. The roasted pellets become harmless through this process, and are recycled as artificial lightweight aggregates. Volatilized metals are collected as secondary fly ash, and recovered at non-ferrous smelting plants. A new pilot plant of this process, with a capacity of approximately 100 kg/hr, was installed and several types of test were carried out. The characteristics of this process technology are complete reduction of heavy metals, strict stabilization of residual metals and high-strengthen pellets production by sintering. Mechanism of roasting is also analysed.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: Lead-Zinc

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday AM — Room: Santa Rosa
March 4, 2003 — Location: San Diego Marriott Hotel

Session Chairs: Yong Hack Lee, Korea Zinc Company Ltd., Seoul Korea; Andreas Siegmund, RSR Technologies Inc., Dallas, TX 75207 USA

8:30 AM Keynote
Modern Applied Technologies for Primary Lead Smelting at the Beginning of the 21th Century: *Andreas Siegmund*[1]; [1]RSR Technologies, Inc., 2777 Stemmons Freeway, Ste. 1800, Dallas, TX 75207 USA

During the decade of the nineties the conventional method of primary lead smelting (sinter machine/blast furnace) was successfully challenged by the introduction of novel direct and continuous smelting processes. These modern technological innovations like the QSL, Kivcet, and Isa/Ausmelt process, became more than major competitors and in combination with a significant shift in market structure as well as more stringent government regulations, caused radical changes in many different aspects. They have proven to be economically and environmentally viable. The intensification of the metallurgical reaction by applying the bath or flash smelting principle in conjunction with the usage of oxygen resulted in cost savings and a higher flexibility with respect to raw materials and additives usage. By synchronizing individual auxiliary plant sections with the smelting process nearly optimum energy exploitation, a virtually waste-free production and low-emission mode of operation is achieved. The primary lead industry is at a crossroad where novel high-energy efficient technologies will gradually substitute the sinter-blast furnace operation in the new millennium.

9:05 AM
Thermodynamic Study on Recovery of Lead and Antimony from a Used Lead-Battery: *Satoshi Itoh*[1]; Atsushi Kikuchi[1]; Mitsuhisa Hino[2]; [1]Tohoku University, Grad. Sch. of Eng., Dept. of Metall., Aramaki-Aza-Aoba 02, Aoba-ku, Sendai 980-8579 Japan; [2]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Katahira, Aoba-ku, Sendai 980-8577 Japan

Phase equilibria and activities of the components in the liquid lead-antimony-oxygen ternary system, which is of fundamental importance for discussing the recovery of lead and antimony from a used lead-battery, have been investigated at 1173 and 1223 K. The two liquid phases of metal and oxide were observed at the temperatures studied, and the tie lines indicating isoactivities were determined. The activities of PbO, SbO1.5 and Sb2O3 in the PbO-SbO1.5 and PbO-Sb2O3 pseudo-binary systems were then obtained by applying the Gibbs-Duhem equation to the phase relation of metal-oxide equilibrium. Both in the pseudo-binary systems the activities exhibit negative deviations from Raoultís law. The vapor pressures of lead, antimony and lead oxide, antimony oxide were calculated by using the activities of the components in the lead-antimony-oxygen ternary system. As a result, an oxidation of antimony concentrated in an anode slime followed by evaporation as antimony oxide Sb4O6 was found to be considerably effective for the recovery of lead and antimony from a used lead-battery.

9:30 AM Invited
Simulation of Imperial Smelting Furnace Operation Using a Mathematical Model: *Kenji Matsuzaki*[1]; Fumio Tanno[2]; Akio Fuwa[3]; [1]Mitsui Mining & Smelting Company, Ltd., Corporate R&D Ctr., 1333-2 Haraichi, Ageo-shi, Saitama 362-0021 Japan; [2]Metal Economics Research Institute, Japan, 2-6-4 Toranomon, Minato-ku, Tokyo 105-0001 Japan; [3]Waseda University, Dept. of Matls. Sci. & Eng., 3-4-1 Ohkubo, Shinjuku-ku, Tokyo 169-8555 Japan

A mathematical simulation model of the Imperial Smelting Furnace Operation has been developed to clarify the internal state of the furnace and to improve the furnace operation. The model consists of top, shaft and bottom regions. Analysis in the shaft region is based on differential heat and materials balances taking account of reaction kinetics and heat transfer rate, and those in the top and the bottom regions are based on heat and mass balances under steady state condition. Longitudinal distributions of process variables such as temperatures of gas, sinter and coke, gas composition and reaction extend under a practical operating condition are then calculated. The ISF performance under various operating conditions has also been estimated. A new process for zinc smelting has also been proposed and analyzed using this model.

10:00 AM
The Nature of Accretion Formation During Roasting of Zinc Concentrates in Fluidized Bed Furnace: *N. M. Komkov*[1]; V. A. Luganov[2]; [1]D. Serikbaev East-Kazakhstan Technical University, 19 Serikbayev Str., Ust-Kamenogorsk 492010 Kazakhstan; [2]K. Satpaev Kazakh National Technical University, 22 Satpaev Str., Almaty 480013 Kazakhstan

The main operation of obtaining zinc from sulfide raw materials by hydrometallurgical technology is oxidizing roasting of zinc concentrates in fluidized bed furnaces. The technical-economic parameters are determined by the parameters of the roasting process which in their term depend on the composition of initial raw materials, the temperature of the bed and on other factors. Keeping up of the predetermined temperature of roasting is due to the change of specific efficiency as well as to quantity of heat removed from the bed. The efficiency the caissons depends in a high degree on the composition and thickness of the accretion formed on the surface of the caissons. Forecasting the roasting results is impossible unless we know the accretion formation mechanism. The aim of the present paper is the thermodynamic analyses of behavior of the charge compositions under roasting and determination of the potential mechanism accretion formation. The thermodynamic analyses show that under conditions of oxidizing roasting the thermodynamic probability of zinc sulfate formation is higher than the probability of oxide formation till temperature of 873 K is reached. The thermodynamic probability of lead sulfate formation is higher than the probability of oxide formation in the whole temperature range of roasting. The probability of formation of ferrites and silicates of zinc only insignificantly decreases the sulfate stability. The results of investigation of accretion composition formed under roasting of concentrates with the particle size less than 0.02 mm (60%), containing 65% of sphalerite (ZnS), 12% of pyrite and chalcopyrite (FeS_2 and $CuFeS_2$) each, 3% of galenite (PbS), 7% of silica and other non-ore minerals, have shown the following. The composition of accretion varies depending on the depth of the accretion layer. The accretions on the caissons and the thermo-siphons consist mostly of zinc sulfate (more than 50%). The accretions of forchamber consist mostly of zinc oxide. They also contain ferrites and silicates. Formation of sulfate zinc accretion on cold parts of fluidized bed furnace (on the caissons and on the thermo-siphons) may be explained by a lower temperature on the surface and probably by catalytic influence of oxides present at caissons and thermo-siphons surface on the formation of SO_2 and sulfates. The nature of the accretion structure on the hotter parts of the furnace may supposedly be explained by formation of elemental vaporous zinc during roasting.

10:25 AM Break

10:35 AM Keynote

The Experience of Lead Direct Smelting in Korea Zincís Onsan Refinery: *Yong Hack Lee*[1]; Young Min Park[1]; [1]Korea Zinc Company, Ltd., 142 Nonhyon-dong Kangnam-Ku, Seoul 135-749 Korea

Nonferrous smelting processes have been rapidly changed since the beginning of 1980. The copper smelting technology has been changed to the Flash Smelting and MIB process from the conventional Blast Furnace process. In the field of lead smelting, the technology has changed from Blast Furnace Smelting to the Direct Smelting such as QSL and KIVCET. At 1992, Onsan Refinery of Korea Zinc successfully commissioned Direct Smelting QSL technology for lead. The QSL can produce more than 160,000 ton per year of lead bullion from lead concentrate and various scraps. Moreover, since 2000, the TSL (Top Submerged Lance) Technology utilizing the same Direct Smelting process has been successfully commissioned to treat low grade of lead concentrate as well as lead sulfate residue produced from a hydrometallurgical zinc plant. This paper presents and compares the energy flows of lead smelting processes such as a conventional process and the Direct Smelting Processes plotted on the Pb-O-S phase diagram. The differences of enthalpy and free energy change are also discussed based on the each smelting path. The potential diagram of CO/CO_2 suggests how to control the oxygen potential in furnace. Also, it describes the recent operational experience in TSL plant of Onsan Refinery with necessary mass balance of key elements.

11:10 AM Invited

Direct Production of Metallic Zinc from EAF Dust: *Takeshi Azakami*[1]; Hirofumi Sugimoto[2]; Sachio Kojima[2]; [1]Saitama Institute of Technology, Grad. Sch. of Eng., Okabe, Saitama 369-0293 Japan; [2]Sotetsu Metal Company, Ltd., Bandai 1414, Bandai, Fukushima 969-3301 Japan

In Japan, more than 500 thousand tons of EAF dust is generated every year. Zinc source in the dust reaches 17 to 25% of annual zinc production in Japan. Most of this zinc in EAF dust has been recovered as crude zinc oxide by carbon reduction in rotary kilns or other furnaces. Produced crude zinc oxide is valued very cheap because it has to be treated again as a raw material of zinc pyrometallurgy. With the view of direct production of metallic zinc, fundamental experiments were carried out by applying ìIron Reduction Vaporization Methodî. The purpose of the study is to cut down the treatment costs and to make more profit on dust treatment process. Also, by this process, decreasing in CO_2 generation would be attainable. By larger scale tests, zinc ingots have been produced, and industrialization of the process will be hopefully expected.

11:40 AM

Installation of Arsenic Removal into the Hematite Process: Hitoshi Msauda[1]; Shigeki Sato[1]; Yoshito Kudo[1]; *Yutaka Shibachi*[1]; [1]Akita Zinc Company, Ltd., Hematite, 217-9 Shimo-Kawabata Furumichi Iijima, Akita 011-0911 Japan

The Iijima Zinc Refinery started operation in 1972, and has an annual capacity of 200,000t-Zn today. The hematite process was employed for the first time and we have been pursuing the ideal of waste-free refinery for 30 years. In recent years, however, only a half of the hematite produced could be sold to the cement industry and reducing arsenic level had been anticipated for selling them all. The arsenic cementation process with zinc powder, which was developed in the long time laboratory works, was determined best for hematite process among some options. The construction works started in 2000 and was put into operation in 2001, and all hematite were sold to the cement industry in the same year. The outline of this project, present operation and technical issue will be reported in this paper.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: General II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday AM Room: Point Loma
March 4, 2003 Location: San Diego Marriott Hotel

Session Chairs: Ramana G. Reddy, The University of Alabama, Dept. of Metallurgl. & Matls. Eng., Tuscaloosa, AL 35487-0202 USA; Brajendra Mishra, Colorado School of Mines, Metallurgl. & Matls. Eng., Golden, CO 80401 USA

8:30 AM Keynote

Application of Molten Salts in Metals Production: *Brajendra Mishra*[1]; [1]Colorado School of Mines, Metallurgl. & Matls. Eng., 1500 Illinois St., Golden, CO 80401 USA

Molten Salt electrolytic processes offer unique opportunities to extract and refine metals where gaseous or metallothermic reduction, hydrometallurgical extraction and aqueous electrolytic methods are thermodynamically constrained. Production of aluminum and magnesium by molten salt electrolysis are well known commercial processes. Several other reactive metals, such as lanthanides and actinides, as well as beryllium and calcium, make use of molten salt processing for extraction and refining. This presentation describes the science of molten salt chemistry and electrochemistry for winning and refining of several metals. In addition, recovery of metals from waste process salts by molten salt reduction as well as oxidation have been discussed. Material issues in design of molten salt reactors have been included. Various applications have been presented through case studies. Experimental data have been included to justify the suitability as well as limitations of these specific processes.

9:05 AM

Thermogravimetric Study of the Sulfurization of TiO_2 Using CS_2 and H_2S: *Nobuaki Sato*[1]; Jhon Cuya[1]; Katsutoshi Yamamoto[1]; Atsushi Muramatsu[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

Titanium dioxide is the well-known catalyst for the photodecomposition of water in uv region. On the other hand, titanium sulfides absorb the visible light, though it is rather unstable in water because of its photo-dissolution. In this regard, partially sulfurized TiO_2 is expected to be chemically stable and have an enhanced photocatalytic activity compared with TiO_2. For the analysis of sulfurization behavior of TiO2, thermogravimetric study on the sulfurization of TiO2 was made using a thermo-balance with a quartz spring. In the case of CS2, sulfurization reaction started at around 773 K with heating rate of 1 K/min. The weight increase curve showed a maximum of c.a. 30 wt% at around 1023 K and then gradually decreased to c.a.12 wt% at 1273 K. This would be caused by the formation of TiS2 followed by the decomposition of TiS2 to lower sulfides. The sulfurization behavior of TiO2 using H2S was also investigated compared with that of CS2.

9:30 AM

Decomposition of Sulfide Concentrates Under Concentrated Solar RadiationñA Novel Approach to Effect the Direct Decomposition of Sulfides: Lenny Winkel[1]; Christine Guesdon[1]; *Marcel Sturzenegger*[1]; [1]Paul Scherrer Institut, High-Temp. Solartech., OVGA/103A, Villigen PSI 5232 Switzerland

The decreasing demand for sulfuric acid as well as attempts to reduce CO_2 emissions stimulated research activities aiming at new process routes for metal extraction. The most intriguing approach to avoid formation of sulfur dioxide (SO_2) and that of intermediate oxides is the direct conversion of metal sulfides into the metal and elemental sulfur. A promising path to realize the direct conversion is the use of concentrated solar radiation. Thermodynamic calculations suggest that many non-ferrous metal sulfides decompose into the metal and sulfur at temperatures between 1300 and 2000°C. Such temperatures are readily accessible in solar chemical reactors and metal extraction with high maximum efficiencies and reduced or zero emission of SO_2 and CO_2 can be envisioned. Experimental work on solar metal extraction has been initiated for zinc and copper. Chemical reactivity studies by means of thermogravimetry and powder x-ray diffractometry have shown that extraction of copper is easier to effect than that of zinc: During the decomposition of copper sulfides gaseous sulfur naturally separates from liquid copper, while decomposition of zinc sulfide generates a vapor with zinc and sulfur being mixed. To prevent zinc and sulfur from reacting back to the parent zinc sulfide, additional measures, e.g. fast cooling, are required. Chemical equilibrium calculations have confirmed that the solar decomposition of copper sulfide concentrates provides an additional benefit with regard to removal of volatile impurities. Since the solar decomposition can be conducted in absence of oxygen, slag forming reactions will be suppressed and impurities such as arsenic or antimony can easily be separated from copper metal by evaporation. After a short description of the novel approach the paper will firstly report on the chemical equilibrium calculations carried out for evaluating the potential of impurity removal and secondly present an imaging furnace, set up for studying the decomposition of sulfides at temperatures up to 2000°C.

9:55 AM

Removal of Boron from Metallurgical-Grade Silicon by Applying CaO-Based Flux Treatment: *Mitsuru Tanahashi*[1]; Hideo Nakahigashi[1]; Kunihiko Takeda[1]; Chikabumi Yamauchi[2]; [1]Nagoya University, Dept. of Matls. Sci. & Eng., Grad. Sch. of Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan; [2]Chubu University, Dept. of Mechl. Eng., Sch. of Eng., 1200 Matsumoto-cho, Kasugai, Aichi 487-8501 Japan

In order to develop economical production process from metallurgical-grade silicon (MG-Si) to solar-grade (SOG-Si), removal behavior of boron from molten MG-Si was investigated at 1773 K by two following CaO-based flux treatment processes, which can establish special condition of both high basicity of flux and high oxygen partial pressure at boron removal reaction sites: [1] Boron removal from MG-Si by flux addition onto the molten silicon followed by oxygen gas injection into the melt (Two-stage connecting process). [2] Boron removal from MG-Si by simultaneous injection of flux powders and oxygen gas into the molten silicon (Simultaneous injection process). By these treatment processes, especially simultaneous injection process using a flux powder injection equipment, boron content in MG-Si can be reduced efficiently. Based on the results obtained, optimum conditions for boron removal by the flux treatment were examined from the viewpoints of operating time, oxygen gas flow rate, and so on.

10:20 AM Break

10:40 AM

Sulfur and Oxygen Potential Ratios Prediction in Copper Flash Smelting Plants Using Reddy-Blander Model: *Bora Derin*[1]; Ramana G. Reddy[1]; [1]Istanbul Technical University, Dept. of Metallurgl. & Matls. Eng., Istanbul 80626 Turkey; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., Tuscaloosa, AL 35487-0202 USA

In smelting furnace conditions, copper losses in liquid fayalitic slags are both as entrained matte and as chemically dissolved species. Using Reddy-Blander (RB) model, the entrained sulfur content in the slags and the exact potential ratios of sulfur and oxygen for several copper flash smelting plants were derived. The sulfide capacities (Cs) of six different flash smelting plants multicomponent ($FeO-CuO_{0.5}-CaO-MgO-FeO_{1.5}-AlO_{1.5}-SiO_2$) slags were calculated. The log (PO_2PS_2) versus matte grades of the industrial furnaces was determined. The PO_2PS_2ratio increases with an increase of copper grade and for the copper flash smelting condition, the calculated PO_2PS_2 ratios were found to be in very good agreement with the experimental and plant data. Hence, the RB Model predicted that sulfide capacity could be calculated a priori, based on a simple solution model and on knowledge of the chemical and solution properties of sulfides and oxides.

11:05 AM Invited

Roasting Mechanisms of Impure Zinc Concentrates in Fluidised Bed: Maija-Leena Mets‰rinta[1]; *Pekka A. Taskinen*[1]; Satu K. Jyrkˆnen[1]; Aija Rytioja[2]; Jens Nyberg[2]; [1]Outokumpu Research, PO Box 60, Pori 28101 Finland; [2]Outokumpu Zinc, PO Box 26, Kokkola FIN-67101 Finland

The behaviour of copper and lead in zinc roasting has been studied in laboratory scale roasting and large scale trials in an industrial fluidized bed, with the background of Outokumpu Kokkola Zinc smelter. Commercial zinc concentrates with a low and high (up to 3 wt %) copper content and with a low and high (up to 3.5 wt %) lead content were oxidized to various desulphurisation degrees in the fluidised bed to establish the reaction mechanisms and the paths for copper and lead oxidation in the roasting. Influences of the agglomerate size on the roasting kinetics and micro-scale phenomena were also determined. Optical microscopy was used on the calcine and the findings are presented as numerous photographs of the polished cross sections. SEM/EDS techniques were used for analyzing chemically the phases and particle morphologies present in the samples. Computational thermodynamics was applied to the complex phase equilibria occurring during roasting in the agglomerating calcine. A complete oxidation of zinc concentrate with a high copper and lead content requires a careful control of oxygen coefficient in the roasting in order to maintain a stable operation of the fluidised bed. Oxygen coefficient thus seems to have a significant impact on the hydrodynamic stability of the fluidized bed with fine raw materials.

11:30 AM Invited

Some Aspects of Cleaning of Ni, Co and Cu-Containing Slags Via Oxide Melt Blowing by Reducing Gases: *L. Sh. Tsemekhman*[1]; A. G. Ryabko[1]; L. B. Tsymbulov[1]; M. V. Knyazev[1]; V. B. Fomichev[1]; A. A. Ryumin[1]; L. A. Pavlinova[1]; [1]AO ìGipronickelî, 1 Grazhdansky Pr., Saint-Petersburg 195220 Russia

One of the methods of cleaning slags containing Ni, Co, and Cu is their blowing by reducing gas mixtures formed in the process of gas or liquid fuel firing. The investigations carried out by us have shown that the acceptable residual Ni and Co content is achieved by means of natural gas combustion at a<0,7 and solid reducer consumption at the level of 6-10%. The values of cleaning are independent of gas-solid fuel consumption ratio and determined by partial oxygen pressure achieved during both fuels oxidizing. The results obtained from cleaning of industrial slags of different compositions by gas mixtures corresponding by composition to various conditions of natural gas combustion are given in the report. The influence of introduced sulphur-containing collector on the cleaning values, as well as the importance of interaction of produced alloys, mattes, slags, and a gas phase are considered.

Actinide Materials: Processing, Characterization, and Behavior: Advanced Fuel Cycles

Sponsored by: Light Metals Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS), LMD-Reactive Metals Committee

Program Organizers: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA; Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Tuesday PM — Room: 4
March 4, 2003 — Location: San Diego Convention Center

Session Chair: David Senor, Pacific Northwest National Laboratory, Richland, WA 99352 USA

2:00 PM Keynote

An Overview of Actinide Processing: Chemical Separations Methods for Recycle and Transmutation: *James J. Laidler*[1]; [1]Argonne National Laboratory, Chem. Tech. Div., 9700 S. Cass Ave., Bldg. 205, Argonne, IL 60439-4837 USA

Commercial nuclear power reactors in the United States presently generate about 2,000 tons of spent fuel each year. At this rate, the accumulated spent fuel in temporary storage at reactor sites will exceed the legislated capacity of the recently-approved Yucca Mountain Repository almost as soon as the repository is opened for spent fuel disposal. The need for a second repository can be precluded by a system of selective chemical separations that partition the spent fuel into (1) a comparatively large mass of uranium that can be disposed cheaply or recycled, (2) transuranic elements that can be recycled into advanced reactors for recovery of their energy value, (3) short-lived fission products with high heat generation rates that can be stored inexpensively until they decay completely, and (4) a relatively small mass of other fission products that must be stored in a high-level waste repository. The separations methods to accomplish this partitioning are presently under development in the United States.

2:40 PM

Electrochemical Reduction of Spent Oxide Fuel: *Karthick Gourishankar*[1]; Lazlo Reedy[1]; Diane Graziano[1]; [1]Argonne National Laboratory, Chem. Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439-4837 USA

Argonne National Laboratory (ANL) has developed a direct electrochemical reduction process for converting oxides to metals with application in the recovery of actinides from spent oxide fuels. The major components of the electrochemical cell are: (1) a molten-salt electrolyte, (2) an oxygen-evolving electrode, (3) a cathode at which solid oxides are reduced, and (4) a reference electrode for monitoring and controlling cell operations. The reduction occurs by a solid-state electrochemical transformation without the need for dissolution of the feed oxide in the electrolyte. Complete reduction of UO2 has been demonstrated using a platinum anode. Our work with UO2 has led to an improved understanding of the reduction mechanism and the parameters affecting the process rate. Bench-scale experiments have been initiated to develop the process for reduction of the transuranic and rare-earth oxides that are present in the spent oxide fuel. In this paper, we will present and discuss results from our recent bench-scale experiments.

3:00 PM

Direct Preparation of Niobium Based Superconducting Alloys by Electro-Deoxidation of the Oxides: Xiao Y. Yan[1]; *Derek J. Fray*[1]; [1]University of Cambridge, Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Niobium based superconductors, Nb, NbTi and Nb3Sn are usually made from the powders of the metallic elements that are expensive and relatively difficult to prepare in the low oxygen form. Electro-deoxidation consists of starting with an intimate mixture of the oxides and making compacted mixture the cathode in a bath of fused calcium chloride and sodium chloride. On the application of a potential below the decomposition potential of calcium chloride, the cathodic reaction is the ionisation of oxygen and its subsequent transfer to the anode as oxygen ions, dissolved in the melt. The metallic product was found to be an alloy or intermetallic compound of the two elements whose superconducting properties were identical to those reported in the literature.

3:20 PM

Recent Advances in Uranium High Throughput Electrorefining: *James L. Willit*[1]; G. A. Fletcher[1]; R. J. Blaskovitz[1]; J. Figueroa[1]; M. A. Williamson[1]; [1]Argonne National Laboratory, Chem. Tech. Div, 9700 S. Cass Ave., Bldg. 205, Argonne, IL 60439-4837 USA

Uranium electrorefining lies at the heart of an electrometallurgical treatment process for spent metallic reactor fuel. The challenges in developing a device that can efficiently electrorefine several tons of uranium per year are in the engineering of the device, not in the fundamental electrochemistry. The fundamental electrochemistry of uranium electrorefining in a molten chloride salt has been understood since the 1950ís. Over the past seven years, we have tested various uranium electrorefiners with capacities ranging from a few kilograms to 150 kilograms. One device, the Mk-V electrorefiner, has been installed in the Fuel Conditioning Facility at Argonne-West and is presently being used to treat spent EBR-II blanket fuel. Results from recent tests that focus on a new anode geometry and the improvement in efficiency arising from a third set of electrodes in the cell will be presented.

3:40 PM Break

4:00 PM

Pyrochemical Separations of TRU and Fission Product Content from Metal Alloy Fuels: *Michael K. Richmann*[1]; William E. Miller[1]; Zygmunt Tomczuk[1]; Diane J. Graziano[1]; [1]Argonne National Laboratory, Chem. Tech. Div., Bldg. 205, 9700 S. Cass Ave., Argonne, IL 60439 USA

A primary goal of the U.S. Advanced Accelerator Applications (AAA) program is the provision of an alternative to the direct disposition of commercial spent nuclear fuel in a repository. This is achieved by converting the transuranic elements and long-lived fission products in this waste to stable or short-lived fission products. A head-end step (UREX) is used to separate the uranium, technetium and iodine from the transuranics (TRU) and other fission products in commercial spent LWR fuel. Pyro-A, a pyrochemical process, is used to partition the transuranics from the fission products to form a transmuter blanket fuel that is a zirconium/plutonium metal alloy. This is accomplished by an oxide-reduction step to metal on the UREX product followed by an electrorefining step to recover the TRU content from the fission products prior to alloy fuel fabrication. We will present data for the Pyro-A process electrorefining experimentation demonstrating the kinetics and degree of TRU recovery.

4:20 PM

Processing Experiences During the Electrometallurgical Treatment of Spent Nuclear Fuel: *Brian R. Westphal*[1]; [1]Argonne National Laboratory, PO Box 2528, Idaho Falls, ID 83403 USA

An electrometallurgical process is currently being developed by Argonne National Laboratory for the treatment of spent nuclear fuel. Sodium-bonded spent nuclear fuel is being processed in a hot cell environment as a demonstration of applicability for the technology to other types of spent fuel. During the process development program, optimal operating conditions for the equipment are being investigated with respect to throughput, product purity, and recovery. A steady increase in the current processing rates to a production level allows for the continual development of the process. Although as yet undetermined, the disposition of the final uranium products will be based on the contribution of impurities, primarily plutonium, to the product. Recovery of the uranium during processing is influenced by both throughput requirements and the secondary waste streams. The results and experiences from the development program will be discussed as well as challenges encountered and their resolution.

4:40 PM

Electrochemical Investigation of the Uranium Exchange Current Density in LiCl-KCl Eutetic: *Valerie Goss*[1]; K. V. Gourishankar[1]; J. L. Willit[1]; [1]Argonne National Laboratory, Chem. Tech. Div., 9700 S. Cass Ave., Bldg. 205, Argonne, IL 60439-4837 USA

The exchange current density (ECD) of uranium has not been adequately documented under various experimental conditions and fuel types. In this study, electrochemical methods were used to measure the ECD of depleted uranium metal in a molten LiCl-KCl salt solution. The experimental set-up included a direct power source, a uranium foil cathode, and a uranium strip as the anode. The electrolyte employed was a LiCl-KCl-UCl3 eutetic molten salt. UCl3 concentrations were 1.36- and 1.83-weight %, and reactions were performed at 450-, 500-, and 550-K inside a helium glovebox. In the electrochemical cell, small currents were applied, and overpotentials were recorded. The open-circuit potentials ranged from 0.1- to 0.4-mV. The experimental data indicates that the exchange current density is approximately 50 mA/cm2 suggesting that electrode reactions involving uranium may not be as fast as previously expected.

5:00 PM
Electrochemical Deoxidation of CaO in a CaO-CaCl2 Melt with a Solid Oxide Membrane: David Lambertin[1]; Laurent Pescayre[1]; Gilles BourgËs[1]; Alice Martin[3]; Michel P. Allibert[2]; Jacques Fouletier[3]; Jean-Claude Poignet[3]; [1]CEA/Valduc, DTMN/SRPU, Centre díÈtudes de Valduc, Is Sur Tille 21120 France; [2]Ecole Nationale SupÈrieure díElectrochimie et díElectromÈtallurgie de Grenoble (Groupe INPG), LTPCM, 1130 Rue de la piscine, BP 75, Saint Martin díHËres 38402 France; [3]Ecole Nationale SupÈrieure díElectrochimie et díElectromÈtallurgie de Grenoble (Groupe INPG), LEPMI, 1130 Rue de la piscine, BP 75, Saint Martin díHËres 38402 France

Oxide reduction by calcium metal in a CaCl2 melt produces metal and calcium oxide (CaO), dissolved in the molten chloride. In order to minimize waste salts, the reacted salts have to be regenerated. It can be done (i) by converting CaO to CaCl2 by chlorination with Cl2 and adding calcium metal or (ii) by the electrochemical deoxidation of CaO (reduction of Ca2+ to Ca and oxidation of O2- to O2). In the first method, the addition of calcium metal increases the amount of reacted salts, and consequently the waste salts, while in the second method the quantity of reacted salts is constant. Finally the electrochemical deoxidation of CaO occurs in one step and the produced oxygen is evacuated. The experimental set up comprised a magnesia crucible containing a CaO-CaCl2 melt at 830∞C, in which was dipped an iron cathode and an yttria-stabilized zirconia tube. The anode was the platinized inner wall of the zirconia tube. A current density of 50 mA/cm≤ with a potential difference of 6 V has been applied during 24 hours. The current efficiency for oxygen production was close to 100%.

Alumina and Bauxite: Bauxite and Alumina Extraction

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: David Kirkpatrick, Kaiser Aluminum & Chemical Group, Gramercy, LA 70052-3370 USA; Steve Rosenberg, Worsley Alumina Pty, Ltd., Process Chemistry Group, Collie, West Australia 6225 Australia; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday PM — Room: 3
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: David Kirkpatrick, Kaiser Aluminum & Chemical Group, Gramercy, LA 70052-3370 USA; Steve Rosenberg, Worsley Alumina Pty Ltd., Process Chem. Grp., Collie, W. Australia 6225 Australia

2:00 PM Introductions

2:10 PM
Recent Innovations in Controlling Fugitive Dust Emissions in the Bauxite/Alumina Industry: *D. Casey Roe*[1]; [1]General Electric, GE Betz, 4636 Somerton Rd., Trevose, PA 19053 USA

Fugitive dust emissions are becoming a growing Global concern. In the bauxite/alumina industry, dust emissions are generated during the mining, transport and handling of bauxite, and in red mud disposal areas. While environmental, health and safety issues are the primary reasons for concern, dust can also have a major economic impact on an operation, e.g., increased maintenance and clean-up costs, and the loss of bauxite prior to digestion. While water can minimize certain dust problems, the results are short-term, and water addition can cause wet bauxite handling problems and increased transportation costs. This paper summarizes recent innovations for reducing dust emissions from bauxite mines to red mud disposal areas. Examples include a method for controlling dust during mining operations, automated equipment and chemicals for controlling bauxite dust, road and surface stabilization, and red mud disposal area dust control.

2:40 PM
Improvements of the Processes to Produce Alumina from Chinese Diasporic Bauxite: *Songqing Gu*[1]; *Zhongling Yin*[1]; [1]Zhengzhou Light Metal Research Institute, No. 82 Jiyuan Rd., Shangjie Dist., Zhengzhou, Henan Province 450041 China

In this paper the characteristics of Chinese diasporic bauxite resource and its main behaviors in the alumina production are analysed. The important aspects of the processes and technology applied in Chinese alumina industry including Bayer process, sintering process and combined process are presented. The potential and direction of technology development of Chinese alumina industry are discussed.

3:10 PM
Investigation on the New Digesting Process of Diaspore: *Zhao Qingjie*[1]; *Yang Qiaofang*[1]; Qi Lijuan[1]; [1]Aluminium Corporation of China, Ltd., Zhengzhou Rsrch. Inst., Zhengzhou, Henan 450041 China

Based on the extensive study on the reaction behaviors and digesting kinetics of silicon & titanium minerals, the features of process itself and equipments in the current high-temperature-intensifying Bayer process are analyzed and compared. It is indicated that there are disadvantages in the current high-temperature-intensifying Bayer process for the complicated diaspore with difficulty to be digested. A new digesting process is proposed, i.e., tube preheating removal of silicon & titanium digestion in retention tank. This new process bears the advantages of the current digesting technology and better suitability for the complicated diaspore. It not only ensures better digesting results and lower energy consumption, but also reduces or eliminates the scales on the heat exchange surfaces, which makes the running period longer and investments smaller.

3:40 PM Break

3:50 PM
Bayer Process Chemical Calculations ìA Systematic Approachî: *Pradeep K. Maitra*[1]; [1]Balco, B-475 Cross St., 24 Smriti Nagar, Bhilai 490020 India

Till early 1990ís not much emphasis was given on productivity of Alumina. Subsequently because of stiff market competitions, the producers of alumina have to impose strict measures to optimise the specific consumption (SC) of raw materials e.g. bauxite, caustic soda, lime, etc. to reduce the cost of alumina production. In European bayer plant where huge quantity of caustic liquor recirculate continuously, it is extremely difficult to verify the process stock by physical method of stock taking, as a result the chemical stock often appeared to be in consistent with respect to its book values. To counter above discrepencies it was opined that the rate of consumption of raw material should be compared with the plant performances and the chemistry of the raw material, intermediate and finished product as well. In absence of National and International guidelines on chemical calculations of bayer process, the calculated values and expressions varies plant to plant. In above context an attempt has been made by the author to publish the simplified version of chemical calculations, so that in future time the same can be used for preparation of guidelines.

4:20 PM
Influence of K2O in Spent Liquor on Bayer Process of Diasporic Bauxite: *Zhonglin Yin*[1]; [1]Zhengzhou Research Institute, Aluminum Corporation of China, Ltd., Alumina Rsrch. Dept., Zhengzhou Light Metal Rsrch. Inst., Shangjie, Zhengzhou, Henan 450041 China

Illite, an impurity mineral existing in diasporic bauxite, causes K2O to accumulate in spent liquor and the concentration of K2O to increase gradually. The influence of K2O in spent liquor on the preheating process of slurry and digestion results is discussed in this paper. K2O in spent liquor can slow down the reaction rate of illite in preheating process of slurry and can reduce the Al2O3/SiO2 ratio Na2O/SiO2 ratio in red mud to a certain extent at some conditions. Therefore, the influence of K2O on the Bayer process must be considered in research studies of the Bayer process for diasporic bauxite.

4:50 PM
Processing of Aluminium Ores and Human Safety: Vadim A. Lipin[1]; Ludmila E. Safarova[1]; *Anatoly V. Sysoev*[2]; Alexander N. Aminov[2]; Valery P. Lankin[2]; [1]Russian National Aluminium-Magnesium Institute (VAMI), 86 Sredny pr., St. Petersburg 199106 Russia; [2]Bogoslovsky Aluminium Plant-JSC SUAL, 1 K. Marx str., Krasnoturinsk, Sverdlovsk Region 624440 Russia

The neutralization of industrial waste, including the alumina refineries was developed to create and improve the water sewerage and gas and dust scrubbing facilities at the final stages of process, less attention was paid to the establishing of waste free technology. The comprehensive way of alkaline alumo silicate ore practically excludes the solid and liquid wastes, typical for Bayer process as well as for conventional technology to produce the by-products. It can be achieved through the entire use of the products involved in the process, wide product range and diversity of thermal and hydro chemical areas.

5:20 PM
New Salicylic Acid Containing Red Mud Flocculants: *Everett C. Phillips*[1]; [1]Ondeo Nalco Company, Ondeo Nalco Ctr., Naperville, IL 60563-1198 USA

New, high molecular weight salicylic acid containing polymers have recently been developed at Ondeo Nalco Co. These new flocculants provide substantial improvements for the clarification of red mud decanter slurries from numerous Bayer plants, worldwide. The preparation and chemistry of these new polymers are summarized and compared to others used in the industry. Also presented are the developments to date, including mud settling, overflow clarity and liquor filtration results from both static and dynamic laboratory tests. The lab results demonstrate that the new salicylic acid containing flocculants can be applied without the need for starch, as is the case for traditional 100% polyacrylate polymers. A recent plant evaluation has confirmed this result.

Aluminum Reduction Technology: Modeling

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Martin Segatz, VAW Aluminum Technology, Bonn D-53117 Germany; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday PM — Room: 6B
March 4, 2003 — Location: San Diego Convention Center

Session Chair: Don Ziegler, Alcoa Inc., Alcoa Tech. Ctr., Alcoa Ctr., PA 15069-0001 USA

2:00 PM
An Analytical Model for Cathode Voltage Drop in Aluminum Reduction Cells: *Richard M. Beeler*[1]; [1]Alcoa Inc., Hall Proc. Improv., 100 Technical Dr., Alcoa Ctr., PA 15069-0001 USA

This paper presents the derivation of an analytical model for cathode voltage drop (CVD) for typical cathode assemblies in aluminum reduction cells. The model includes an estimate of the current density profile on the cathode surface; the voltage profile along the length of the [current] collector bar; and a breakdown of total CVD into components between the carbon block, the collector bar, and the bar-block joint. Comparisons with a three dimensional finite element model are presented for evaluation of the severity of geometric simplifications. Model results, with joint resistance tuned to match typical plant CVDs, are presented to illustrate the distribution of total CVD between carbon blocks, collector bars, and bar-block joints.

2:25 PM
START-Cuve: Thermo-Electro-Mechanical Transient Simulation Applied to Electrical Preheating of a Hall-HÈroult Cell: *Martin DÈsilets*[1]; Mario Fafard[2]; Daniel Marceau[3]; [1]Alcan International Ltd., Arvida R&D Ctr., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada; [2]Laval University, Sci. & Eng. Fac., Adrien-Pouliot Bldg., Sainte-Foy, Quebec G1K 7P4 Canada; [3]University of Quebec at Chicoutimi, Appl. Sci. Dept., 555 Blvd. Universite, Chicoutimi, Quebec G7H 2B1 Canada

The phenomena taking place during the start-up of an aluminum reduction cell are complex and difficult to analyze. Experimental tests on industrial cells are difficult, time-consuming and expensive. Mathematical modeling offers here a good way to study the impact of design and start-up methods on thermo-mechanical equilibrium in a cell. An overview of the research program, called START-Cuve, involving industrial and university researchers, will be given. The material characterization part will be briefly described. The finite element modeling approach of the research program will then be developed more thoroughly. The numerical resolution of the fully coupled thermo-electro-mechanical problem, solved with a finite element code able to cope with complex constitutive laws, is a great challenge. In this paper, the architecture of the software is presented. The emphasis will be put on the multi-physic resolution in the context of simulating an electrical cell preheat, with a focus on three aspects: multi-field material constitutive laws, thermo-electro-mechanical contact between surfaces and numerical resolution of the problem. Finally, we present how finite element analysis can advantageously be combined with experimental test results to identify specific parameters using reverse engineering approaches.

2:50 PM
Performing Fast Trend Analysis on Cell Key Design Parameters: *Marc Dupuis*[1]; Warren Haupin[2]; [1]GeniSim, Inc., 3111 Alger St., Jonquiere, QC G7S 2M9 Canada; [2]Consultant, 2820 Seventh St. Rd., Lower Burrell, PA 15068-3717 USA

Four fast algebraic models that respectively calculate the anode panel heat loss, the cathode bottom heat loss, the anode voltage drop and the cathode voltage drop have been developed, compared with more complex ANSYSÆ based 3D thermo-electric finite element models and incorporated into the Dyna/Marc 1.7 lump parameters+ cell simulator. With the addition of those four algebraic models to the lump parameters+ cell simulator, it is now possible to perform fast trend analysis on key design parameters like the anode studs diameter, the stud holes depth, the collector bars size or the type of cathode blocks. In order to illustrate the increased power of the lump parameters+ cell simulator as a brainstorming session ìwhat ifî tool, the previously published retrofit study of a 300 kA cell into a 350 kA cell is repeated, this time using only the lump parameters+ cell simulator as modeling tool.

3:15 PM
Modeling of the Coke Bed Used in the Electrical Preheat of an Aluminum Reduction Cell: *Patrice Goulet*[1]; Carl Laberge[2]; RenÈ Lacroix[1]; Laszlo Kiss[2]; Jean Perron[3]; [1]Laval University, Sci. & Eng. Fac., Adrien-Pouliot Bldg., Sainte-Foy, QuÈbec G1K 7P4 Canada; [2]University of Quebec at Chicoutimi (UQAC), Appl. Sci. Dept., 555 Blvd. de líUniversitÈ, Chicoutimi, QuÈbec G7H 2B1 Canada; [3]Alcan International Ltd., Arvida R&D Ctr., PO Box 1250, JonquiËre, QuÈbec G7S 4K8 Canada

In the electrical preheat of aluminum reduction cell, deformations and stresses in all components of the cell are principally due to the temperature gradients. The temperature distribution is essentially established by the Joule effect in the electrical conductors. Furthermore, electrical current distribution is strongly dependent on the use of petroleum coke bed, playing the role of electrical resistor, placed between anode and cathode panels. The thermoelectric behavior of the bed is then an important factor on the preheat quality and must be included in a complete cell preheat model. The behavior of granular materials is usually represented by effective properties. However, it was observed experimentally that the contact quality between coke bed and a plane surface like anode blocks has an important effect on the electrical current and temperature distributions. In this respect, both thermal and electrical constitutive laws have to be developed. Constitutive laws parameters are calibrated by comparing experimental data with finite element model results using new contact elements.

3:40 PM Break

3:50 PM
The Effect of Ambient Temperature and Ventilation on an Electrolysis Cellís Shell Temperature, Heat Balance and Ledge: *Elin Haugland*[1]; *HÂvard B□rset*[1]; HÂvard Gikling[1]; Helge H□ie[2]; [1]Hydro Aluminium AS, Tech. Ctr. ≈rdal, PO Box 303, ÿvre ≈rdal 6884 Norway; [2]Hydro Aluminium AS, Karm□y Metal Plant, HÂvik 4265 Norway

The influence of ambient temperature and ventilation on an electrolysis cellís shell temperature, heat balance, and ledge thickness has been studied. Calculations have been carried out, using the commercial code FLUENT, to study the effect of variations in the ambient temperature on the shell temperature. The effect on the heat balance and the ledge has been calculated by means of Hydroís heat balance simulation model SMASH. Measurements have been carried out by placing thermocouples at the shell wall and in the ledge. A close correlation between the shell temperature and the ambient temperature has been found. No correlation has been found between ambient temperature and the total heat flux from the cell. Variations observed in heat flux can be traced to incidents at the cell; like anode changes, anode effects, changes in the reference resistance etc. The effect on the shell temperature, local heat flux and ledge thickness when the shell is cooled by means of air lances has also been studied. For a cell with ledge thickness 5-10 cm a relatively high decrease in shell temperature is needed before any changes in ledge thickness can be measured.

4:15 PM
Minimization of Outward Wall Deformation of Cathode Casing Steel Structure During Operation: *M. M. Megahed*[1]; H. S. Sayed[1]; Sh. A. Mohamed[1]; A. Abdel-Al[1]; F. M. Ahmed[1]; [1]Cairo University, Mechl. Design & Prod., Egyptalum, Nag-Hammadi Egypt

The paper describes the elaborate effort devoted at Egyptalum smelter toward minimizing the outward wall deformation during operation while keeping the weight of the cathode steel casing to a minimum. Starting from an existing pilot casing design, 16 design parameters are identified as the key controlling parameters affecting cathode structural behavior. Deformation and weight minimization is achieved through conducting consecutive systematic non-linear finite element structural analysis. A total of 17 consecutive design trials is investigated with each design trial stemming from its predecessor. The finite element analysis takes into account carbon swelling and its variation with time, plastic yielding and deformation of steel and its dependence on temperature. The finite element model is built using shell, beam and gap elements to represent the cathode steel structure and truss elements to represent the cathode carbon. Carbon swelling analysis is conducted using Dewing model with new parameters identified to reflect cathode technology employed at Egyptalum. The analysis is continued in real time up to a life of 1055 days of cell operation. Following each design trial, cathode weight, peak outward deformations of longitudinal and transverse walls were monitored and compared with earlier trial. On this basis, the key influencing structural parameters are identified and modified to reduce weight and deformations. The final cathode casing design exhibited almost the same weight as the original pilot design but with much less peak deformations. The results indicate that after three years of operation, the peak outward deformation of the longitudinal wall is about 60% of the corresponding value in the original pilot design. Similarly, the peak outward deformation of the transverse wall was about 34% of the corresponding value in the original pilot design.

4:40 PM
Thermo-Chemo-Mechanical Aspects of Refractory Concrete Used in a Hall-HÈroult Cell: *Daniel Richard*[1]; Mario Fafard[1]; Martin DÈsilets[2]; [1]Laval University, Sci. & Eng. Fac., Adrien-Pouliot Bldg., Ste-Foy, QuÈbec G1K 7P4 Canada; [2]Alcan International Ltd., Arvida R&D Ctr., PO Box 1250, JonquiËre, QuÈbec G7S 4K8 Canada

As part of the on-going project START-Cuve, involving the thermo-electro-mechanical modelling of a P-155 coke bed electrical preheating, the behaviour of the pier insulating refractory concrete had to be assessed. Preliminary experimental work has shown that even before preheating, large cracks were present in the concrete actually used for that technology. It was found that concrete hydration and drying caused shrinkage; also, the hydration reactions in calcium aluminate cement are extremely exothermic in nature, leading to substantial temperature gradients and free water vaporisation in the concrete while hardening. Therefore, the initial conditions in the pier concrete are rather inhomogeneous and complex to predict. As a first step, a macroscopic thermochemical constitutive law was used to represent the hydration reactions and their thermal effects. The hydration kinetics was reverse engineered by comparing experimental measurements in a quasi-adiabatic setup to a finite element model results. An experimental representative setup was then instrumented and modelled to validate the approach. A good agreement between the numerical and the experimental results was found.

5:05 PM
On Some Features of Thermal State of HSS Cells: *A. V. Sysoev*[1]; A. N. Aminov[1]; N. V. Markov[1]; G. S. Pryakhin[1]; T. V. Mezhberg[1]; [1]Bogoslovsky Aluminium Plant Russia

In 1997-2000 Bogoslovsky Aluminium Plant (Siberian-Ural Aluminium Company), equipped with HSS Soderberg cells, implemented an intensive technology in one potroom. The production rate increase amounted to 13.2%; in addition, specific consumptions of raw materials and power were reduced. Unusual technological features of the intensification were as follows: simultaneous increase of current density i‡ and current efficiency Ái; increase of Ái with increase of electrolyte overheating ƒT, electrolyte temperature decrease "e at increase of thermal losses in anode-to-cathode distance (ACD). Managerial and technological measures, providing the achievement of these performances, were based on optimization of the cell parameters and thermal balance. The investments were minimal, the repayment period was less than one year. The abnormality of the aforementioned features required thorough investigations on the peculiarities of distribution of heat sources and sinks in a cell by means of applied aspects of thermodynamics and electrochemical kinetics. Heat exchange mechanism is studied in ACD and at interface boundaries on the basis of the works by Polyakov P.V. and Mozhaev V.M., as well as Flem B.E. (L.M., 1996), Xu Q and Kjelstrup S (L.M., 1998). Under the cell normal state, when the Peltier heat at the anode completely compensates the heat of overvoltage of anode reaction (TƒS > ÁaI), the thermal flow from electrolyte to the anode is transferred mainly via anode side walls wetted with electrolyte. The areas of anode sole free from gaseous bubbles are characterized with very low transfer coefficient in comparison with the areas of anode walls and bubbled areas of anode sole. As the place of anode effect is approached (TƒS < ÁaI) a powerful heat source appears at the interface. Many abnormal features of anode effect occurrence can be explained by interference of physical phenomenon of intensive electrolyte evaporation into the electrochemical process. Analytical methods of the cell thermal state are developed on the basis of individual heat balances of ACD, anode and cell. The estimated value i‡ agrees with the actual one only in the case of application of the ACD heat balance and elimination of equilibrium voltage of decomposition (~1.19 V) from heating voltage. Comparative estimation of the features of heat losses from ACD of HSS, VSS Ë prebaked cells, highlighting the abnormal features of the intensive technology has been made.

Carbon Technology - II

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Amir A. Mirchi, Alcan Inc., Arvida Research and Development Centre, Jonquiere, QC G7S 4K8 Canada; Don T. Walton, Alcoa Inc., Wenatchee Works, Malaga, WA 98828-9784 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday PM — Room: 6D
March 4, 2003 — Location: San Diego Convention Center

Session Chair: Juan Romero, Alcan Inc., Alcan Primary Metal Grp, Robards, KY 42420 USA

2:00 PM Cancelled
The Effect of Al-Containing Additives on Air/CO2 Reactivity of Carbon Anode in Aluminum Electrolysis: *Yanqing Lai*[1]; Qingyu Li[1]; Jie Li[1]; Jianhong Yang[1]; *Yexiang Liu*[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

2:25 PM
Drilling of Stub Holes in Prebaked Anodes: *Hogne Linga*[1]; [1]Hydro Aluminium, HAL Tech. Ctr. ≈rdal, PO Box 303, Ovre Ardal NO-6882 Norway

The stub holes may be a troublesome part in the production of anodes. Tension and cracks can be created around the stub hole. Vibrating the stub holes often introduce limitations for the optimisation of the production process. Stub holes oriented towards the cassette wall during baking will be filled with packing material. This requires the stub holes to be thoroughly cleaned after baking. Stub holes which are oriented away from the cassette walls, are unprotected during the baking process. This may lead to an increased rejection rate or additional costs to protect the stub holes. The paper will present four years of experience with drilling of the stub holes after the baking of the anodes. This technology allows for precision drilling of different designs of stud holes, and the paper will summarize the results from trials where three different designs are tested in the same reduction cell.

2:50 PM Cancelled
Survey on Worldwide Prebaked Anode Quality: *Raymond Perruchoud*[1]; Kirstine Hulse[2]; Markus Meier[1]; Werner Fischer[1]; [1]R&D Carbon, Ltd., Sierre 3960 Switzerland; [2]Alusaf Empageni S. Africa

3:15 PM
The New AP-FCBA Paste Plant Technology: *Christian Dreyer*[1]; *Corinne Jouault*[1]; AndrÈ Pinoncely[2]; Jean-FranÁois Andre[2]; [1]Aluminium Pechiney, LRF-BP 114, Saint Jean de Maurienne 73300 France; [2]FCB Aluminium, 32 Rue Fleury-Neuvesel, BP24-69702 Givors 69702 France

Aluminium Pechiney and FCB Aluminium jointly developed a new production process for the dry mix preparation. This new flow sheet reduces significantly both the investment and operating costs of a Paste Plant and allows for the production of an optimized G/S ratio. Based on promising preliminary Research & Development results and in order to fully validate such an innovative process, AP and FCBA invested in a full-scale trial program. The 35 tph industrial scale Pilot Plant which was built at the Aluminium Dunkerque smelter in Northern France has been successfully started in July 2002. This paper recalls the operating philosophy of that new plant and highlights the promising first technical results on anode quality obtained after the start up.

3:40 PM Break

3:50 PM
Alcan Alma New Anode Paste Plant Start-Up and Early Operation: *Claude Lavoie*[1]; Emmanuel Bergeron[1]; AndrÈ L. Proulx[2]; [1]Alcan Inc., Alcan Primary Metal Grp., Alma Works, 3000, Des Pins Ouest Ave., Alma, Quebec G8B 5W2 Canada; [2]Alcan Inc., Alcan Primary Metal Grp, 1955, Mellon Blvd., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada

Alcan announced in February 1998 the construction of an aluminium smelter in Alma, Quebec. The Alma smelter project was part of Alcanís corporate growth strategy and its Quebec smelter rebuilt program. For the anode paste plant, new principles and ideas originated from Alcan people experiences brought a new design. The new paste plant, an Alcan design, would be different compared to others in the aluminium industry. The Alma plant mixing line combines a discontinuous/continuous process based on three Eirich mixer/cooler units. The team preparation, the equipments commissioning and the start-up occurred in 2000-2001. The production ramp-up and the anodes quality were achieved in a short period of time despite necessary modifications. This paper will describe the Alcan Alma paste plant, its start-up and early operation.

4:15 PM

New Plant for Anode Paste Conditioning and Anode Pressing with a Hydraulic Vacuum Press: *Alfred Kaiser*[1]; [1]Laeis Bucher GmbH, Plant Eng., PO Box 8065, Trier D-54181 Germany

A new concept is presented for production of high quality anodes including innovative paste conditioning, dosing and mold filling system and a high performance hydraulic press with vacuum system. All components starting from outlet of the paste mixer are adjusted to provide a throughput of up to 60 large or 120 small anodes/h. Changes in paste properties are detected by the press control that immediately adjusts the paste conditioning/cooling mixer parameters. The precision of dosing system and press allows for an outstanding accuracy of anode height (?b 2 mm) with a very good reproducibility and an even density distribution within the anodes. The low pressing temperature of 115 to 130¢XC assures stable green blocks without water cooling and reduces emission of pitch volatiles (PAH etc.) Further advantages are low noise level (< 76 dBA), high flexibility and availability (e.g. mold change in less than 0.5 h) and low maintenance.

4:40 PM

Paste Granulometry and Soderberg Anode Properties: Morten Sorlie[1]; *Turid Vidvei*[1]; [1]Elkem Aluminium ANS Research, PO Box 8040, Vagsbygd, Kristiansand N-4675 Norway

There are almost as many different soderberg paste recipes as there are soderberg smelters, and most of them seem to perform satisfactory for the cells and cell operating parameters the pastes are made for. The baked anode properties may, however, be optimized by changes made to individual fractions and the total sieve curve. The paper discusses how green and baked soderberg anode properties change as the recipe particle distribution is changed, with special emphasis on the fineness of the fine fraction and the form of the total sieve curve.

5:05 PM

The Technology Development of the Carbon Materials Industry for Aluminium Electrolysis in China: *Fengqin Liu*[1]; [1]Zhengzhou Light Metal Research Institute, Dept. of Aluminum Electrolysis & Carbon, No. 82 Jiyuan Rd., Shangjie Dist., Zhengzhou, Henan Province 450041 China

The characteristics of raw material resources for the carbon material industry in China are discussed in this paper. The main production processes and equipment used to manufacture prebaked anodes, semi-graphitized cathodes, graphite cathodes and cathode paste in the industry are reviewed. The development trend and future of Chinese carbon material industry are pointed out in the paper as well.

Cast Shop Technology: Filtration/Metal Treatment

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; David H. DeYoung, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Tuesday PM March 4, 2003 — Room: 6C — Location: San Diego Convention Center

Session Chairs: Corleen Chesonis, Alcoa Inc., Alcoa Techl. Ctr., Alcoa Ctr., PA 15069 USA; Peter Waite, Alcan Inc., Arvida R&D Ctr., Jonquiere, QC G7S 4K8 Canada

2:00 PM

Study of Al Degassing by Hydrogen Analysis with Supersonic Expansion Mass Spectrometry: Marc Bertherat[2]; Elisabeth Blanquet[3]; *Michel Paul Allibert*[1]; Pierre Le Brun[4]; [1]Consultant, 23 Ave. Marcellin Berthelot, Grenoble 38100 France; [2]Aluminium Dunkerque, ZIP Ouest, BP81, Loon Plage 59279 France; [3]INPGrenoble, ENSEEG, BP75, St Martin díHeres 38402 France; [4]Pechiney Centre de Recherches de Voreppe, 725 rue Aristide Berges BP27, Voreppe 38341 France

The hydrogen extraction kinetics from liquid Aluminum by Argon degassing was studied using a mass spectrometer analyzing the out coming Argon, after supersonic expansion. This technique has a response time of less than 1 minute. Measurements were carried out on a 50 kg aluminum bath at 720∞C. Gassing of the metal was achieved by bubbling an Ar-5%H2 mixture for a long period. Argon was then distributed by one or several static nozzles. After a residence time of about 1 second the bubbles containing Ar and H2 were collected and a part of the resulting hot gas stream was expanded into a vacuum chamber where it was analyzed by a quadripole mass spectrometer. Hydrogen contents in this gas ranged from 0.1 to 2% with a measurement accuracy of about 0.1%. An on-line calibration by known Ar-H2 mixtures (1 to 10%) was used during the degassing period. Instantaneous variation of degassing parameters such as Ar flow rate, bubble size or stirring intensity were used to evidence the main features of the degassing macro-kinetics.

2:25 PM

Critical Review of Published Values of Hydrogen Diffusion in Aluminum and its Alloys: *Prince N. Anyalebechi*[1]; [1]Grand Valley State University, Padnos Sch. of Eng., L. V. Eberhard Ctr., Ste. 718, 301 W. Fulton, Grand Rapids, MI 49504-6495 USA

Published values of hydrogen diffusion in pure aluminum and its alloys have been critically assessed against three criteria, viz: (i) reliability of experimental techniques and methodologies, (ii) self-consistency of results reported by the same investigators, and (iii) agreement of reported results with theoretically predicted and well established empirical relationships. Empirical equations for calculating hydrogen diffusion in aluminum and its alloys derived from the most reliable published values are given. Reasons for the significant disparities between reported hydrogen diffusion values and for the paucity of reliable experimental data on hydrogen diffusion in commercial aluminum alloys (especially in the liquid and semi-solid states) are discussed.

2:50 PM

Investigation of Inclusion Re-Entrainment During Filtration: *Duygu Kocaefe*[1]; Audrey Murray-Chiasson[1]; Yasar S. Kocaefe[1]; Peter Waite[2]; [1]University of Quebec, Dept. of Appl. Scis., 555 Blvd. de líUniversitÈ, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International Ltd., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada

The fabrication of high quality aluminum products, requires filtration of the liquid alloy prior to casting. Inherent to deep bed filtration is not only inclusion separation from the metal (desired), but also a re-entrainment process whereby inclusions already deposited in the filter pores re-enter the liquid metal. Abrupt changes to the metal flow also cause inclusion re-entrainment negatively affecting metal quality. A better understanding of the fundamental mechanisms governing inclusion re-entrainment could be applied to improve industrial filtration processes. To investigate inclusion re-entrainment during deep bed filtration, an experimental water modeling study was carried out. It is a common practice to use water instead of aluminum due to similarities of key physical properties of the two liquids. PVC particles were used as inclusions. During experimentation, inlet and outlet inclusion concentrations are measured using LiMCA probes adapted for water. A kinetic expression defining inclusion deposition and re-entrainment is proposed taking into account the effects of inlet inclusion concentration, inlet liquid velocity and filter media particle size.

3:15 PM

Aerospace Alloy Refining Efficiency Data for LARS (LARS: Liquid Aluminum Refining System): *Ravi Tilak*[1]; Jose Curiel[2]; [1]Almex USA, Inc., Cast House Sys., Long Beach, CA 90831 USA; [2]Vista Metals Company, QC, Fontana, CA 92335 USA

Since LARS metal treatment systems have been put into operation on aerospace alloys in 1997, several hundred million pounds of 2XXX and 7XXX alloys have been processed through LARS in conjunction with different downstream filtration media. This paper analyzes the quality of the finished product (i.e., plate, forgings and extrusions) as evaluated with various instruments and co relates it with the operating parameters of LARS. It is demonstrated that metal cast with in-line LARS and 50 PPI ceramic foam filtration is capable of consistently passing the Mil-2154-AA sonic inspection at the finished product stage. It is also demonstrated that by using LARS system, a plant can successfully eliminate the requirement of holding furnace in the typical cast-house set-up. (LARS is an acronym for Liquid Aluminum Refining System and is designed around three novel concepts; viz, in-situ preheating of inert process gas, attrition mixing of gas in liquid metal and prevention of bubble coalescence. LARS removes physical, chemical and metallurgical impurities from molten aluminum.

3:40 PM Break

3:50 PM
Recent Experiences with the Use of SiO2 for Removing of Magnesium from Molten Aluminum Alloys: *Alfredo Flores Valdes*[1]; Jose Escobedo Bocardo[1]; Jose Hernandez Gamez[1]; Sergio Escobedo Bocardo[2]; [1]CINVESTAV, Unidad Saltillo, PO Box 663, Saltillo, Coahuila 25000 Mexico; [2]Instituto Tecnologico de Saltillo, Blvd. V. Carranza y Ave. Universidad, Saltillo, Coahuila 25000 Mexico

The use of SiO2 for removing of magnesium from molten aluminum alloys has been studied in laboratory using submerged powders injection. The effects on the final magnesium and inclusions content of powder size, temperature of the molten alloy, and injection time were determined. The results obtained indicated the best experimental conditions to scaling up the process at cast shop level, for demagging of alloys containing of up to 3 wt. % Mg. In this paper it is reported both the results of the experiments conducted in laboratory, and the results of trials conducted in a cast shop facility. For the industrial trials, conducted in a reverberatory furnace, it was found that the use of fine grained SiO2 particles, and the powders injection at the point of higher turbulence, allowed to attain removal efficiencies above 60%. The presence of inclusions was not detected in the ingots obtained.

4:15 PM
Computer Simulation of the Removal of Solid Particles from Molten Aluminum in the Rotating Impeller Degasser: *Virendra Sitaram Warke*[1]; Makhlouf M. Makhlouf[1]; [1]Worcester Polytechnic Institute, Adv. Casting Rsrch. Ctr., Metal Processing Institute(MPI), 100 Institute Rd., Worcester, MA 01609 USA

One of the main tasks in aluminum refining is the removal of dissolved hydrogen and solid particles from the molten metal. This task is typically accomplished by means of a rotating impeller degasser. In this process, an inert gas, or a mixture of an inert and a reactive gas, is bubbled into the molten metal through a spinning nozzle. While the gas bubbles rise to the surface, they come in contact with suspended solid particles and dissolved hydrogen and carry them to the top slag. Currently, optimization of the rotating impeller degassing process relies to a large extent on operator experience; however, the demand for ever-increasing metal quality and cost effectiveness necessitates new approaches. Better understanding of the process can be achieved through computer modeling and simulations can be used to determine causes and corrective actions for specific operation problems. In this article, the rotary degassing melt treatment process is modeled using two interdependent modules. The first module simulates the flow field inside the furnace, and is based on two-phase fluid flow with free boundaries. The output from this module is the molten metal flow pattern, the gas bubble distribution, and the turbulence energy dissipation rate within the furnace. These parameters are used as input to the second module, which simulates the solid particles dynamics, including agglomeration, settling and flotation, as well as particle attachment to the gas bubbles and their subsequent rise to the sludge layer. The computer model is used to illustrate the effect of the various rotary degassing process parameters on process efficiency.

4:40 PM
Physical Modeling of the Aluminum Degassing Process: Experimental and Mathematical Approaches: *Emmanuel Waz*[1]; JÈrÙme CarrÈ[2]; Pierre Le Brun[1]; Alain Jardy[2]; Catherine Xuereb[3]; [1]PECHINEY CRV, 725, rue Aristide BergËs, BP 27, Voreppe, Cedex 38341 France; [2]LSG2M, Parc de Saurupt, Ecole des Mines de Nancy, Nancy, Cedex 54042 France; [3]LGC, UMR CNRS 5503 INP-ENSIACET, 5, rue Paulin Talabot, BP 1301, Toulouse, Cedex 1 31106 France

The paper proposes an experimental and numerical approach to analyze the removal of hydrogen from aluminum using rotor-based gas dispersion treatments. Liquid flows have been investigated experimentally in a water model (by Laser Doppler Velocimetry) in order to model molten aluminum flows in a degassing unit. A batch aluminum lab-scale treatment unit has been used to obtain data related to the kinetics of the degassing process. X-ray measurements have been used to assess relevant bubble characteristics (size and shape). These experiments have enabled to develop a numerical model of the aluminum degassing process, based on aluminum/argon two-phase flow. The model that accounts for the actual size and shape of the argon bubbles and the interfacial mass transfer phenomena allows the prediction of hydrogen removal kinetics in batch reactor, or hydrogen removal efficiency in in-line reactor, according to the experimental conditions.

5:05 PM
Measurements of Bubble Dispersion and Other Bubble Parameters in a Gas Fluxing Unit at Alcoa Using a Capacitance Probe: *James W. Evans*[1]; N. Mittal[1]; A. Fjeld[1]; D. Corleen Chesonis[2]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]Alcoa Inc., Casting Tech. Div., Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

A capacitance probe has been developed at Berkeley that is intended to measure the frequency with which bubbles pass a point in liquid aluminum. The probe is comprised of an inner conducting wire (in some cases two wires) that is separated from the liquid aluminum by an alumina sheath. It works by detecting the change in capacitance, between the wire and the aluminum, which occurs when the aluminum moves back from the sheath as a bubble passes. Because only alumina is in contact with the melt, the probe is robust and can survive several hours in the metal. The software necessary for processing the signals from the probe is described. The probe has been used to measure bubbles in a fluxing unit at the Alcoa Technical Center and the difficulties in doing so are described. Results are presented and their relevance to modeling and optimization of fluxing units discussed. Research cofunded by DOE Energy Efficiency and Renewable Energy.

Computational Methods in Materials Education: Market Perspectives and Teaching Materials

Sponsored by: TMS-Education Committee
Program Organizers: Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, State College, PA 16082-5006 USA; Mark D. Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA

Tuesday PM Room: 13
March 4, 2003 Location: San Diego Convention Center

Session Chair: Duane D. Johnson, University of Illinois Urbana-Champaign, Dept. of Matls. Sci & Eng. & Physics & the Frederick Seitz Matls. Rsrch. Lab., Urbana, IL 61801 USA

2:00 PM Invited
A Perspective of Aerospace Structural Alloys and Computational Methods: *Herbert A. Chin*[1]; John A. Miller[1]; [1]Pratt & Whitney, Matls. & Processes Eng., MS 114-42, 400 Main St., E. Hartford, CT 06108 USA

A perspective (past, present and future) of Structural Alloy in advanced gas turbine engines and opportunities for Computational Methods such as Thermo-Calc and DICTRA will be presented. Selected components from front to back of the engine will be discussed; goal requirements, challenges, and opportunities for Computational Methods to cost effectively bring those materials technologies to component insertion.

2:30 PM Invited
Educational Needs for a New Materials Industry Based on Design: *Charles J. Kuehmann*[1]; [1]QuesTek Innovations, LLC, 1820 Ridge Ave., Evanston, IL 60201 USA

A new revolution in computational materials design is changing what industry needs from new materials science graduates. This paradigm shift to design is being driven by new abilities to create the next generation of materials by computational design as well as the ever increasing cost of traditional empirical discovery. Currently, computational materials design expertise is limited to a small set of individuals, almost all with advanced degrees, many of whom have pioneered these enabling computational methods within their own graduate research. For the materials design revolution to take hold and grow, providing our profession a future, industry will need an available supply of young graduates with the computational and synthetic skills necessary to be effective contributors in the new companies and established organizations that embrace this coming revolution. Ad hoc attempts to provide computational content in curricula are not nearly as effective as approaches which integrate the theme into all aspects of teaching including basic thermodynamic and kinetic theory as well as applied course work specific to classes of materials, their processing, and ultimately their behavior. Ideally, these experiences should be reinforced by their integration via systems design within a capstone design course or project.

3:00 PM Invited
The Enhancement of Materials-Oriented Textbooks by the Addition of Computer Software: James W. Evans[2]; David R. Gaskell[3]; *Arthur E. Morris*[1]; [1]Thermart Software, 12102 Calle de Maria, San

Diego, CA 92128-2720 USA; [2]University of California, Matls. Sci. & Eng., 585 Evans Hall #1760, Berkeley, CA 94720-1760 USA; [3]Purdue University, Sch. of Matls. Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907-1289 USA

Computational techniques are playing an increasing role in engineering education. One indicator is the number of engineering textbooks that contain CD-ROM disks with associated software. The content of several CD-ROM discs from materials textbooks is reviewed, and compared to CD content from chemical and mechanical engineering texts. The use of web pages to enhance the educational value of texts is also reviewed. Details are given of the CD disk material from new revisions of the following texts: ìProduction and Processing of Inorganic Materialsî, Second Edition, by James W. Evans and Lutgard C. De Jonghe (2002), and ìIntroduction to the Thermodynamics of Materialsî, Fourth Edition, by David R. Gaskell (2002). The CD for each text has two types of content. First, a large set of thermodynamic data as an Excel database, which extends the rather limited data often found in text appendices. Second, many examples (keyed to problems and exercises from the text) are solved using Excelís charting, data analysis, goal-seek and solver tools. Typical examples from each text are described.

3:30 PM Break

4:00 PM Invited
Education in Computational Materials Science: Perspective from a National Laboratory: *Andrew A. Quong*[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., L-231, PO Box 808, Livermore, CA 94550 USA

At Lawrence Livermore National Laboratory, we have been involved in a number of activities that assist in the education of the Materials Science community. In particular, I will discuss two programs that I have been involved with over the past two years, the Lawrence Livermore National Laboratory Computational Materials Science and Chemistry Summer Institute for graduate students and a tutorial at the Materials Research Society meetings in Computational Materials Science. The goal of the Institute is to provide an opportunity for graduate students to explore cutting-edge methods in computational materials sciences, computational chemistry, and other related areas of computational science during their first few years of graduate study. Each student spends eight weeks at LLNL as the guest of an LLNL host scientist working on a computational project in the hostís area of expertise. The purpose of the tutorial at the MRS was to introduce the many facets of multi-length scale modeling. Because of the growing interest in the field of multiple length scale modeling, this tutorial provided the attendees with a balanced description of the main facets of this field as applied in materials science. The tutorial consisted of lectures and a hands-on computer session where the attendees were able to run actual applications.

4:30 PM Invited
Recommended Skill Sets for Future Engineers in the Aluminum Industry: *Hasso Weiland*[1]; [1]Alcoa Technical Center, 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

Development times for new products in the materials industry have shortened significantly. While earlier, the development of a new alloy took several decades, now two to three years are the standard. To achieve such short development times, it is required to have well integrated materials simulation tools available. Such tools are to be based on accurate understanding of the underlying processes. These changing needs in the materials industry requires engineers and scientists to have sound knowledge in several disciplines. This presentation attempts to outline the skill sets necessary for successful future candidates.

5:00 PM Panel Discussion

Computational Phase Transformations: Effect of Interface on Phase Transformation

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Tuesday PM Room: 11B
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Long-Qing Chen, Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA; J. E. Morral, University of Connecticut, Dept. of Metall. & Matls. Eng., Storrs, CT 06269-3136 USA

2:00 PM Invited
Impurity-Induced Grain Boundary Phase Transformations in Metals and Oxides: *Gerd Duscher*[1]; [1]North Carolina State University, Dept. of Matls. Sci. & Eng., Solid State Div., 2156 Burlington Nuclear Lab., Raleigh, NC 27695-7916 USA

It has been suspected for a long time that impurities can induce grain boundary phase transformations. It is difficult, however, to prove this transformation, since to do so we must study unexpected atomic arrangements at the core of grain boundaries. This can only be accomplished by using a combination of experimental and theoretical studies at atomic resolution. In this study, we used Z-contrast imaging, electron energy-loss spectroscopy (EELS) and ab initio materials simulations to study the atomic structure of grain boundaries. Z-contrast imaging provides us with directly interpretable images of the grain boundary structure, and EELS characterizes the chemical composition locally at the core of the grain boundary. The resolution of both of these techniques is greatly enhanced with an aberration corrector, which allows subangstrom analysis. The first results obtained with an aberration-corrected STEM are shown in this study. These experimental results allowed us to produce a starting model for ab initio calculations. We used ultrasoft pseudopotential and a plane wave basis set method in local density approximation. The total energies for the models with and without impurities allowed us to find the lowest energy configuration. While the grain boundary structure changed dramatically with Ca impurities in MgO, we saw only a slight modification in sigma 5 Al grain boundaries with Cu doping. However, there is no grain boundary phase transition of Cu grain boundaries with heavy dopant atoms (Bi, Ag).

2:30 PM Invited
Beyond the Local-Equilibrium Paradigm in Phase Transformation Modeling: *John A.L. ≈gren*[1]; [1]Royal Institute of Technology, Dept. of Matls. Sci. & Eng., Stockholm 100 44 Sweden

Finite interfacial mobility and solute diffusion across interfaces may cause a deviation from local equilibrium during phase transformations and cause, for example, a transition to massive transformation at some critical supersaturation. Exactly where this transition occurs depends on the kinetics of the interfacial reactions. Another question concerns the transition to paraequilibrium during the transformation of austenite in steels. Even though these phenomena have been discussed for almost half a century the progress has been very slow until the last few years. Many papers have been published recently. There are several reasons for this, e.g. the new development of bainitic steels has awakened the old paraequilibrium concept, the recent phase-field theory includes (unknown?) details of phase interfaces and new experimental techniques allows to characterize the chemical constitution of interfaces with a higher precision. In this presentation some recent advances in the modeling of the above phenomena will be discussed.

3:00 PM
Effect of Grain Boundaries on Spinodal Decomposition: *Hariharaputran Ramanarayan*[1]; Thennathur A. Abinandanan[1]; [1]Indian Institute of Science, Dept. of Metall., Bangalore, Karnataka 560 012 India

We have used a combination of a phase field model (a continuum version of the Potts model) and the Cahn-Hilliard model to study the effect of grain boundaries (g.b.) present in a polycrystalline sample on spinodal decomposion. In particular, when $\gamma_\alpha < \gamma_\beta$ (i.e., when the grain boundary (GB) energy of the A-rich α phase is less than that of the B-rich β phase), the early stage is characterized by a preferential migration of A atoms to the GB. This process leads to a composition wave that travels normal to the GB into the grain interior, and to the formation of alternating B-rich and A-rich bands near the GB. Simultaneously, grain interiors, unaffected by the GB, undergo normal SD. Thus, the late stage microstructures in large grains reveal alternating bands of α and β phases near GBís coexisting with normal SD microstructure in the grain interiors. On the other hand, small grains (whose sizes are 2 to four times the characteristic spinodal wavelength), exhibit only concentric, alternating rings of the α and β phases. In this paper, we will present these results with an emphasis on the factors that affect the extent of the banded regions near the GB relative to the normal SD region in the interiors.

3:20 PM Break

3:40 PM Invited
Some Recent Comparisons Between Experiment and Modeling of Interfaces in Phase Transformations: *James M. Howe*[1]; William C. Johnson[1]; [1]University of Virginia, Dept. of Matls. Sci. & Eng., 116 Engineerís Way, Charlottesville, VA 22904-4745 USA

This presentation discusses two recent examples where experiments and modeling were combined to better understand the role of interfaces in phase transformations in solids. In one example, a two-dimensional Cahn-Hilliard equation was used to model the experimentally observed spinodal decomposition in the presence of a solute-depleted zone adjacent to Ag-rich plates in an Al-Ag alloy. In the second example, variational calculus was used to model the experimentally observed dependence of the dihedral angles at a triple junction on the fraction of Ag and Cu-rich phases in spherical two-phase, Ag-Cu alloy particles. In both cases, the modeling was found to reproduce the experimentally observed behavior, thereby leading to a better understanding of the fundamental phenomena causing the behavior as well as enabling new behaviors to be explored. This research was supported by NSF under Grants DMR-9902110 and 9908855.

4:10 PM Invited
Phase Field Modeling of Polycrystals: *James A. Warren*[1]; [1]NIST, CTCMS, 100 Bureau Dr., Stop 8554, Gaithersburg, MD 20899 USA

A two dimensional phase field model of grain boundary statics and dynamics has been developed. The model, which introduces an orientation parameter to the well known phase field model of solidification, reproduces many of the phenomena observed during grain boundary evolution, as well as the phenomenon of grain rotation. The theory also predicts the onset of grain boundary wetting, above a critical grain misorientation where the grain boundary energy exceeds twice the liquid-solid surface energy. In this talk we examine some of the new areas of application of this model, including alloys, fractal structures, spherulitic growth and some of the mathematical idiosyncrasies of the model (in particular, how to deal with the non-analytic free energy density). Finally, we will discuss future avenues for research.

4:40 PM
Integrity of Grain Boundaries in an Al-Li-Cu-Mg-Zr Alloy: Experimental Observations and Modelling Opportunities: *Graham B. Winkelman*[1]; Stan P. Lynch[2]; Barry C. Muddle[1]; [1]Monash University, Sch. of Physics & Matls. Eng., PO Box 69M, Victoria 3800 Australia; [2]Defence Science and Technology Organisation, PO Box 4331, Melbourne, Victoria 3001 Australia

A limiting factor in the up-take of Li-containing Al alloys for commercial applications remains the susceptibility of the alloys to low energy intergranular fracture at low temperatures. In the present work, the susceptibility to embrittlement of an Al-Li-Cu-Mg-Zr alloy (commercial alloy 8090) has been associated with the segregation of Li to grain boundaries during ageing. The fracture behaviour of the material thus sensitised is observed to be strongly temperature-dependent, with the sensitised alloy undergoing a sharply-defined transition from ductile to brittle intergranular fracture with decreasing temperature. Preliminary evidence suggests that this transition may be associated with a two-dimensional structure change within the Li-modified grain boundaries as a function of temperature. The experimental approaches to this problem will be summarised, along with consideration of the needs for computational modelling to assist in elucidating details of both the structural transitions within the grain boundaries and the subsequent fracture behaviour.

5:00 PM
Thermal Transient FEA of Lead Anode Casting Mould: *Zane Jia*[1]; Reg Davis[1]; Jenny Cassidy[1]; Gary Morin[2]; [1]H. G. Engineering, 400 Carlingview Dr., Toronto, Ontario M9W 5X9 Canada; [2]Falconbridge, Ltd., Kidd Creek Div., PP Box 2002, Timmins, Ontario P4N 7K1 Canada

A thermal transient finite element analysis (FEA) study was performed to help solve problems associated with the poor lead anode quality of the lead anode casting mould. The study was intended to evaluate the transient heat transfer between the lead anode and the casting mould. A 3D FEA model of lead anode casting mould, including multi-phase lead anode was created. Non-linear transient thermal analyses were performed to evaluate the lead anode solidification process. FEA results of the current casting process match the field observation and measurements closely. Therefore the FEA model now provides the client a unique tool in quantifying the current casting process, and consequently identifying the problems. A preliminary modification design was performed to improve the mould cooling to produce better quality lead anodes. The proposed modifications were further evaluated through the modified FEA models. Results of the FEA clearly demonstrate the benefits of the proposed modifications.

Defects and Deformation of Crystalline Solids - in Honor of Dr. Man H. Yoo - IV

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
Program Organizers: Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Department, Houghton, MI 49931-1200 USA; Sean R. Agnew, University of Virginia, Materials Science and Engineering Department, Charlottesville, VA 22904-4745 USA; K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA

Tuesday PM Room: 17A
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Armen Khachaturyan, Rutgers University, Dept. of Matls. Sci. & Eng., Piscataway, NJ 08855 USA; Tresa Pollock, University of Michigan, Matls. Sci. & Eng. Dept., Ann Arbor, MI 48109-2136 USA

2:00 PM Invited
Relationship Between Deformation and Recrystallization Textures: *Dong Nyung Lee*[1]; [1]Seoul National University, Sch. of Matls. Sci. & Eng., Seoul 151-744 Korea

The recrystallization process basically occurs to reduce the energy stored during fabrication. The stored energy may include energies due to vacancies, dislocations, grain boundaries, surface, etc. The energy is not directional, but the texture is directional. No matter how high the energy of defects may be, they cannot directly be related to the recrystallization texture, unless they give rise to some anisotropic characteristics. The most important driving force for recrystallization is known to be the stored energy due to dislocations. Dislocations cannot be related to the recrystallization texture, unless they give rise to some anisotropic characteristics. In this article, we discuss how dislocations generated during deformation can give rise to anisotropic characteristics for recrystallization textures.

2:25 PM Invited
Deformation Mechanisms of Slip Transmission in Alpha/Beta Titanium Alloys at Lower Temperatures: *Michael J. Mills*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 478 Watts Hall, Columbus, OH 43210 USA

Two-phase titanium alloys such as Ti-6242 and Ti-6Al-4V are used extensively in aeroengine and biomedical applications. In spite of their high strength, Ti alloys must be employed conservatively because of their tendency to creep significantly at room temperature, even at stresses well below the macroscopic yield strength. The transmission of slip across α-β interfaces, which conform to a near-Burgers orientation relationship, is an important precess during creep. Testing of single colony crystals of both Ti-5Al-2.5Sn and Ti-6242 has shown that they are highly anisotropic with respect to the operative slip system in the α-phase. Extensive TEM investigation has revealed the mechanisms of slip transmission for various orientations, which provides insight into the observed anisotropy in yield and creep behavior. The implications of these results with respect to modeling of deformation and fatigue in Ti alloys will also be discussed. Funding for this work has been provided by the US Air Force Office of Scientific Research and the Federal Aviation Administration.

2:50 PM
Displacement Mapping of Intermetallics at the Length Scale of Individual Grains: *Gurjeev Chadha*[2]; Aomin Wu[2]; Qidi Chen[1]; Marc De Graef[1]; Tresa M. Pollock[2]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; [2]University of Michigan, Dept. of Matls. Sci. & Eng., H. H. Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

In many intermetallics, local microstructural variations affect plastic deformation. Such variations include texture (differently oriented grains react differently to a given load), segregation, multiple phases, etc. We have developed a two-dimensional displacement mapping technique to determine in-plane surface displacements during mechanical testing (tension and compression). The method requires the deposition of a square grid of markers on the sample surface. Images are taken at different load levels, and a semi-automated pattern recognition system determines the displacements of the markers with respect to reference marker positions. Strain levels of less than 0.1% can thus be measured, with multiple measurements for each individual grain (grain size 10-100 micron). A combination of displacement mapping and orientation imaging microscopy can provide sufficient information to analyse strain levels in terms of the available slip systems for each individual grain. We will present strain maps for a variety of materials, including RuAl, IN100, and Mg.

3:10 PM
Microcracking in Brittle Materials Due to Thermal Expansion Anisotropy: *Phani Kumar Nukala*[1]; Srdan Simunovic[1]; Balasubramaniam Radhakrishnan[1]; Gorti B. Sarma[1]; [1]Oak Ridge National Laboratory, Compu. Sci. & Math. Div., Oak Ridge, TN 37831-6359 USA

Thermal cooling of a variety of polycrystalline materials from high processing temperatures results in microcracking due to thermal expansion anisotropy of single phases and/or thermal expansion mismatch between multiple phases. This study investigates the damage evolution in polycrystalline materials due to thermally induced microcracking using three-dimensional discrete lattice spring networks. In particular, the effect of grain size distribution on inter- and intragranular microcrack nucleation, growth and coalescence is investigated. Realistic 3D microstructure based discrete lattice simulations include the interaction of grains in parallel planes and the additional crack propagation paths for growth and coalescence of cracks. Numerical simulations are also used to estimate the critical grain size below which nucleation of microcracks does not occur. This critical grain size plays an important role in designing material applications with improved fracture properties. This work also highlights the significant differences between 2D and 3D numerical simulation of thermally induced microcracking in polycrystalline materials. Work performed under the auspices of the US Department of Energy by Oak Ridge National Laboratory under Contract DE-AC05-00OR22725 with UT-Batelle LLC.

3:30 PM Break

3:50 PM Invited
Mechanical Properties of Single Crystals of Mo_5SiB_2 and Related Compounds: Kazuhiro Ito[1]; *Masaharu Yamaguchi*[1]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan

Mo_5SiB_2 is a new refractory metal silicide with great potential for ultra-high temperature structural applications. We have grown high purity single crystals of Mo_5SiB_2 and related compounds by an optical floating zone method. Single-crystal electric resistivity, thermal expansion coefficient (CTE) and elastic constants of these compounds were measured in a wide temperature range. Compression and creep tests at high temperatures were also performed. The results of these experiments are presented all together.

4:15 PM
Phase Field Microelasticity Theory and Modeling of Elastic Inhomogeneities, Voids and Cracks: *Yu U. Wang*[1]; Yongmei M. Jin[1]; Armen G. Khachaturyan[1]; [1]Rutgers University, Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA

Technologically advanced solid materials are frequently inhomogeneous, both elastically and structurally. The effect of elastic inhomogeneities on material properties is less understood than that of structural inhomogeneities due to its more complicated theoretical treatment. The Phase Field Microelasticity approach is employed to formulate a general theory of different modulus solid, which provides an effective tool to model arbitrary elastic and structural inhomogeneities. Voids and cracks are the particular cases of this theory. They are treated as inhomogeneities of zero elastic modulus. This approach reduces the problem of elastic equilibrium and evolution of an inhomogeneous system to a solution of the non-linear integro-differential Ginzburg-Landau equation. In the Phase Field formalism, the long-range strain-induced interaction of individual inhomogeneities is directly taken into account. The proposed model does not impose a priori constraint on possible inhomogeneity configurations or their evolution paths. Examples of effect of elastic inhomogeneities on material microstructures and properties are presented.

4:35 PM Invited
Microstructural Stability in Non-Equilibrium Processed Alloys: Yong C. Kim[1]; Sang B. Lee[1]; Seung J. Hwang[2]; Kwang S. Shin[3]; *Nack J. Kim*[1]; [1]POSTECH, Ctr. for Adv. Aeros. Matls., San 31, Hyojadong, Pohang 790-784 Korea; [2]Daejin University, Dept. of Matls. Sci. & Eng., Kyung-gi 487-711 S. Korea; [3]Seoul National University, Seoul 151-742 S. Korea

In recent years, there has been a renewed interest in non-equilibrium processes such as mechanical milling/alloying and rapid solidification, since these can produce powders with a nanoscale grain size and a homogeneous distribution of nanoscale dispersoids. However, it is known that the nanoscale microstructures of non-equilibrium processed powders are lost during high temperature consolidation, often resulting in undesirable microstructures. Although most of the non-equilibrium processed alloys still show much finer grain size after consolidation than conventionally processed alloys, it is needed to restrict grain growth as much as possible to maximize the potential of non-equilibrium processed alloys. The present study is aimed at investigating the evolution of microstructure in non-equilibrium processed alloys during subsequent consolidation. Various types of alloys have been investigated in the present study, including Al-, Mg-, Ni-, and Ti-based alloys. Emphasis has been placed on the role of dispersoid particles in microstructural stability of these alloys.

5:00 PM
The Role of Polytwin Interfaces for the Deformation Behavior of L1o-Ordered Iron-Palladium Intermetallics: *J^rg M.K. Wiezorek*[1]; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

The frequent polytwin (PT) interfaces present in L1o-phases after appropriate heat treatments strongly influence the properties and behavior of this interesting class of intermetallics. The interactions of dislocation glide and mechanical twinning with the PT-interfaces has been studied through crystallographic and geometric analyses for {101}-conjugated PT-interfaces in FePd based L1o-phases. PT-FePd alloys exhibit a yield stress anomaly and considerable work-hardening under tensile conditions. Defect-interface interactions in room temperature deformed PT-FePd have been studied experimentally by transmission electron microscopy (TEM). A model for the defect-interface interactions in PT-FePd, based on the most suitable boundary reactions, identified through theoretical analyses, has been proposed and is consistent with the experimental TEM observations. This model enables a micro-mechanism based rationalization of the plastic flow behavior typical of polycrystalline PT-FePd. Defect-interface interactions in {101}-conjugated PT-L1o-phases and their role for the deformation behavior of are discussed. This work has been supported by the NSF, grant DMR 0094213, with Dr. K.L. Murty as program manager.

Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong: Dynamic Fracture

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Marc Andre Meyers, University of California-San Diego, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; George T. Gray III, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA; Kenneth S. Vecchio, UC San Diego, Dept of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA

Tuesday PM Room: 16B
March 4, 2003 Location: San Diego Convention Center

Session Chair: Kenneth S. Vecchio, University of California-San Diego, Mechl. & Aeros. Eng., La Jolla, CA 92093-0411 USA

2:00 PM
Role of Disperson Forces in Fracture: *John J. Gilman*[1]; [1]University of California-Los Angeles, Matls. Sci. & Eng., 6532 Boelter Hall, Los Angeles, CA 90095-1595 USA

Dispersion forces between atoms are small and therefore usually ignored. However, they are always positive, and when integrated over many atoms in a dense solid, they become significant over short distances. In particular, near the tips of cracks. As the local chemical bonding forces become weak due to tensile strains, the dispersion forces become important. The former forces increase proportionally with strain for small strains (Hookeís Law), whereas the latter forces decrease in proportion to large strains cubed (Lifshitz Law). A single functional dependence describes the combined forces from zero to large strains in terms of the elastic stiffness and the polarizability (no disposable parameters). High polarizability extends the size of the region of plastic deformation; and the roughness caused by plastic deformation increases the dispersion forces. Thus the nature of ductility can be understood.

2:30 PM
The Structure and Distribution of Voids in Spall Fracture from 3D X-Ray Tomography and 2D Microscopy: *James Belak*[1]; James Cazamias[1]; David Haupt[1]; John Kinney[1]; Mukul Kumar[1]; Roger Minich[1]; Robert Rudd[1]; Chris Schuh[1]; Adam Schwartz[1]; Eira Seppala[1]; [1]Lawrence Livermore National Laboratory, PO Box 808, Livermore, CA 94550 USA

Gas gun recovery experiments were used to study incipient spall fracture in polycrystalline aluminum and single crystal aluminum and copper. In addition to in-situ VISAR waveprofiles, the recovered samples were first analyzed using 3D X-ray tomography and then sectioned for 2D microscopy. Void nucleation and growth in the polycrystal samples occurs at grain boundaries with an exponential distribution of void sizes. The void size and spatial distribution are determined directly from the X-ray tomography. The single crystal samples show a bimodal distribution of small voids with large (50-100 micron) well separated voids. The plastically damaged region surrounding the large voids is quantified using optical and electron backscattering microscopy. Microhardness measurements indicate this region to be harder than the surrounding metal. This work was performed under the auspices of the US Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

2:45 PM
Laser-Based Experiments Investigating the Dynamics of Ductile Material Failure: *Kimberly S. Budil*[1]; Daniel H. Kalantar[1]; Richard C. Becker[1]; Geoffrey H. Campbell[1]; F. Xabier Garaizar[1]; James F. Belak[1]; Daniel J. Nikkel[1]; [1]Lawrence Livermore National Laboratory, DNT/B Div., PO Box 808, L-97, 7000 East Ave., L-97, Livermore, CA 94550 USA

We will describe an effort to develop a laser-based testbed to study dynamic material failure. Laser drivers produce extremely high strain rates (10^6-10^8 /sec) and can be used to produce shocked or shockless, quasi-isentropic compressions. A main focus is the development of in situ diagnostics including probing the pressure history within the sample (via free surface velocity measurements), observing the formation and evolution of voids (via x-ray radiography) and measuring the response of the lattice to compression (via dynamic diffraction). We are developing an experiment to measure the growth rate of an isolated void in a material that has gone into tension after shock passage and will present calculations and preliminary data for the proposed experiment. This work was performed under the auspices of the US Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

3:00 PM
Spall Behavior of Laser-Shocked Sn: Comparison of Modeling to Experimental Data: *Jeffrey D. Colvin*[1]; E. R. Ault[1]; T. L. Clauson[1]; W. E. King[1]; M. Kumar[1]; I. H. Zimmerman[1]; [1]University of California, Lawrence Livermore Natl. Lab., L-356, PO Box 808, Livermore, CA 94550 USA

Few-joule table-top lasers can generate pressures up to the 100-kbar range in materials by propagating a low-intensity beam through a transparent dielectric, which confines the ablation pressure, onto an ablation layer in contact with the material of interest. We have applied this technique to the study of spall in Sn and other metals. Application of this technique to material deformation studies has been hampered until recently by lack of a physically based model of the laser absorption processes. We describe a new computational model for these processes incorporated into a 2D radiation-hydrodynamics code. We discuss this model through a comparison of simulated and measured free-surface velocity histories for Sn, and review the implications for spall models. This work was performed under the auspices of the US DOE by the Univ. of Calif., LLNL under Contract No. W-7405-Eng-48.

3:15 PM
Spall Studies in Single and Polycrystalline Copper: *James U. Cazamias*[1]; Adam J. Schwartz[2]; Roger L. Minich[3]; Mukul Kumar[4]; [1]Lawrence Livermore National Laboratory, L414, PO 808, Livermore, CA 94551 USA; [2]Lawrence Livermore National Laboratory, L-355, PO 808, Livermore, CA 94551 USA; [3]Lawrence Livermore National Laboratory, L-097, PO 808, Livermore, CA 94551 USA; [4]Lawrence Livermore National Laboratory, L-356, PO 808, Livermore, CA 94551 USA

We are executing a systematic study to quantify the effects of specific microstructural features on the spall behavior of 99.999% copper. Spall in ductile metals is a 1D strain dynamic tensile failure caused by the nucleation, growth, and coalescence of voids. Previous work reported an increase in spall strength of polycrystalline copper with increasing grain size. Here, we will present results of single crystals with [100], [110] and [111] orientations, and internally oxidized [100] single crystals (0.15 and 0.5 wt.% Si) that are shocked with Cu flyers at velocities ranging from 300 to 2000 m/s using a 35-mm single/two-stage light gas gun. The presence of hard silica particles in the microstructure provide potent nucleation sites for void nucleation and reduce the spall strength dramatically in comparison to the pristine single crystals. Laser interferometric measurements of the free surface velocity are used to characterize the spall pullback signal and details of the ringing. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

3:30 PM
Stress-Strain Description of Elastic, Plastic, Cracking Behavior of Microstructurally Biased Two-Phase TiB2+Al2O3 Ceramic: *Louis Ferranti*[1]; Naresh N. Thadhani[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30332 USA

Vickers microindentation hardness testing was used to investigate the elastic, plastic, and cracking behaviors of microstructurally biased two-phase TiB2+Al2O3 ceramic. An indentation hardness stress-strain description considering the classical analysis of Hertz was constructed to evaluate the theoretical elastic behavior for the total indentation event. Comparison of the presumed entirely plastic hardness measurement with the elastic Hertzian hardness stress-strain description indicated a significant elastic contribution to the measurement. Individual Hertz elastic stress-strain curves were constructed for pure TiB2 and Al2O3 as well as the two-phase TiB2+Al2O3 ceramic determined by the rule of mixtures. These curves were accompanied by experimentally measured hardness values on four quantitatively unique microstructures. This analysis enables the comparison of hardness measurements with those predicted by the Hertz description and the contributing elastic effect during the measurement processes. The analysis also illustrates the need for precise nanometric scale measurements to determine this elastic effect directly from experimental material testing methods. Continuous ball indentation hardness testing from previous studies dramatically shows the importance of the elastic component for the predicted hardness values (Armstrong 1995, Hammond 1988).

3:45 PM Break

4:00 PM

Morphology of Fracture Domains in Brittle Solids: *Michael Grinfeld*[1]; T. W. Wright[1]; [1]Educational Testing Service and National Research Council, 9 Carlton Cir., Princeton, NJ 08540 USA

The appearance of intensively fractured zones (IFZ) is a rather widespread phenomenon when dealing with brittle materials in various engineering systems or with geophysical media. Since even individual defects of a crystalline medium like cracks, dislocations, interfaces, etc. are complex objects on their own, researchers limit themselves with an overall, integral description of the IFZís material. When dealing with IFZ it is often desirable to extend the information about the overall material properties with the information about the overall shape and morphology of the IFZ. In this paper, following Gibbs and Griffith minimum energy approaches, we propose one such thermodynamic approach allowing one to determine the shape of the IFZ simultaneously with the distributions of stresses and strains within the systems of interest. The general methods and results of the suggested approach are illustrated by consideration of some particular brittle systems taken from engineering and geomechanics.

4:15 PM

A Model for Fracture of Explosively Driven Metal Shells: *Vladimir M. Gold*[1]; Ernest L. Baker[1]; [1]US Army TACOM-ARDEC, Attn: AMSTA-AR-WEE-C, Bldg. 3022, Picatinny Arsenal, NJ 07806-5000 USA

A model for fracture of explosively driven metal shells presented in this work is based on integrating three-dimensional axisymmetric hydrocode analyses with analyses from a newly developed fragmentation computer code MOTT. The developed model was based on the Mottís theory of break-up of cylindrical ìring-bombsî, in which the length of the average fragment is a function of the radius and velocity of the shell at the moment of break-up, and the mechanical properties of the metal. The validation of the MOTT code fragmentation model was accomplished using the existing explosive fragmentation munition arena test data. After having established the crucial parameters of the model, a new explosive fragmentation munition was designed and optimized. Upon fabrication of the developed munition, the performance of the new charge was tested in a series of small-scale experiments including the flash radiography, the high-speed photography, and the sawdust fragment recovery. The accuracy of the MOTT code predictions is rather remarkable.

4:30 PM

Failure Behavior of Tungsten-Reinforced Amorphous Metal Matrix Composites: *L. J. Kecskes*[1]; L. S. Magness[1]; T. Jiao[2]; T. C. Hufnagel[2]; K. T. Ramesh[2]; [1]US Army Research Laboratory, Weapons & Matls. Rsrch. Direct., Aberdeen Proving Ground, MD 21005 USA; [2]The Johns Hopkins University, Baltimore, MD 21218 USA

We evaluated the ballistic behavior of tungsten-reinforced bulk-metallic glass (BMG) matrix composites. Special techniques were employed to recover the residual penetrators and penetrator erosion products. To delineate the ballistic behavior from those observed at lower strain rates, we conducted a series of compressive split-Hopkinson-pressure-bar (SHPB) tests at strain rates of about 103 s-1. The failure surfaces of monolithic and composite samples obtained in the high-strain-rate tests as well as those of the ballistic erosion products were examined with scanning electron microscopy (SEM). X-ray diffraction (XRD) analysis of the ballistic debris and SHPB sample fragments revealed little or no crystallization. These results are consistent with the primary failure mechanism of the BMG matrix being governed by a drop in viscosity due to a temperature increase associated with shear localization.

4:45 PM

The Dependence of Spall Strength on Length Scales: *Roger W. Minich*[1]; Mukul Kumar[1]; James Cazamias[1]; Adam J. Schwartz[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., Livermore, CA 94550 USA

The spall process involves nucleation, growth and coalescence of voids leading to final fracture. It is now widely accepted that the microstructure exerts a strong influence on the resistance of a material to spallation. Data in the literature indicates that the spall strength of high purity Cu single crystals is about twice that of polycrystalline Cu. This clearly indicates the influence of intercrystalline defects such as grain boundaries and triple junctions. Systematic variation in pressure suggests that the spall strength increases with increasing grain size, peaking for the single crystal case. This functional dependence of the flow stress on microstructural length scales is being developed into a model for implementation into 1-D finite element formulations. The model incorporates statistical stress fluctuations due to microstructural effects that increase the probability that a void can nucleate and grow thereby decreasing the spall strength, as for example, as a function of grain size.

5:00 PM

AFM Studies of Fracture Surfaces of Composition B Energetic Materials: *Yvonne D. Lanzerotti*[1]; Jagadish Sharma[2]; R. W. Armstrong[3]; [1]US Army TACOM-ARDEC, AMSTA-AR-WEE, Bldg. 3022, Picatinny Arsenal, NJ 07806-5000 USA; [2]Naval Surface Warfare Center, Carderock Div., W. Bethesda, MD 20817-5700 USA; [3]AFRL-MNME, 2306 Perimeter Rd., Eglin AFB, FL 34542-5910 USA

The characteristics of TNT (trinitrotoluene) crystals in Composition B have been studied using atomic force microscopy (AFM). The size of TNT crystals has been examined by analyzing the surface structure that is exhibited after mechanical failure of the Composition B. The mechanical failure occurs when the material is subjected to high acceleration (high g) in an ultracentrifuge and the shear or tensile strength is exceeded. AFM examination of the topography of the Composition B fracture surface reveals fracture across columnar grains of the TNT. The width of the columnar TNT grains ranges in size from 1 micron to 2 microns. Their height ranges in size from 50 nanometers to 300 nanometers.

5:15 PM

Effect of Triaxiality on Void Growth in Dynamic Fracture: A Molecular Dynamics Study: *Eira T. Sepp%d%o*[1]; James Belak[1]; Robert E. Rudd[1]; [1]Lawrence Livermore National Laboratory, L-415, 7000 East Ave., Livermore, CA 94550 USA

Dynamic fracture in ductile metals occurs through the nucleation and growth of voids. In this paper the effect of stress triaxiality on the evolution of the void growth has been studied in a single-crystal copper under dilational strain. Molecular dynamics simulations have been performed with a pre-existing void using high strain rates ranging from 10^7/sec to 10^10/sec and uniaxial, biaxial, and triaxial loading modes. Stress-strain curves, void shape evolution, and dislocation structures for the different loading modes are analyzed showing that the yield stress, the growth-rate and isotropicity of the void, and the dislocation activity are dependent on the triaxiality. Acknowledgment: This work was performed under the auspices of the US Dept. of Energy at the University of California/Lawrence Livermore National Laboratory under contract no. W-7405-Eng-48.

5:30 PM

Void Initiation and Growth in Spalling: *Vlado A. Lubarda*[1]; Bimal K. Kad[1]; Matthew S. Schneider[1]; Fabienne Gregori[2]; Marc A. Meyers[1]; Daniel H. Kalantar[3]; Bruce A. Remington[3]; [1]University of California-San Diego, Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92093-0411 USA; [2]University of Paris, 13 Villetaneuse, Paris 93430 France; [3]Lawrence Livermore National Laboratory, Livermore, CA 94720 USA

The various existing mechanisms for void initiation and growth are discussed in terms of their applicability to the high-strain rate regime. The relative importance of diffusional processes (vacancies) and slip processes (dislocations) are discussed in the establishment of void initiation and growth relationships. The mechanisms are compared with observations in plate impact and laser shock experiments. Research supported by the Department of Energy.

Friction Stir Welding and Processing II: Friction Stir Joining: Corrosion & Corrosion Fatigue

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee

Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Thomas J. Lienert, University of South Carolina, Mechanical Engineering Department, Columbia, SC 29208 USA; Rajiv S. Mishra, University of Missouri-Rolla, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Tuesday PM Room: 7B
March 4, 2003 Location: San Diego Convention Center

Session Chair: Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA

2:00 PM
Corrosion and Stress Corrosion Susceptibility of Friction Stir Welded in Al-Li-Cu Alloy AF/C458: *Rudolph G. Buchheit*[1]; Barbara N. Padgett[1]; Christian S. Paglia[1]; [1]Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Among Al-Li-Cu alloys, AF/C458 (Al-1.8Li-2.7Cu-0.6Mg-0.3Zn) exhibits good localized corrosion and stress corrosion cracking (SCC) resistance in aqueous chloride solutions. However, susceptibility to both is increased near of friction stir welds due to microstructural changes induced by the thermal excursion associated with welding. Intergranular corrosion has been detected in the weld nugget, thermomechanically affected zone (TMAZ), and in the heat affected zones (HAZ). Under dynamic straining in chloride solutions with loads applied transverse to the weld, SCC occurs in the HAZ near the interface with the TMAZ. In this location, deformation is concentrated due to local softening during welding. At this interface, grain boundaries become oriented perpendicular to the load direction. These boundaries are rendered electrochemically active due to heavy precipitation of T_1 (Al_2CuLi) on them. Special consideration will be give to the possibility that boundaries containing the T_B (Al_7Cu_4Li) are less susceptible to attack than those where T_1 has precipitated.

2:25 PM
Localized Corrosion and Stress Corrosion Cracking of Friction Stir Welds in 7XXX Aluminum Alloys: *Rudolph G. Buchheit*[1]; Christian S. Paglia[1]; Anthony P. Reynolds[2]; [1]Ohio State University, Fontana Corrosion Ctr., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]University of South Carolina, Dept. of Mechl. Eng., Columbia, SC 29208 USA

Localized corrosion and stress corrosion cracking (SCC) behavior of friction stir welds in 7075-T6 and 7150-T7 is presented. In the as-welded condition, localized corrosion susceptibility is increased in the heat affected zones (HAZ) of the weld. Localized corrosion occurs as intergranular corrosion and pitting at constituent particles in the alloy. TEM and local electrochemical measurements suggest that precipitation at grain boundaries leads to intergranular corrosion. SCC failures also occur in HAZs. SCC was characterized by constant extension rate tests of samples cut transverse to the weld. SCC susceptibility results in decreased yield stresses and ductility. Deformation is concentrated in soft HAZs sections occurs in heat affected zones due. Local softening and increased grain boundary precipitation appear to be the primary contributing factors. Several post weld heat treatment processes are described with particular attention paid to short term high temperature treatments that result in uniform weld hardness and good SCC resistance.

2:50 PM
Improving Corrosion Properties of 7475 Friction Stir Welds by Localized Heat Treatment: Ali A. Merati[2]; Don Raizenne[2]; *Claudio Dalle Donne*[1]; [1]German Aerospace Center, Inst. of Matls. Rsrch., Linder Hoehe, Cologne 51147 Germany; [2]National Research Council Canada, Inst. for Aeros. Rsrch., 1500 Montreal Rd., Bldg. M-13, Ottawa, Ontario K1A 0R6 Canada

Recent work in the field of 7xxx aluminum alloy friction stir welds revealed that joints of these materials have to be heat treated after welding to stabilize the weld microstructure and to increase the stress corrosion properties. Even though only specific areas of the weld and heat affected zone require post-weld heat treatment, the entire welded plate is normally aged in an oven. From a production point of view this procedure is cost and labor intensive. This paper deals with the retrogression and re-aging (RRA) heat treatment carried out locally on double and single pass welds of 10 mm thick 7475-T7351. The RRA heat treatment was specifically developed for 7075 alloys in an attempt to achieve high levels of both strength and stress corrosion cracking (SCC) resistance. The hardness and conductivity measurements of the locally heat treated welds are compared to the values of plates treated in an oven. Moreover the results of tensile and stress corrosion (ASTM G139) tests are presented.

3:15 PM
High-Cycle Corrosion Fatigue of 7050-T7451: *Michael Dunlavy*[1]; Kumar V. Jata[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, 2230 Tenth St., WPAFB, OH 45503 USA

Due to their heterogeneous microstructures and susceptibility to pitting corrosion extensive research has been performed on the effects of friction stir welding (FSW) on corrosion fatigue behavior of 2xxx and 7xxx series high strength aluminum alloys. However limited work has been done on high-cycle corrosion fatigue behavior of high strength FSW aluminum alloys, such as 7050-T7451. The objective of this work was to evaluate the effect of pitting corrosion on high cycle fatigue strengths of friction stir welded aluminum alloy 7050-T7451 after exposure to prohesion spray. Various exposure times (100 and 500 hrs) during prohesion tests resulted in corrosion pits, which were measured after fatigue testing. HCF tests were conducted near the endurance limit and fracture surface analysis determined whether failure originated at corrosion pits or inclusion sites.

3:40 PM Break

3:50 PM
Effects Pre and Post Weld Heat Treatments on the Corrosion Properties and Microstructure of FSW Zones of AA7075 and AA7050: *Jesse B. Lumsden*[1]; Cecil Rhodes[1]; Murray Mahoney[1]; [1]Rockwell Scientific, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA

The corrosion properties of high strength aluminum alloys is strongly dependent on grain boundary composition as well as the composition and morphology of the intermetallics. Thus, the corrosion behavior of the recrystallized microstructure in the nugget of a FSW aluminum alloys is usually different from that of the parent material. In addition the local temperature occurring during FSW is sufficiently high to cause dissolution, nucleation, and/or coarsening of the strengthening intermetallics in the weld heat affected zone (HAZ) and the thermo-mechanically affected zone (TMAZ). Our investigations have shown that transformations in the TMAZ/nugget interface of AA7050-T7 and the HAZ of AA7075-T6 and -T7 produce a sensitized microstructure, increasing susceptibility to intergranular attack, pitting and stress corrosion cracking (SCC). Measurements of the pitting potential and the SCC susceptibility, using the slow strain rate technique, have identified pre and post weld heat treatments which restore the corrosion resistance of the weld zones in these alloys. Changes in the corrosion properties of these materials will be compared with changes in the composition and morphology of the precipitates.

4:15 PM
Microstructure and Stress Corrosion Cracking Behavior of Friction Stir Welded Al Alloys: *Efstathios I. Meletis*[1]; [1]Louisiana State University, Mechl. Eng. Dept., Nicholson Ext., Baton Rouge, LA 70803 USA

Friction stir welding (FSW) was utilized to join a series of high-strength Al alloys, namely AA 2195, AA 2219 and AA 7075. All alloys were in the form of 0.23" thick plate, in the peak-aged temper. The microstructures of the various FSW zones were characterized by transmission electron microscopy. The stress corrosion cracking (SCC) behavior of the welded alloys was studied by conducting two types of experiments. First, four-point bending experiments were carried out in 3.5% NaCl solution under alternate immersion for 90 days. Second, slow extension rate tests (SERT) were conducted in laboratory air after different pre-exposure periods in NaCl solution under alternate immersion. Selective open circuit potential measurements of the various FSW zones were also conducted. The SCC behavior of the FSW alloys is discussed in view of the present experimental findings and current understanding of SCC behavior of Al alloys.

4:40 PM
Corrosion Fatigue Crack Growth and Initiation in FSW and GMA Welded Al 2519: *Peter S. Pao*[1]; E. Lee[2]; C. R. Feng[1]; H. N. Jones[1]; [1]Naval Research Laboratory, Code 6323, Washington, DC 20375 USA; [2]Naval Air Warfare Center, Aircraft Div., Patuxent River, MD 20670 USA

The corrosion-fatigue crack propagation and initiation characteristics of friction stir welded (FSW) and gas metal arc (GMA) welded Al 2519 in air and in salt water were investigated. TEM studies reveal the presence of very fine θí in the base metal, the coarsening of θí and the formation of //021 in the FSW HAZ, and the dissolution of most of the θí in the FSW weld. Microhardness mapping was obtained for both FSW and GMA welds to illustrate the drop in hardness in both weld and HAZ. Fatigue crack growth rates in the FSW weld and HAZ are significantly lower the threshold is significantly higher than those in the base metal. These changes may be attributed to the compressive residual stresses at the crack tip. Fatigue crack initiation lives of FSW welds are longer than GMA welds and the fatigue threshold of FSW weld approaches that of the base metal and is significantly higher than GMA weld.

5:05 PM
The Joint Properties of Dissimilar Formed A356 and 6061 Al Alloys by Friction Stir Welding Method: *Won Bae Lee*[1]; *Seung Boo Jung*[1]; *Yun-Mo Yeon*[2]; [1]SungKyunKwan University, Adv. Matls. Eng., 300 ChanAn-Gu, ChunChun-Dong, Suwon, Kyonggi-Do 440-746 Korea; [2]Suwon Science College, Automatic Welding Eng., Jungnam-myun, Botong-ri, Whasung, Kyoggi-do Korea

We observed the mechanical and metallurgical properties of friction stir welded joints of casting A356 Al alloy and wrought 6061 Al alloy whether these specimens fixed at retreating or advancing side. The specimens which was fixed at the retreating side mainly dominated the microstructure of stir zone. In case the 6061 Al alloys was fixed at the retreating side, the microstructure of stir zone was composed of mainly recrystallized structure of 6061 Alloys and some A356 Al alloy. The hardness of weld zone was slightly increased compare to that of A356 base metal due to the dispersed Si particles on Al matrix, but it showed lower hardness than that of 6061 Al alloys because the precipitates existed in base metal were dissolved at the weld by welding heat. In case of longitudinal tensile test, 6061 Al alloys which was fixed at the retreating side showed higher value than opposite case.

General Abstracts: Steels and Non-Metals

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Tuesday PM Room: 19
March 4, 2003 Location: San Diego Convention Center

Session Chair: Eric M. Taleff, University of Texas, Mechl. Eng. Dept., Austin, TX 78712-1063 USA

2:00 PM
The Effects of Carbon Content on the Tempering Response of R-Phase Strengthened Martensitic Stainless Steels: *Aytekin Hitit*[1]; Warren M. Garrison[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

We have investigated the effect of nickel contents varying from 4.5 to 6 wt.% on the strength and toughness if a 0.005C/12Cr/12Co/5Mo base steel. It was found that nickel additions enhanced strengthening by particles of R-phase and also improved the room-temperature Charpy impact energy. Nickel contents of 6 wt.% were associated with much lower ductile-to-brittle transition temperatures than those of the alloys containing smaller amounts of nickel. In this work the effects of carbon contents of 0.005, 0.25 and 0.05 wt.% on the properties of 12Cr/12Co/5Mo/4.5Ni and 12Cr/12Co/5Mo/4.5Ni alloys were investigated. For a nickel level of 4.5 wt.% increasing the carbon content increased the peak strength after tempering and lowered the temperature at which the peak strength was observed from 550∞C to 525∞C. Also, at a nickel content of 4.5 wt.% increasing the carbon content from 0.005 to 0.25 wt.% increased the peak hardness by 3 Rc and increasing the carbon level from 0.025 to 0.05 wt.% resulted in no further increase in hardness. At the nickel content of 6 wt.% increasing the carbon content increased the peak strength after tempering but did not change the temperature at which the peak strength was observed, 525∞C. In general it would appear that small additions to alloys of this type can result in substantial increases in strength without substantially reducing the room temperature impact energy.

2:20 PM
The Effects of Combining R-Phase and NiAl Strengthening on the Strength and Toughness of Martensitic Precipitation Strengthened Stainless Steel: *Aytekin Hitit*[1]; Warren M. Garrison[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

We have investigated the effect of nickel contents varying from 4.5 to 6 wt.% on the strength and toughness if a 0.005C/12Cr/12Co/5Mo base steel. It was found that nickel additions enhanced strengthening by particles of R-phase and also improved the room-temperature Charpy impact energy. Nickel contents of 6 wt.% were associated with much lower ductile-to-brittle transition temperatures than those of the alloys containing smaller amounts of nickel. While the toughness characteristics of the 6 wt.% nickel alloy were excellent the strength of the alloy was not sufficient for the alloy to be considered for landing gear applications. The purpose of this work was to investigate whether or not NiAl strengthening due to the introduction of aluminum would result in strength and toughness levels necessary for an alloy of this type to be considered for use in landing gear applications. For this work aluminum additions of 0.5, 0.72 and 0.9 wt.% were made to a 0.005/12Cr/12Co/5Mo/6Ni base steel. The required strength levels could only be achieved at an aluminum content of 0.9 wt.% but the toughness at this strength level was low.

2:40 PM
Crystallographic Influences on Bainite Kinetics and Morphology in Fe-C-Mo: *R. E. Hackenberg*[1]; G. J. Shiflet[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div. (MST-6), MS G770, Los Alamos, NM 87545 USA; [2]University of Virginia, Dept. of Matls. Sci. & Eng., Charlottesville, VA 22904-4745 USA

Diffusive processes often limit the growth of pearlite and bainite in alloy steels; crystallographic effects are thought to play only a minor role. Recent work on Fe-0.24C-4Mo decomposed at its bay temperature will be presented which highlights the significant influence of crystallography on the austenite decomposition microstructures. Observations of a significant substructure of differently oriented ferrite subunits (containing alloy carbides) in grain boundary-nucleated bainite are instrumental for understanding the faster thickening kinetics of grain boundary bainite relative to twin boundary bainite. This substructure originates in the crystallography of nucleation at grain and twin boundaries, and has significant consequences for the growth behavior. An additional finding, the observation of stepped ferrite + fibrous carbide growth fronts, calls into question the prior understanding that such growth fronts are incoherent.

3:00 PM
Investigation of Bainite Formation Using Neutron Bragg-Edge Transmission: *Jin Huang*[1]; Sven Vogel[2]; Mark Bourke[2]; Warren J. Poole[1]; Vincent Yuan[2]; Pascal Jacques[3]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., #309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Los Alamos National Laboratory, Struct. Prop. Relations (MST-8), Matls. Sci. & Tech. Div., MS H805, Los Alamos, NM 87545 USA; [3]University catholique de Loivain, Faculte des Scis. Appliquees, Dept. des Scis. des Materiaux, UCL-PCIM, 2 Place Sainte Barbe, Louvain-La-Neuve B-1348 Belgium

Structural phase transformations in solids can be investigated in-situ and in real-time using the Bragg-edge transmission technique. Neutron Bragg-edge transmission provides the crystallographic parameters volume fraction, lattice parameter and reflection width of participating phases with temporal resolutions in the order of tens of seconds. Additionally to the good temporal resolution, the neutron transmission setup allows to rapidly change sample temperature of bulk samples relatively homogeneously by splitting the sample into several disks. We demonstrate the technique by applying it to the investigation of the austenite decomposition to bainite in a silicon-rich steel. From the volume fraction, lattice parameter and edge width information we are able to derive information on transformation kinetics, carbon concentration and carbon homogeneity in the two phases, respectively. The findings of the neutron transmission measurements are compared with independent materials characterization methods like dilatometry, X-ray diffraction and metallography.

3:20 PM
Characterization of the Nucleation and Growth Behavior of Copper Precipitates in a Low Carbon Steel: *Michael S. Gagliano*[1]; Morris E. Fine[2]; [1]Ondeo Nalco, Dept. of Metall., One Ondeo Nalco Ctr., Naperville, IL 60563 USA; [2]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The nucleation and growth behavior of copper precipitates in ferrite were investigated both theoretically and experimentally in low carbon steel with and without niobium additions. Theoretical nucleation and growth rate models were constructed using calculated thermodynamic data in conjunction with classical theories. The maximum nucleation and growth rates for Cu were determined to be about 1016 nuclei/cm3s at 612C and 0.038 nm/s at 682C. Using an experimentally determined ìeffectiveî activation energy for the diffusion of copper, the theoretical nucleation rate curve compared very well with the hardness data after only 5 minutes of aging. The growth and coarsening behavior of the Cu precipitates were investigated through conventional TEM for isothermally aged samples cooled directly to the aging temperature immediately following austenitization. For aging times up to five hours, the average precipitate size scaled with a time dependence of while longer aging times showed a time dependence of t0.28.

3:40 PM Break

3:50 PM
Structured Methodologies for Material Processing Solutions: *ReneÈ Miller*[1]; [1]John Deere, Heat Treat, PO Box 270 D188, Waterloo, IA 50704-0270 USA

Data Focus, Decision Analysis, Juran QIP and Design of Experiments are a few of the quality and management tools available to assist Metallurgists in resolving material processing issues. This paper will briefly outline the concepts of these tools, their advantages, and examples of their applications in material processing.

4:10 PM
Quantification of Structural Disorder in Oxide Glasses: Quantum Chemical Calculations and Spectroscopic Analysis: *Sung Keun Lee*[1]; [1]Carnegie Institution of Washington, Geophysl. Lab., 5251 Broad Branch Rd., Washington, DC 20015 USA

Oxide glasses, including borosilicates and aluminosilicates and their precursor liquids, have long been studied because of their diverse applications in the glass and ceramic industry and the strong implications of their structures to the properties of magmas. Essential to the macroscopic thermodynamic and transport properties of oxide glasses and melts is the full understanding of the atomic arrangements and the extent of disorder among atoms. However, quantitative estimation of the extent of the inherent disorder in silicate glasses and melts has remained a complex and difficult problem, largely because of the limits of resolution of conventional experimental techniques. Here, we present our recent results on quantification of the disorder and site connectivity among framework cations and oxygens in alumino- and borosilicate glasses using multi nuclear multiple quantum (MQ) magic angle spinning (MAS) NMR and quantum chemical calculations based on density functional theory (DFT). These methods combined with statistical mechanical modeling allowed us to evaluate the extent of disorder in terms of the degree of Al-avoidance and degree of phase separation and provided improved understanding of the configurational thermodynamic properties of corresponding melts. Several oxygen sites (e.g. Si-O-Al, Al-O-Al, B-O-B, B-O-Si, and Si-O-Si) are clearly resolved in O-17 MQMAS NMR spectra of alumino- and borosilicate glasses. About 10% Al-O-Al in NaAlSiO4 glasses suggests that Al-avoidance is imperfect in fully polymerized aluminosilicate glasses and melts. The degree of Al avoidance (Q) in Ca- and Na- aluminosilicate glasses from the experimental data ranges from 0.95-0.85 where Q=1 and 0 refers complete Al avoidance and random distribution respectively. From the DFT calculated relative energy differences among each oxygen cluster in binary borosilicate glasses (Si-O-Si, Si-O-B, and B-O-B), the stability of Si-O-B was quantified. The fraction of Si-O-B at XB=0.57 is about 36.5 % (±1), suggesting a significant inter-dispersion of B and Si, while a random distribution of Si and B and clustering of similar framework lead to 50 and 0% of Si-O-B respectively. This prediction is consistent with experimental results from O-17 3QMAS NMR. The similar methods have been extended to the study of the extent of disorder in mixed cation glasses where several types of non-bridging oxygen such as Na-O-Si, Ca-O-Si, Ba-O-Si and (Ca,Na)-O-Si are well resolved under O-17 MQMAS NMR. The oxygen site population data as well as Na-23 NMR results supports the random distribution of each cation. We also calculated configurational thermodynamic properties including configurational heat capacity and entropy as a function of composition, temperature and as well as the extent of Al avoidance and phase separation, which shows remarkable similarity with experimental results by solution calorimetry.

4:30 PM
Evaluation of Ionic Liquids as Heat Transfer Fluids for Solar Thermal Parabolic Trough Technology: Ramana G. Reddy[1]; *Mario F. Arenas*[1]; Zhijing Zhang[1]; [1]The University of Alabama, Metallurgl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487 USA

Long-term thermal stability and corrosivity against engineering materials are important for the evaluation of ionic liquids as potential energy storage and heat transfer fluids for solar thermal power systems. In this investigation, thermogravimetric analyses (TGA) and electrochemical corrosion tests were carried out on various ionic liquids, namely 1-butyl-3-methylimidazolium chloride ([C_4mim]Cl), 1-hexyl-3-methylimidazolium hexafluorophosphate ([C_6mim]PF_6), 1-octyl-3-methylimidazolium hexafluorophosphate ([C_8mim]PF_6), and 1-butyl-3-methylimidazolium bis(trifluromethanesulflonyl)imide ([C_4mim][Tf_2N]). Experimental results showed that these ionic liquids exhibit a highest decomposition temperature of 400∞C. However, long-term thermal stability tests indicated significant weight losses after 20 h of exposure at the temperature of 333∞C. Among the ionic liquids investigated, [C_8mim]PF_6 and [C_4mim][Tf_2N] were found to be the more thermally stable. The corrosivity of ionic liquids against 316 stainless steel and 1018 carbon steel was investigated at room temperature. Both Tafel plots and polarization curves were used to characterize corrosion behavior of the alloys in ionic liquids. The alloys were found to exhibit outstanding resistance to uniform corrosion.

4:50 PM
Mutual Effects of Electro Slag Remelting and Open Die Radial Forging on the Properties of V-Microalloyed Steel: *Taha MohamedTaha Mattar*[1]; [1]CMRDI, Steelmaking, PO Box 87, Helwan, Cairo 11421 Egypt

Both electro-slag remelting (ESR) and forging processes have a pronounced effect on the steel properties. ESR process as a refining process improves the cleanliness, soundness, homogeneity of alloying elements, and consequently the properties of steel. At the same time, metal forming processes are usually carried out not only for dimensional purposes but also enhancing the mechanical properties of steel. Better properties are obtained through grain refining of a desired microstructure. The present work aims at studying the mutual effects of both ESR using different refining slag compositions and open die radial forging process on the mechanical properties and structure of V-microalloyed steel. In this study V-HSLA steel consumable electrodes were produced in an electric arc furnace (EAF). These electrodes were electro-slag remelted into ingots of 120 mm diameter. The ingots produced by ESR were subjected to open die radial forging in the temperature range 1200-800°C. Forging has been carried out to different amounts of reduction in cross-sectional area. The forged bars were subjected to both mechanical testing and microstructure investigations. The obtained data showed pronounced improvements in the microstructure and mechanical properties upon controlling ESR slag composition and amount of reduction in cross-sectional area.

5:10 PM
Quantitative Investigation of Hydrogen-Induced Additive Stress in Steel: *Wuyang Chu*[1]; [1]University of Science and Technology Beijing, Dept. of Matls. Physics, Haidianqu Xueyuan lu No. 30, Beijing 100083 China

The flowing stress of a high-strength steel before unloading is different with the yield stress of the same sample extended in air after unloading and charging with hydrogen. The difference is the hydrogen-induced additive stress, which can help the applied stress to enhance the plastic deformation. The hydrogen-induced additive stress, Sigama(ad), induced through being immersed in a 3.5% NaCl solution with pH=4 increases linearly with the yield strength of the sample, e.g., Sigama(ad)=-106.6+0.14Sigama(ys). On the other hand, hydrogen-induced additive stress increases linearly with the logarithm of hydrogen concentration in the samples with the yield strengths of 900 and 1050MPa, i.e., Sigama(ad)=-55.5+63.6lnC0(Sigama(ys)=900MPa) and Sigama(ad)=-23.5+64.2lnC0(Sigama(ys)=1050MPa). To sum up, the hydrogen-induced additive stress is Sigama(ad)=-260+0.226Sigama(ys)+63.9lnC0.

Global Development of Copper and Gold Deposits - II

Sponsored by: Extraction & Processing Division, EPD-Process Mineralogy Committee, EPD-Precious Metals Committee
Program Organizers: Tzong T. Chen, CANMET, Ottawa, Ontario K1A 0G1 Canada; Corby G. Anderson, Montana Tech of the University of Montana, Center for Advanced Mineral & Metallurgical Processing, Butte, MT 59701-8997 USA; Steven L. Chryssoulis, Amtel, London, Ontario N6G 4X8 Canada

Tuesday PM Room: 1B
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Stephen Chryssoulis, Amtel, London, Ontario N6G 4X8 Canada; James Y. Hwang, Michigan Technological University, Inst. of Matls. Procg., Houghton, MI 49931-1295 USA

2:30 PM
Removal and Recovery of Tellurium/Selenium from Copper Slimes Leachate: *Shijie Wang*[1]; Brad Wesstrom[1]; Jean Fernandez[1]; [1]Phelps Dodge Mining Company, El Paso Ops., 6999 N. Loop Dr., El Paso, TX 79915 USA

In pressure oxidation leaching of copper anode slimes, more than 65% of the tellurium is co-extracted during decopperizing. About 10-15% of the selenium is also dissolved simultaneously by severe leach conditions. Current operation for the treatment of the leachate is to constantly recirculate the leach solution through a bed of copper turnings. The tellurium and selenium are precipitated from the leach liquor as copper telluride and copper selenide and separated from the solution by filtration. In order to improve the reaction efficiency and reduce the process cost, viz. minimize the copper addition, cut retention time, and reduce the energy consumption; a chemical reducing agent is studied and the cementation process tested in the laboratory. In this paper, the test results are presented and the process chemistry is discussed.

3:00 PM
Characterization of Copper Vermiculite Antibacterial Nanomaterial: Bowen Li[1]; Xiaodi Huang[1]; Zhiyong Xu[1]; *J. Y. Hwang*[1]; [1]Michigan Technological University, Inst. of Matls. Procg., 1400 Townsend Dr., Houghton, MI 49931-1295 USA

Silver, copper, and zinc are elements with good antibacterial and antifungal capabilities. Copper vermiculite has been prepared by ion exchange method. The antibacterial and antifungal properties of the prepared products have been demonstrated. Chemical and mineralogical characterization of the material is provided in this study.

3:30 PM
Reducing the Calcium Hydroxide Consumption in Copper Flotation: *Cristiano Zanforlin*[1]; Richard Araya[2]; [1]Cal Itau-Votorantim Group, Av. Dr. Jorge Dias de Oliva, 3301, Sao Jose da Lapa-Minas Gerais CEP: 33.350-000 Brazil; [2]Codelco Chile

Reducing costs in the flotation process is an important issue for the copper industry. However it commonly focuses the attention only on the chemical products which are more value added like flocculants or depressors. In spite of playing an important role in the flotation process, calcium hydroxide has never been subject of studies. Calcium hydroxide is used to correct the pH in the flotation in order to depress the pyrite from the copper ore. It is obtained by slaking the quicklime in water. The purpose of this work is investigating the performance of a quicklime developed for flotation process, the Flotlime Series\@. The product was approved in lab and pilot plant tests by Codelco-Chile, and then were conduced plant trials in El Teniente, Andina and Chuquicamata. The quicklime consumption was reduced in 40% and was observed an increase in molybdenum recovery.

4:00 PM
Diagnostic Metallurgy of Porphyry Copper Ores: *Joo Kim*[1]; [1]120 Pendennis Dr., Pointe-Claire, QC H9R 1H6 Canada

The processing of porphyry copper ores is highly variable mostly due to differences in ore mineralogy and nature of oxidations. Statistically designed flotation experiments were carried out with a number of different porphyry ores around the world to correlate the mineralogical significances to the metallurgical prediction. Various option of processing porphyry ores, such as flotation, gravity concentration and cyanidation are considered singularly or in combination with one another.

4:30 PM
Bio-Oxidation of Arsenic-Bearing Gold Ores in China: *Yang Hongying*[1]; Yang Li[2]; He Yongjun[2]; Fan Youjing[1]; Wang Junfeng[2]; [1]Northeastern University, Shenyang 110006 China; [2]Shandong Tarzan Bio-Gold Company, Ltd., Laizhou 261441 China

Many arsenic-bearing gold deposits have been found recently in China. The ores are treated using biological, roasting, or pressure-oxidation techniques, with bio-oxidation considered the best processes. Bio-oxidation plants of 50 t/d to 100 t/d have been built in Yantai and Laizhou, Shandong province during 2000 to 2001. The plants treat arsenic-bearing gold concentrates which contain 4-6% As, and gold recoveries are over 95%. Also, several companies in Shaanxi apply heap leaching technology to treat the ores. However, because of possible arsenic pollution, most arsenic-bearing gold deposits in China have not been exploited. The Chinese government now promotes the ìfriendly environmental metallurgyî; consequently, it is expected that bio-oxidation technology will become more important for treating arsenic-bearing gold ores.

5:00 PM
Effects of Roasting Conditions on Copper and Zinc Ferrites ($CuFe_2O_4$, $Cu_xZn_{1-x}Fe_2O_4$ and $ZnFe_2O_4$), Production and an Investigation on Parameters Affecting Cuo and Zno Co-Leacheability: *A. F. Mulaba-Bafubiandi*[1]; [1]Technikon Witwatersrand, Rsrch. Dvlp. Unit, Fac. of Eng., PO Box 526, Wits, Johannesburg 2050 S. Africa

In the hydrometallurgy of zinc, copper and zinc are generally extracted from calcine produced by roasting zinc-copper sulphides concentrate. The concentrate in a form of sulphides is subjected to high temperature conditions to convert the sulphides into oxides. The resulting calcine contains copper and zinc in a form of copper oxide (CuO), zinc oxide (ZnO), copper sulphate ($CuSO_4$), zinc sulphate ($ZnSO_4$), and copper and zinc ferrites $CuFe_2O_4$, (Zn_{1-x}, Cu_x)OFe_2O_3, and ($ZnFe_2O_4$), ferrites being co-produced during the high temperature roasting process. Their insolubility in relatively dilute sulphuric acid and low temperatures results in a significant amount of zinc and copper remaining in the residue. Zinc ferrite and copper ferrite are therefore sources of metal loss. The ferrite formation has been scrutinized in order to minimize their formation. XRD, the sulphur removal test and Mˆssbauer spectroscopy were used to study the optimization process. A relatively dead roasting was achieved at 900°C for 3 hours. At 800°C Copper and zinc oxides (CuO and ZnO) formation dominated over the competing zinc ferrite and other ferrites (copper ferrite). Mˆssbauer results show the ferrites in different forms with hyperfine interactions parameter values confirmed by the literature (Stevens, et. al, 1998). Their abundances are found to depend on the roasting conditions. The rate of hematite formation decreased with increase in temperature and on the contrary, the ferrites were forming with increase in temperature. The optimum temperature was achieved at 800°C for 3 hours. Results on the optimisation of the roasting process were published somewhere else (Sibiya-Magagula and Mulaba-Bafubiandi, 2002). Leaching experiments were done using three different acids (HCl, H_2SO_4 and HNO_3) at 50°C and later at 90°C using mild (15g/l) and strong (55g/l) concentrations respectively. The HCl resulted in the highest percentage extraction of all the metals. Above 90% of copper was extracted from neutral leach and a percentage of only 80-85 for iron and zinc could be extracted from neutral leach. References: 1. J.G. Stevens, A.M. Khasanoy, J.W. Miller, H. Pollak and Z.Li. Mˆssbauer Mineral Handbook. Mˆssbauer Effect Data Centre, 1998. F.N. Sibiya-Magagula and A.F Mulaba-Bafubiandi, SAIMM Proceedings, July 2002.

Hot Deformation of Aluminum Alloys: Superplasticity and Creep

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA; Armand J. Beaudoin, University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; Balasubramaniam Radhakrishnan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6359 USA

Tuesday PM — Room: 6E
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Terence G. Langdon, University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA; Rajiv S. Mishra, University of Missouri, Dept. of Metallurgl. Eng., Rolla, MO 65409 USA

2:00 PM Invited
Creep and Superplasticity in Aluminum Alloys: *Farghalli A. Mohamed*[1]; [1]University of California, Dept. of Cheml. Eng. & Matls. Sci., 916 Eng. Tower, Irvine, CA 92697-2575 USA

It is well-documented that trace levels of impurities, solute atoms, precipitates, and dispersion particles play an important role in the deformation and fracture of polycrystalline materials. At temperatures above about 0.5 Tm, where Tm is the melting point of the material, this role is reflected in several phenomena that include the segregation of impurities at boundaries, the occurrence of viscous glide creep, and the introduction of a threshold stress for creep in dispersion strengthened (DS) alloys. This paper provides recent examples that demonstrate the role of trace elements, solute atoms, and dispersion particles in modifying high-temperature deformation of aluminum and its alloys. These examples deal with creep in powder metallurgy (PM) Al alloys, very low-stress creep behavior in Al, and superplastic flow in Zn-22% Al.

2:25 PM
Creep Properties of Heat-Treatable Al-Mg-Sc Alloys at 300∞C: *Emmanuelle A. Marquis*[1]; David N. Seidman[1]; David C. Dunand[1]; [1]Northwestern University, Matls. Sci. & Eng. Dept., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

A study of the creep properties at 300∞C of precipitation-strengthened Al-Sc alloys containing Mg in solid solution is presented. Threshold stresses were measured in the range of 0.09sOr to 0.6sOr, where sOr is the Orowan stress. At large applied stresses, the creep strength is significantly improved as compared to binary Al-Sc alloys, and is independent of the size of the Al3Sc precipitates with a stress exponent close to 5. To study the effect of precipitate dimensions on the threshold stress, aging treatments were varied to form Al3Sc precipitates with average radii ranging from 2 to 30 nm. The effect of coherency state of the matrix/precipitate interface was also investigated, as precipitates lose coherency for radii larger than ~12 nm. Existing models for climb-controlled bypass mechanisms are extended to describe the precipitate radius dependence of the measured threshold stresses, as well as the effect of dislocations at the precipitate interfaces.

2:45 PM Invited

High Strain Rate Superplasticity of Metal Matrix Composites: *Tsunemichi Imai*[1]; Naofumi Saito[1]; Shangli Dong[1]; [1]National Institute of Advanced Industrial Science & Technology, Inst. of Structl. & Eng. Matls., 2266-98 Shimoshidami, Moriyama-ku, Nagoya 463-8650 Japan

Hot-rolling after extrusion are used as thermomechanical processing to build fine grain size in matrix, to control interface between matrix and reinforcement and to disperse reinforcement uniformly for Metal Matrix Composites(MMC) and the high strain rate superplasticity (HSRS) of MMC is investigated. (1) Pure aluminum (1N90) fabricated by PM method exhibits maximum total elongation more than 500% and at the strain rate of 10-2s-1 and at 893K, which is below of melting temperature of pure aluminum. And TiN/1N90, TiC/1N90 and AlN/1N90 indicate about 200% elongation at the strain rate of 10-1s-1 and at 913K. It is thought, therefore, that HSRS could produce by fine grain boundary sliding and accommodation mechanism with dislocation movement. (2) TiN/2124, TiC/2124 and AlN/6061 Al composites exhibits larger total elongation of 300~500% at the strain rate of 10-0s-1 and above solidus temperature of matrix alloys so that these composites could produce HSRS by interfacial sliding with partial melting phase in addit ion with grain boundary sliding. (3) Also nitrided mullite short fiber reinforced 60661 Al composites produces HSRS because mullite short fibers are covered by Sialon particle on the surfaces and Sialon is available to avoid reaction of mullite with magnesium.

3:10 PM

High Strain Rate Superplastic Deformation of 30 vol.% AlNp/6061Al Composite: *Lihong Han*[1]; Jitai Niu[2]; Henry Hu[1]; [1]University of Windsor, Dept. of Mechl., Auto. & Matls. Eng., Windsor, Ontario N9B 3P4 Canada; [2]Harbin Institute of Technology, Sch. of Matls. & Tech. Eng., Harbin, Hei Long Jiang 150001 China

The superplasticity of 30 vol.% AlN particulate-reinforced 6061Al composite, prepared by Powder Metallurgy techniques (PM), was investigated. In this study, superplastic tensile tests of the composite were carried out at strain rates ranging from 100-10-3 s-1 and at temperatures from 823K to 893K. Hot rolling of the composite after extrusion was employed to obtain a fine-grained structure prior to superplastic testing. More than 300% total elongation was achieved at a temperature of 853~873 K and at an initial strain rate of 1.67x10-1 s-1. The results indicate that the highest value of the strain rate sensitivity index (m) is 0.42 for the composite. A differential scanning calorimeter (DSC) was used to ascertain the possibility of any partial melting in the vicinity of optimum superplastic temperatures. Microstructure and fracture behavior of the composite were examined with a Scanning Electronic Microscope. Characteristics of the interface between the reinforcement and the matrix were also investigated by using a Transmission Electronic Microscope.

3:30 PM Invited

Review of Current Commercial Applications for High Strain Rate Superplastic Aluminum Alloys: *Kenji Higashi*[1]; *Toshiji Mukai*[2]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1, Gakuen-cho, Sakai, Osaka 599-8531 Japan; [2]Osaka Municipal Technical Research Institute, 1-6-50 Morinomiya, Joto-ku, Osaka 536-8553 Japan

Superplasticity is a viable technique to fabricate a hard-to-form material into complex shape. The detailed feature has been investigated in the past two decades. The limitation to apply for commercial mass-production is the low optimum forming speed and the material cost. One of the innovations in superplasticity is especially noted to establish the high strain rate superplasticity(HSRS) in early 1990s. Detailed study of the phenomenological aspect in HSRS revealed its deformation mechanism and pointed out the importance of the presence of accommodation helper such as discontinuous liquid phase with grain boundary sliding. Recently, HSRS has been applied for net-shape forging of engine piston with rapidly-solidified Al-Si system alloy powders. Another commercial application of HSRS is gas-pressure forming of Al-Mg system alloys to produce large scale components, i.e., constructing outer parts, roof of automobiles. In this study, we discuss the phenomenological deformation mechanism of HSRS in aluminum alloys and review of the current applications in Japan.

3:55 PM

Characterization of Superplastic Response in Al-Mg Alloys: *P. A. Friedman*[1]; W. Copple[1]; [1]Ford Research Laboratory, 2101 Village Rd., MD 3135, Dearborn, MI 48121 USA

The ability to achieve large strains to failure coupled with extremely low flow stresses makes superplastic forming an attractive option in the automotive industry for the manufacture of complex parts from aluminum sheet. However, a barrier to increased usage is the cost penalty associated with alloys that are specially processed to have a small and stable grain size which makes them suitable for superplastic forming. In this paper, a series of elevated-temperature tensile tests are used to characterize the superplastic response of Al-Mg alloys that were specially processed for superplastic forming as well as an alloy which was conventionally processed. Results from these tensile tests coupled with optical microscopy are used to both establish the superplastic forming potential of these alloys as well as to highlight the different mechanisms that control deformation in the two types of materials.

4:15 PM

Thermo-Mechanical Process Optimization for Enhancing Superplastic Ductility of 5083 Aluminum Sheet: *Ravi Verma*[1]; [1]General Motors, Matls. & Processes Lab., 30500 Mound Rd., Warren, MI 48090 USA

Effect of sheet rolling practice on superplastic response of 5083 aluminum alloy has been investigated. A synergistic effect between hot rolling and cold rolling processes has been observed, which helps optimize an efficient sheet rolling practice for producing superplastic sheet. In addition, a ìskewedî cold-rolling practice is proposed in which successive rolling reduction levels are tailored to maximize the alloyís hardening response to cold work. This optimized cold rolling practice increases the amount of work introduced into the sheet (as measured by sheet hardness) for a given net gauge reduction, and thereby improves the superplastic response of the 5083 sheet on recrystallization. Superplastic ductility values greater than 400% have been obtained with the optimized rolling practice.

4:35 PM

Superplasticity in a Commercial Al-Mg-Mn-Sc Alloy Subjected to Intense Plastic Straining: *Fanil Musin*[1]; Rustam Kaibyshev[2]; Yoshinobu Motohashi[1]; [1]Ibaraki University, Rsrch. Ctr. for Superplasticity, Nakanarusawa-cho, 4-12-1, Hitachi, Ibaraki 316-8511 Japan; [2]Institute for Metals Superplasticity Problems, Khalturina str., 39, Ufa 450001 Russia

The superplastic behavior of an Al-5.76%Mg-0.3%Mn-0.32%Sc alloy (1570 Al) subjected to intense plastic straining by equal-channel angular extrusion (ECAE) was studied in the temperature interval 200-500°C at strain rates ranging from $1.4x10^{-5}$ to $1.4\ s^{-1}$. The grain size after ECAE was about 1µm. The highest elongation to failure of 2000% was recorded at a temperature of 450°C and initial strain rate of $5.6x10^{-2}\ s^{-1}$ with corresponding coefficient of the strain rate sensitivity of 0.6. It was shown that the ECAE processed 1570 Al exhibits superior superplastic properties in the temperature range 300-500°C with the strain rate sensitivity higher than 0.4. Microstructural evolution and cavitation during high strain rate superplastic deformation were examined.

4:55 PM

Achievement of High Superplastic Properties of Al-Mg-Si Alloys in Partially Melted State: *Rustam Kaibyshev*[1]; Fanil Musin[1]; Dmitry Gromov[1]; T. G. Nieh[2]; Donald R. Lesuer[2]; [1]Institute for Metals Superplasticity Problems, Khalturina str., 39, Ufa 450001 Russia; [2]Lawrence Livermore National Laboratory, L-342, PO Box 808, Livermore, CA 94551 USA

The superplastic properties of a commercial grade of 6061 aluminum alloy (6061Al) and a modified (0.15%Zr-0.7%Cu) 6061Al were examined in tension at T=475-620∞C and at a strain rate range of $7x10^{-6}$-$2.8x10^{-2}s^{-1}$. The same two-step thermomechanical processing was used to obtain the refined microstructure in both alloys. It was shown using differential thermal analysis that the highest superplastic characteristics were obtained in partially melted state. The modified 6061Al exhibited a maximum elongation-to-failure of 1300% at 590∞C and at a strain rate of $2.8x10^{-4}s^{-1}$. In contrast, the highest total elongation of 350% was achieved in the commercial grade 6061Al at 600∞C and at a strain rate of $1.4x10^{-4}s^{-1}$. Significant enhancement of the superplastic properties of the modified 6061Al in partially melted state was provided by optimum amount of liquid phase contributing proper accommodation of grain boundary sliding as well as by essential stability of the fine-grained structure with Al_3Zr dispersoids.

International Symposium on Gamma Titanium Aluminides: Environmental Effects and Applications

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee

Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

Tuesday PM Room: 6F
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Andrew H. Rosenberger, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA; Shigehisa Naka, ONERA, Materiaux Metalliques et Procedes, Chatillon Cedex 92322 France

2:00 PM Invited

Oxidation Resistance of γ-TiAl Based Alloys in a Simulated Combustion Atmosphere: *Shigeji Taniguchi*[1]; Xiangyang Li[1]; [1]Osaka University, Matls. Sci. & Procg., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

The cyclic oxidation behaviour of the γ-TiAl based alloys, Ti-48Al-2Cr-2Nb, Ti-48Al-2Cr-2Fe, Ti-48Al-2Cr-2W and Ti-48Al-2Cr-1W-1Ta (at. %), was examined in a simulated automobile exhaust gas at 1123 K for up to 1080 ks (300 h) to evaluate their practical performance under aggressive environment. The oxidation products were identified by XRD and the microstructure, elemental constitution of the oxide scale were characterized by SEM and EPMA. The effects of the alloying element on the oxidation resistance were discussed on the basis of above evidence. The alloys except Ti-48Al-2Cr-2Fe showed excellent oxidation resistance for at least 15 cycles (300 h) in which Ti-48Al-2Cr-2W gives the best performance followed by Ti-48Al-2Cr-1W-1Ta and Ti-48Al-2Cr-2Nb. The alloy additions of W, Ta and Nb could enhance the cyclic oxidation resistance of TiAl alloy in simulated exhaust gas and Fe shows detrimental effect. Further investigation indicated that alloying elements such as W, Ta and Nb played a significant role in promoting the primary oxidation of Al, namely resulted in large amounts of alumina in the oxide scale and eventually led to the overall low growth rate and strong oxidation resistance.

2:30 PM

Environmental Protection of Gamma Titanium Aluminides: *Christoph Leyens*[1]; Reinhold Braun[1]; Papken Eh. Hovsepian[2]; Wolf-Dieter M¸nz[2]; [1]DLR-German Aerospace Center, Inst. of Matls. Rsrch., Linder Hoehe, Cologne D-51170 Germany; [2]Sheffield Hallam University, Matls. Rsrch. Inst., City Campus, Howard St., Sheffield S1 1WB UK

The current development of new generation gamma titanium aluminides is expected to result in alloy chemistries and microstructures capable of resisting temperatures well in excess of 850∞C. Under these conditions, environmental and thermal protection becomes a concern since oxidation and wear/erosion might eventually limit the maximum service temperatures achievable. In the present paper, two approaches will be highlighted. Advanced nitride coatings based on the Ti-Al-Cr-Y-N system have demonstrated excellent oxidation resistance under isothermal and cyclic conditions up to 900∞C in long-term tests of several thousand hours. In addition to oxidation resistance these coatings provide excellent wear protection. Along with the oxidation data a detailed microstructure analysis before and after testing will be presented demonstrating the thermal stability and integrity of the coatings. Furthermore, EB-PVD thermal barrier coatings applied to gamma titanium aluminides will be highlighted. The TBCs survived 1000 h at 850 and 900∞C without failure before the tests were terminated. Microstructure analysis will emphasize the outstanding performance of the coatings.

2:50 PM

The Oxidation Behavior of Engineering Gamma TiAl Alloys: *Michiko Yoshihara*[1]; Young-Won Kim[2]; [1]Yokohama National University, Mechl. Eng. & Matls. Sci., 79-5 Tokiwadai, Hodogaya-ku, Yokohama, Kanagawa 240-8501 Japan; [2]UES, Inc., Matls. & Processes, 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

The present paper is a part of continuing work concerning the oxidation behavior of engineering gamma TiAl alloys under development. The gamma alloys have attractive properties as light weight heat resistant structural materials, however the oxidation resistance of them at elevated temperatures is not enough. For practical applications, it is important to understand the oxidation behavior of the alloys. This paper provides recent knowledge about the oxidation resistance of the gamma alloys in air, with isothermal exposure at 870∞C up to 200h as well as two hours cyclic exposure at 760∞C, 815∞C and 870∞C up to 1000 hours. The materials tested include five cast alloys (K5C, 395E, K1A, K1B and K1C) and seven forged alloys (95D, K5A, 395N, 98B, 99B, 99D and 99G). The alloy with high Nb content showed good oxidation behavior, however other alloying elements also have some influences on oxidation behavior. Detailed results will be discussed.

3:10 PM

Development of Oxidation Resistant Coatings Based on Gamma TiAl: *Panos Tsakiropoulos*[1]; Antonis Zaroulias[1]; Guosheng Shao[1]; [1]University of Surrey, Sch. of Eng., Mechl., Matls. & Aeros. Eng., Guildford, Surrey GU2 7XH UK

The development of gamma TiAl base alloys has addressed their oxidation and mechanical properties. The selected alloying additions to gamma and gamma + a2 alloys do not result in continuous Al2O3 scale formation in air. An intermixed Al2O3/TiO2 scale continues to form in the more complex alloys, but the rate of growth of this scale has been reduced. Alloys with Cr additions at a level generally above 8 to 10 at% can form a two-phase (gamma + Laves phase) microstructure and are capable of continuous Al2O3 scale formation in air. Furthermore, the Ti-Al-TM tau phases form protective Al2O3 in air, with the best oxidation behaviour exhibited by TM = Cr. Thus, the Ti-Al-Cr system offers unique opportunities for the design of coatings that are mechanically compatible with the substrate and also oxidation resistant. The paper will report results of our experimental study of Ti-Al-Cr coatings applied by PVD on gamma base substrates. The coatings have been designed for oxidation resistance using thermodynamic database s and microstructure models developed at the University of Surrey. A distinct feature of our study is the deposition of amorphous coatings with high crystallisation temperatures. The design and processing of coatings will be presented. The microstructures and properties of the as deposited and nanocrystalline coatings will be discussed.

3:30 PM

Moisture-Induced Embrittlement of Isothermally Forged TiAl-Based Intermetallic Alloys with Various Kinds of Microstructures: *Takayuki Takasugi*[1]; Yasuyuki Kaneno[1]; Hirofumi Inoue[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

Isothermally forged TiAl-based intermetallic with various microstructures (gamma grain, duplex, dual phase and fully lamellar microstructures) were prepared. These TiAl-based intermetallic alloys were tensile tested in vacuum and air as functions of strain rate and temperature. All intermetallic alloys and all microstructures showed reduced tensile strength (or elongation) in air at room temperature. The tensile strength (or elongation) of the specimens deformed in air tended to recover to the values of the specimens deformed in vacuum as temperature (or strain rate) increases. From the measured recovering temperatures, it was found that the gamma grain microstructure was the most resistive to the moisture-induced embrittlement, and the dual phase microstructure was the most susceptible to the moisture-induced embrittlement. Also, the moisture-induced embrittlement of the alloys with fully lamellar microstructure was reduced with decreasing lamellar spacing. The observed microstructural effect on the moisture-induced embrittlement was explained, in association with hydrogen properties and kinetics in the constituent phases and at some interfaces.

3:50 PM Invited

Texture Formation in Gamma-TiAl: *Arno Bartels*[1]; Helmut Clemens[2]; Wolfram Schillinger[1]; [1]TU Hamburg-Harburg, Matls. Sci. & Tech., Eissendorfer Str. 42, Hamburg D-21073 Germany; [2]GKSS-Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str. 1, Geesthacht D-21502 Germany

Processing of gamma-TiAl based alloys like forging or rolling often results in strong textures, which lead to undesirable anisotropies of mechanical properties. To avoid these textures the knowledge of texture formation during processing is doubtless necessary. It will be shown that during deformation at high temperatures texture components formed comparable to those known from fcc-metals with low stacking fault energies. Due to the tetragonality of the crystalline structure these components are split with respect to the difference between a- and c-axes. Components predominantly formed by slip of superdislocations or by mechanical twinning are vanishing during recrystallization in the processing heat. After forging and rolling new recrystallization induced texture component occur with strong alignments of the [001]-directions resulting in anisotropies of the mechanical properties. To avoid these anisotropies the recrystallization process must be diminished by a special choice of processing parameters or by an alloy exhibiting a lower inclination to recrystallization.

4:20 PM Invited
Titanium Aluminide Turbine Wheels for Turbocharger Applications: *Stephen J. OíHara*[1]; [1]Caterpillar, Inc., Air Sys., 3701 State Rd. 26 E., Lafayette, IN 61656 USA

Internal combustion engine makers are pressed by legislation to develop and manufacture cleaner engines. At the same time they continue their drive for improved fuel consumption and competitive sales costs in order to maintain market share. Turbocharger technologies such as EGR; VGT; Series and Sequential machines play an ever increasing role in providing the engine maker with enhancements to the engine exhaust and air intake systems. The Titanium Aluminide turbine wheel offers the turbine designer a low mass, low inertia alternative to alloys such as Inconel 713C. In turn this would allow better high temperature materials such as Titanium to be used in the compressor section of the rotating group without increasing rotor inertia. The improved specific strength and stiffness of Titanium Aluminide may allow the designer more scope for Improved turbine design.

4:50 PM Invited
Titanium Aluminides for Automotive Applications: *Hartmut Baur*[1]; Daniel B. Wortberg[1]; Helmut Clemens[2]; [1]DaimlerChrysler AG, RBP/SM, Wilhelm-Runge-Str. 11, Ulm 89081 Germany; [2]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Strafle, Geesthacht 21502 Germany

Titanium aluminides have proven their principle suitability for automotive applications. This paper evaluates the technical benefits of titanium aluminides for rotating and oszillating components based on first engine tests. These promising results on the one hand and the limited availability of high qualitative titanium aluminides components in acceptable quantities and at reasonable costs on the other hand have led to extensive research efforts. This paper summarizes the current state of titanium aluminides with regard to material properties, production process, quality and quality control of cast prototypes as well as cost situation. Critical points and the demand on future research and development will be outlined. These points will be elaborated on the basis of the potential use of titanium aluminides for turbocharger wheels. Besides this component possible other applications in automotive combustion engines will be presented. Finally, the strategy to promote titanium aluminides into series production will be discussed.

5:20 PM
A Microstructural Study of the Fusion Zone of Gas Tungsten Arc Welded Gamma Titanium Aluminde: Mario F. Arenas[1]; *Viola L. Acoff*[1]; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., Box 870202, Tuscaloosa, AL 35487-0202 USA

Although various studies have been performed on the weldability of gamma titanium aluminides, little is known about the microstructure that evolves during the gas tungsten arc welding process. This technique has been proposed for gamma repair operations due to its flexibility and relative cost-effectiveness. In this study cast, hot-isostatic pressed (HIPed) gamma titanium aluminides alloys of composition Ti-48Al-2Cr-2Nb were welded using GTAW without preheating. Microstructural examination revealed a nearly lamellar microstructure in the base metal which was transformed to dendritic with evidence of interdendritic gamma phase and metastable structures in the fusion zone. The solid state transformations that occurred were heavily dependent on the cooling rate. It was found that the selected welding parameters should ensure that the heat affected zone cooling rate will be less than 500 K/s. Such cooling rates prevent the development of deleterious alpha-2 structures, which promote cracking.

5:40 PM
Microstructural Aspects of Diffusion Bonding of High Niobium Containing Gamma TiAl-Based Alloys: *Cesar Justino Buque*[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. of Matls. Rsrch., Max-Planck-Str. 1, Geesthacht 21502 Germany

The paper describes a detailed analysis of diffusion bonding (DB) of a Ti-45Al-10Nb (at.%) representing a new generation of TiAl alloys with enhanced high-temperature capability. The study involves scanning electron microscopy coupled with chemical microanalysis and electron backscattered diffraction. DB of this alloy results in the formation of $\alpha 2$(Ti3Al) grains at the bonding interface surrounded by γ(TiAl) grains. In few cases the formation of β/B2 small particles was also found. Nucleation of the $\alpha 2$ grains occurs at the expense of the $\alpha 2$ lamellae and probably involves migration of antisite defects via antistructural bridges. Remnant deformation structures and the occurrence of a {10-10}-texture of the $\alpha 2$ grains suggest that the recrystallisation processes are driven by localised deformation. These results will be compared with those found by DB of binary alloys with regard to the basic mechanisms involved and discussed with respect to the optimisation of bonding parameters towards improved bond strengths.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C. T. Liu: Intermetallics IV–Bulk Metallic Glasses

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Tuesday PM Room: 8
March 4, 2003 Location: San Diego Convention Center

Session Chairs: Shuji Hanada, Tohoku University, Inst. for Matls. Rsrch., Sendai 980-8577 Japan; Peter K. Liaw, University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA

2:00 PM Invited
Nanoscratch of Metallic Glasses: *T. G. Nieh*[1]; Jessica Lensch[2]; Chris Schuh[3]; [1]Lawrence Livermore National Laboratory, L350, PO Box 808, Livermore, CA 95014 USA; [2]Carnegie Mellon University, Matls. Sci. & Eng., Pittsburgh, PA 15213 USA; [3]Massachusetts Institute of Technology, Matls. Sci. & Eng., 77 Massachusetts Ave., Cambirdge, MA 02139 USA

The tribological and wear behavior of several bulk metallic glasses, including Zr-, Pd-, La-, and Ti-based materials, was investigated. These materials, in both the as-cast and annealed states, were tested using nano-scratch tests, specifically using ramping load scratch techniques. Mechanical properties, such as Youngís modulus, hardness, friction coefficient, and tribological wear were measured. These properties were found to vary with microstructure. An increase in annealing temperature causes an increase in hardness and reduces wear. Samples having a structure consisting of nanocrystals within the amorphous matrix exhibit an improved wear performance. Also, the wear resistance was found to follow the conventional Archardís equation for dry wear, specifically, the volume of material removed by wear is inversely proportional to the hardness of a material. In the present study, special efforts were made to study the wear rate as a function of scratch rate. This work was performed under the auspices of the US Department of Energy (DOE) under contract No. W-7405-Eng-48 with Lawrence Livermore National Laboratory.

2:20 PM Invited
Fatigue Behavior of Bulk Metallic Glasses: *P. K. Liaw*[1]; G. Y. Wang[1]; W. H. Peter[1]; B. Yang[1]; Y. Yokoyama[2]; M. L. Benson[1]; B. A. Green[1]; M. J. Kirkham[1]; S. A. White[1]; T. A. Saleh[1]; R. L. McDaniels[1]; R. V. Steward[1]; R. A. Buchanan[1]; C. T. Liu[3]; C. R. Brooks[1]; [1]The Univer-

sity of Tennessee, Dept. of Matls. Sci. & Eng., TN 37996 USA; [2]Himeji Institute of Technology, Matls. Sci. & Eng., Shosha, Himeji City 2167 Japan; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

High-cycle fatigue (HCF) experiments were conducted on zirconium (Zr)-based bulk metallic glasses (BMGs): Zr-10Al-40Cu, Zr-10Al-30Cu-10Ni, and Zr-10Al-5Ti-17.9Cu-14.6Ni, in atomic percent. The HCF tests were performed using an electrohydraulic machine at a frequency of 10 Hz with a R ratio of 0.1 and under tension-tension loading. Note that R = smin./smax., where smin. and smax. are the applied minimum and maximum stresses, respectively. The test environments were air and vacuum. A high-speed and high-sensitivity thermographic infrared (IR) imaging system was used for nondestructive evaluation of temperature evolution during fatigue testing of BMGs. Limited temperature evolution was observed during fatigue. The effect of chemical composition on the fatigue behavior of the Zr-based BMGs was studied. The fatigue life in vacuum was generally found to be shorter than in air. The dissociation of residual water vapor to atomic hydrogen in vacuum via a hot-tungsten-filament ionization gauge, and subsequent hydrogen embrittlement of BMGs, could have been a factor causing the lower fatigue lifetimes observed in vacuum. The fatigue resistance of Zr-10Al-40Cu and Zr-10Al-30Cu-10Ni seemed not to be as good as that of Zr-10Al-5Ti-17.9Cu-14.6Ni. The fatigue endurance limit of Zr-10Al-5Ti-17.9Cu-14.6Ni was found to be at least comparable to those of structural materials. The cracking shapes on the fatigue surfaces were analyzed and predicted. The present work is supported by the National Science Foundation (NSF) Integrative Graduate Education and Research Training (IGERT) Program with Drs. P. W. Jennings and L. S. Goldberg as contract monitors.

2:40 PM Invited
Toughness of Metallic Glass and Bulk Metallic Glass: *John J. Lewandowski*[1]; Peravudh Lowhaphandu[1]; Paul Wesseling[1]; [1]Case Western Reserve, Dept. Matls. Sci. & Eng., Cleveland, OH 44106 USA

The toughness of metallic glass materials is being measured under a number of different test conditions. The effects of changes in notch root radius on the toughness are being determined in addition to determining the effects of changes in loading rate and test temperature on both amorphous and partially devitrified glasses. The results will be reviewed in the light of ongoing work on such materials. P. Lowhaphandu/JJ Lewandowski, Scripta Metall et Mater., 38,12, 1811-1817, 1998. P. Lowhaphandu, LA Ludrosky, SL Montgomery and JJ Lewandowski, Intermetallics, 8, 487-492, 2000. JJ Lewandowski, Materials Transactions-JIM, 42(4), 633-637, 2001.

3:00 PM
Fracture and Deformation of Bulk Metallic Glasses and their Composites: *Katharine M. Flores*[1]; Reinhold H. Dauskardt[1]; [1]Stanford University, Dept. of Matls. Sci. & Eng., 416 Escondido Mall, Bldg. 550, Stanford, CA 94305-2205 USA

Combining low density, large elastic strains, high strength, and a high fracture toughness, bulk metallic glasses represent an exciting new class of materials with a wide range of potential applications. Plastic deformation of metallic glasses occurs by the formation of shear bands, however, the role of free volume magnitude and distribution, the effects of adiabatic heating and stress state, and the extent of local structural rearrangement are under active investigation. In this work, we investigate the mode II deformation and fracture behavior of a zirconium-based bulk metallic glass. The mode II fracture toughness was found to be ~75 MPa√m, ~4-5 time larger than the mode I toughness. This may be explained by the sensitivity of flow to normal stresses. The fracture and fatigue behavior of a BMG composite utilizing ductile particle reinforcement phases has been examined and compared with that of the monolithic alloy. The second phase blocks the propagation of shear bands and distributes the plastic defo rmation, resulting in extensive stable crack growth at stress intensities double the intrinsic toughness of the unreinforced glass. The composite also exhibits an improved fatigue endurance. Implications for future composite microstructures are discussed.

3:15 PM
Consolidation of Zirconium Based Metallic Glass Powder by Equal Channel Angular Extrusion: *K. T. Hartwig*[1]; I. Karaman[1]; J. Robertson[1]; S. N. Mathaudhu[1]; J.-T. Im[1]; I. E. Anderson[2]; [1]Texas A&M University, Dept. of Mech. Eng., College Station 77843-3123 USA; [2]Iowa State University, Ames Lab., Ames, IA 50011 USA

Vitreloy 106a (Zr58.5Nb2.8Cu15.6Ni12.8Al10.3) gas-atomized powder was consolidated by equal channel angular extrusion (ECAE). Vacuum encapsulated powder was extruded at temperatures above the glass transition temperature (T_g) but below the crystallization temperature (T_x). The oxygen level in the powder is measured to be approximately 1300 ppmw. Microstructure, thermal stability, X-ray diffraction measurements and hardness of the ECAE consolidates were examined and compared with those of the initial powder. The effects of strain rate and consolidation temperature on the consolidation quality and resulting properties were investigated. Compressive experiments were conducted on selected samples at room temperature. Comparison with the initial powder demonstrated that the extrusion rate of 6 mm/sec and the extrusion die temperature of T_g + (25-35 K) results in the best consolidation quality (near full density, near theoretical shear deformation of prior particles and strong in terparticle bonding). No significant crystallization peaks are observed in XRD measurements. The DSC measurements of these consolidates is not significantly different than that of initial powder. Microhardness indentations demonstrate shear localization patterns around indents without crack formation under a 1 kg load. A compressive strength of about 1.6 GPa is observed in the best consolidated samples, which is very close to what is observed in cast material. The fracture surfaces of these compression samples exhibit vein patterns typical of metallic glass fracture. Occasional interparticle debonding in some samples is observed. The results of the present study demonstrate that ECAE consolidation may be a viable method for the fabrication of bulk metallic glass.

3:30 PM Break

3:45 PM Invited
Early Stages of Decomposition of Bulk Metallic Glasses: *Michael K. Miller*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6136 USA

The transformation of bulk metallic glasses from the amorphous state to the fully crystalline state is of interest to evaluate the stability of the glasses. High resolution microstructural characterization of these microstructures provides important information concerning the decomposition processes. Atom probe tomography has been applied to several different bulk metallic glasses to evaluate the microstructural changes that occur between the as-cast state and after annealing in the vicinity of the glass transition temperature. The spatial coordinates and the elemental identities of the atoms in the alloy may be determined with near atomic resolution. The three-dimensional data collected can be analyzed to determine the local atomic configuration around each atom type. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:05 PM Invited
Preparation and Properties of Carbon-Nanotube-Reinforced Zr-Based Bulk Metallic Glass Matrix Composites: *Weihua Wang*[1]; Zan Bian[1]; [1]Chinese Academy of Science, Inst. of Physics, Beijing 100080 China

ZrCuNiAlTi bulk metallic glass composite containing carbon nanotubes (c-tubes) up to 10 vol.% were prepared successfully. The composite consists of c-tubes, ZrC phase and the glass matrix. The mechanical properties, Vickerís hardness and elastic moduli of the composite are markedly improved relative to the undoped bulk metallic glass (BMG). Measurement of acoustic attenuation shows that ultrasonic attenuation coefficient increase drastically after adding c-tubes into BMG, the relative changes in the longitudinal and transverse ultrasonic attenuation (_l and _t) are up to 900% and 536% for BMGC containing 4 vol. % c-tube addition. The results indicate that the composites have excellent acoustic wave absorption ability originating from the structural variation induced by adding c-tubes into the BMG. The excellent acoustic absorption ability may lead to application of BMGs in the field for shielding acoustic sound.

4:25 PM
Structure Property Relations of Simulated Amorphous Ni/Zr Alloys: *Frank J. Cherne*[1]; Michael I. Baskes[1]; Ricardo B. Schwarz[1]; Srivilliputhur G. Srinivasan[1]; [1]Los Alamos National Laboratory, MST-8, Struct. & Prop. Rel., PO Box 1663, MS G755, Los Alamos, NM 87545 USA

Using molecular dynamics and a modified embedded atom method potential developed by our group, we study the structural and elastic properties of amorphous nickel-zirconium alloys as a function of composition and temperature. Previous results indicate that these potentials describe the Ni/Zr system quite well. Liquid alloys were quenched with rates from 2.5 x 10^{11} to 5 x 10^{12} K/s. Using a common neighbor analysis algorithm, we determine the structural units within the liquids and the amorphous solids near the glass transition (T_g). We are interested in X_{Zr} between 12% and 75% where experimentally amorphous materials have been formed. Within this composition range, we observe amorphous material formation. We examine the effects of composition and cooling rates on the local neighbor coordination in the quenched samples.

4:40 PM
In-Situ Study of Decomposition Kinetics in Bulk Metallic Glass $Zr_{52.5}Cu_{17.9}Ni1_{4.6}Al_{10}Ti_5$ by Simultaneous Synchrotron X-Ray Diffraction and Small Angle Scattering: *Xun-Li Wang*[1]; J. D. Almer[2]; Y. D. Wang[1]; J. K. Zhao[1]; D. R. Haeffner[2]; C. T. Liu[1]; [1]Oak Ridge National Laboratory, Spallation Neutron Source & Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA; [2]Argonne National Laboratory, Adv. Photon Source, Argonne, IL 60439 USA

Controlled decomposition of bulk metallic glass precursors offers a promising way for synthesis of nanostructured materials in large quantities. The decomposition of bulk metallic glass involves a multi-stage phase separation of the amorphous and crystalline phases. Despite current research efforts, details of the decomposition kinetics remain sketchy. We report here a simultaneous diffraction and small angle scattering study using high-energy synchrotron X-ray. The diffraction experiment gives the kinetics of the phase transformations, while the small angle scattering experiment reveals the process of phase separation. Our experiment on $Zr_{52.5}Cu_{17.9}Ni1_{4.6}Al_{10}Ti_5$ at T=700 K shows that the crystallization and phase separation initiate simultaneously. The diffraction peak intensity (from the Zr_2Ni phase) increases abruptly at the onset of the phase transformation and quickly saturates. The intensity of small angle scattering, on the other hand, saturates at a slower rate. Analysis of both sets of data suggests that the decomposition in $Zr_{52.5}Cu_{17.9}Ni1_{4.6}Al_{10}Ti_5$ begins with site-saturated nucleation of the crystalline phase, followed by slow growth of the nanosized crystalline precipitates and a redistribution of chemical composition in each phase to reach the thermodynamic equilibrium. Research sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, US Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:55 PM
Low-Temperature Magnetization and Spin-Wave Excitations in Bulk Fe-Based Amorphous Glasses: *Tong D. Shen*[1]; Ricardo B. Schwarz[1]; Joe D. Thompson[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-10, MS K764, Los Alamos, NM 87545 USA

Our flux-purified Fe-Cr-Mo-Ga-P-B-C bulk ferromagnetic glasses have significantly lower AC power losses than rapidly quenched Fe-based glassy ribbons. We have used a SQUID magnetometer to study the temperature dependence of the DC magnetization in bulk Fe-Ni-P-B and Fe-Cr-Mo-Ga-P-B-C glasses. The temperature dependence of the DC magnetization has been extensively studied for glassy ribbons, but not for the newer bulk ferromagnetic glasses. We conclude that: 1) Alloying elements such as Cr, Mo, and Ga decrease the magnetization and Curie temperatures; 2) The low-temperature magnetization-temperature relations for our Fe-based bulk glasses are well described by spin-wave theory; 3) Our bulk metallic glasses have stronger spin-wave stiffness constants than glassy ribbons, indicative of a wider exchange-interaction range. This further suggests that the bulk ferromagnetic glasses have stronger short-range order or are phase separated; 4) Structural relaxation achieved by annealing the bulk glasses below their glass-transition temperatures further enhances the exchange interaction.

5:10 PM
Thermodynamic Prediction of Potential Multicomponent Alloys to Form Metallic Glasses: *K.-C. Hsieh*[1]; D. Ling[2]; Y. Pan[3]; G.-X. Sun[3]; Y. Austin Chang[2]; [1]National Sun Yat-Sen University, Inst. of Matls. Sci. & Eng., Kaohsiung 80424 Taiwan; [2]University of Wisconsin, Dept. of Matls. Sci. & Eng., Madison, WI 53706 USA; [3]Southeast University, Dept. of Mechl. Eng., Nanjing, Jiangsu 210018 China

It is well recognized that the existence of a deep eutectic favors glass formation. One example is soda silicate glass that forms at a eutectic composition of binary Na2O-SiO2. Moreover, we have recently calculated the compositions of liquid alloys at the five-phase invariants of Cu-Ni-Ti-Zr using the CALPHAD methodology (Intermetallics, 2001, 9, p553). These calculated values agree with those measured for bulk metallic glass formation (JAP, 1995, 78, p6514). Based on this success we have been calculating liquid alloy compositions initially for a number of quaternaries such as Al-Cr-Ni-Ti using computer software PANDAT. These calculated values are being used as an intelligent guide to rapidly develop metallic glasses experimentally. In this talk, we will present our preliminary results.

5:25 PM
Preparation and Thermal Stability of Mechanically Alloyed Ni-Zr-Ti-Y Amorphous Powders: *Pee-Yew Lee*[1]; [1]National Taiwan Ocean University, Matls. Eng., 2, Pei-Ning Rd., Keelung 202 Taiwan

Exploring amorphous alloys with a large supercooled liquid region before crystallization is of great importance because the appearance of the large supercooled liquid region is expected to cause the production of a bulk amorphous alloy. The large supercooled liquid region with a temperature interval above 60K had been observed in amorphous Zr-, La, and Mg-based alloys. However, a survey of the available literature indicates that little work has been performed on the formation of Ni-based amorphous alloy with large supercooled liquid region. Ni is a cheaper element as compared with Zr-, and Ti-based amorphous alloys and is expected to have better mechanical properties than that of Zr-based amorphous alloys. In this study, we have investigated the possibility of preparing amorphous Ni-Zr-Ti-Y powders by mechanical alloying of crystalline elemental powder mixtures using a shaker ball mill. The glass-forming ability and thermal stability of mechanically alloyed Ni-Zr-Ti-Y powders were determined by using X-ray diffraction and differential scanning calorimetry (DSC). The results indicated that several amorphous alloy samples were found to exhibit a wide supercooled liquid region before crystallization. This is believed to be the first evidence for the appearance of a supercooled liquid region for mechanically alloyed Ni-Zr-Ti-Y amorphous powders. The finding of the new Ni-Zr-Ti-Y amorphous powders with wide supercooled liquid region is promising the future development of a new Ni-based bulk amorphous alloy through powder metallurgy route.

International Symposium on Structures and Properties of Nanocrystalline Materials: Magnetic and Other Functional Properties

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Tuesday PM — Room: 14B
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: R. Ramesh, University of Maryland, Dept. of Matls. Sci., College Park, MD 20742 USA; Robert D. Shull, NIST, Magnetic Matls., Gaitherburg, MD 20899-8552 USA

2:00 PM
Surface and Interface Magnetism Studied by Depth Selective M^ssbauer Spectroscopy: *Branko Stahl*[1]; [1]Technische Universitat Darmstadt, Matls. & Geo Scis., Thin Films, Petersenstrasse 23, Darmstadt D-64287 Germany

In nanostructured bulk materials the grain boundaries play a crucial role. The experimental characterization of such materials is complicated by the small size of the grains and their complex interaction with neighboring ones. It can be useful for an understanding of the properties of nanostructured materials to choose a simplified geometrical structure that fits to the spatial and time resolution of the experimental method. We have studied the magnetic properties of surfaces of insulating antiferromagnetic single crystals, thin films and nanoparticles to distinguish between pure surface and finite size effects. In two related examples the antiferromagnetic to paramagnetic phase transition could be followed as a function of depth (0-300 nm) and temperature by a depth selective M^ssbauer technique. In the first case (FeBO3), a non-expected magnetic wetting phenomenon is observed. The dynamics of the magnetic moments is changed with respect to the bulk material. In the second case ((Fe,Ga)BO3), the exchange of 24% of the Fe by Ga leads to the expected ordinary phase transition with a gradual decrease of the order parameter towards the surface. These findings support the concept that already the presence of a pure surface is able to alter the magnetic ordering in a certain depth region. The experimental results have parallels in structural phase transitions at surfaces and grain boundaries. Currently experiments are undertaken to study the observed magnetic behavior also in systems with reduced spatial dimensions, i.e. in thin films and nanoparticles. The peculiar properties of nanosized systems is also demonstrated in the case of a metal ferromagnet. 4 nm particles of FePt were characterized by Rutherford backscattering spectroscopy, x-ray diffraction, transmission electron microscopy, x-ray photoelectron spectroscopy, M^ssbauer spectroscopy and magnetometry. The results shed light on the metallic properties of the particles core as well as the chemistry of the particles surface with the organic ligands. Due to the well defined size of the particle-ligand system 3-dimensional superlattices are formed.

2:30 PM

Nano-Structured Alloys with Soft Magnetic Properties: *Ricardo B. Schwarz*[1]; Tong D. Shen[1]; Thomas M. Lillo[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]INEEL, Matls. Div., PO Box 1625, MS 2218, Idaho Falls, ID 83415-2218 USA

Soft ferromagnetism is obtained in both amorphous and nanocrystalline alloys. Amorphous alloys, however, have relatively low saturation magnetization (< 1.2 Tesla) and one dimension less than about 5 mm. The consolidation of alloy powders with nanocrystalline grains may enable the synthesis of bulk materials having both low coercivity and high saturation magnetization. Single-phase Fe-Cu alloy powder with nanosized grains was prepared by mechanical alloying. Annealing the powder at 450K reduces the residual strains without causing significant grain growth. Equal-channel angular extrusion was used to consolidate the powder into rods. The magnetic properties of the consolidated material were measured on small transformers made on toroid-shaped specimens cut from the rods. These toroids had saturation magnetization of 1.5 Tesla and coercivity of 3 Oe. Annealing treatments were used to reduce the internal stresses introduced by the consolidation process and to decrease the coercivity.

3:00 PM

Nanoscaled Magnetostrictive Thin Films for Sensor Applications: *Eckhard Quandt*[1]; Alfred Ludwig[1]; Michael Frommberger[1]; Christel Zanke[1]; Markus Loehndorf[1]; [1]Caesar, Smart Matls., Friedensplatz 16, Bonn D-53111 Germany

Magnetostrictive materials are attractive transducers for applications in MEMS and nanotechnology since they allow simple designs and processing routes which can be scaled down over a wide range of magnitudes without loosing their principle feasibility. While actuators are based on the direct magnetostrictive effect or the □E effect, sensors for mechanical quantities can be realized by combining the inverse magnetostriction with other effects like magnetoimpedance, magnetoresistance or inductivity. In order to meet certain requirements like e.g. the cut-off frequency the thin film materials themselves have to be controlled in terms of their microstructure on a nm-scale. The presentation will discuss the properties of different nanoscaled magnetostrictive materials in view of their possible applications in areas like automotive industry, information technology and biotechnology. Financial support provided by the German Ministry of Education and Research (BMBF 03N3089 and 13N7943) and the Office of Naval Research (N00014-02-1-0231) is gratefully acknowledged.

3:30 PM

Novel Nanostructured Hydride Composites: Hydrogen Storage Materials for Mobile Applications: *R. Bormann*[1]; T. Klassen[1]; W. Oelerich[1]; E. Martinez[1]; [1]GKSS Research Center, Inst. for Matls. Rsrch., Geesthacht D-21502 Germany

A breakthrough in hydrogen storage technology was achieved by preparing nanostructured hydride composites using novel catalysts1. These new composite materials advanced ab- and desorption kinetics, thus qualifying e.g. lightweight Mg-based hydrides for storage application. In this talk, an overview on the sorption behaviour of nanocrystalline Mg and Mg-based composites will be presented, demonstrating their potential as a high-temperature hydride (T=250-300∞C) with a storage capacity up to about 7 wt.% hydrogen. Current research is focussed on alanate-based composites for application temperatures between 100-200∞C. Recent results on the thermodynamics and the sorption behaviour will be presented.

4:00 PM

Ionic Conductivity in Nanometer-Scale Heteroepitaxial YSZ Films: *Christopher M. Rouleau*[1]; Igor P. Kosacki[2]; Paul F. Becher[2]; Douglas H. Lowndes[1]; [1]Oak Ridge National Laboratory, Solid State Div., PO Box 2008, MS 6056, Oak Ridge, TN 37831 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6068, Oak Ridge, TN 37831 USA

Enhanced ionic transport can be achieved in technical oxides, such as zirconia, by generating oxygen vacancies via acceptor doping or by increasing the ionic mobility by accentuating fast diffusion pathways. Recent studies have shown that these methods are effective due to grain boundary and interfacial effects found in materials with nanometer-scale microstructures. These effects dominate in nanocrystalline materials, for example, when the grain size is less than 100nm. Here we examine the influence of film thickness on the ionic conductivity of 10mol% yttria stabilized zirconia thin-films (YSZ) deposited onto MgO by pulsed laser ablation. In this geometry, the fraction of conducting pathways at the surface and heterointerface, versus that within the bulk, varies with film thickness and leads to an overall conductivity having contributions from each. The conductivity was measured in-plane as a function of film thickness and temperature using 2-point impedance spectroscopy and it was found that films having thicknesses >50nm mimicked bulk YSZ while those below 50nm had remarkably higher conductivities. Using a percolation model, it was determined that the surface/heterointerface-related conductivity was two orders of magnitude greater than that of the bulk.

4:30 PM

Nanocrystalline FeCo Powders Produced by Mechanical Milling: *I. Baker*[1]; M. Robson[2]; Ryan G. Quiller[3]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA; [2]The Cooper Union for the Advancement of Science and Art, Cooper Sq., New York, NY 10003-7120 USA; [3]Rensselaer Polytechnic Institute, Dept. of Math., 110 8th St., Troy, NY 12180 USA

Fine powders of Fe and Co were mechanically-milled for 60 hours using stainless steel balls in a Svegari attritor operated under an argon atmosphere. The effects of various elemental additions, including B, C Cu, Zr and Ni, on the microstructure of approximately stoichiometric Fe:Co powders were studied. The morphologies and sizes of the powders were examined using a scanning electron microscope. The grain sizes and phases present before and after annealing the milled powders at 873 K were examined using X-ray diffractometry. Phase transformations in the milled powders were examined using constant heating in a differential scanning calorimeter. The resulting magnetic properties were measured using a vibrating sample magnetometer. Research sponsored by NSF grants DMR-9820408, DMR-9973977 and DMR-0139085.

4:50 PM

Sol-Gel Derived Nanocrystalline Tin Oxide Based Hydrogen Gas Sensor: *Satyajit Vishnu Shukla*[1]; Sudipta Seal[1]; Suresh Kuiry[1]; Swanand Dilip Patil[1]; Lawrence Ludwig[2]; Clyde Parish[2]; [1]University of Central Florida, Dept. of Mechl. Matls. Aeros. Eng., 4000 Central Florida Blvd., Eng # 381, Orlando, FL 32826 USA; [2]Kennedy Space Center (NASA) and University of Central Florida, Orlando, FL 32826 USA

Nanocrystalline tin oxide (SnO2) based hydrogen (H2) gas sensor, in the form of thin film, operating at room temperature is synthesized using sol-gel technique utilizing alkoxide and non-alkoxide precursors. The effect of sol-gel synthesis parameters such as solution as well as polymer concentrations and firing temperature on various sensing parameters such as sensitivity, response and recovery time are systematically investigated. The use of hydroxypropyl cellulose (HPC) polymer is shown to control the nanocrystallite size and the amount of porosity within the thin film. The effects of surface and bulk modifications of SnO2 obtained via sputtering and doping techniques respectively are demonstrated as useful techniques to achieve maximum H2 gas sensitivity at room temperature. The sensor is characterized using various analytical techniques such as XPS, SEM, and HRTEM to optimize its performance.

5:10 PM

Magnetic Nanomaterials for Defense and Biotechnology Applications: *R. V. Ramanujan*[1]; H. F. Li[1]; S. W. Du[1]; [1]Nanyang Technological University, Sch. of Matls. Eng., Nanyang Ave. 639798 Singapore

Novel soft magnetic nanomaterials have been shown to exhibit excellent properties, ongoing work on developing such iron and cobalt based alloys will be presented. The Herzer model has shown that in suitable alloy systems, nanosized precipitates embedded in an amorphous matrix can yield superior magnetic properties. Such nanocrystalline magnetic materials can be produced crystallization, through heat treatment, of amorphous precursors, however the optimum alloy composition and heat treatment conditions have to be experimentally determined. Therefore, melt spun iron based alloys with the composition (in wt. %) Fe40-Ni38-B18-Mo4 and Fe67-Ni18-B14-Si1 as well as cobalt based alloys of composition Co69-Si15 -B14-Fe4-Ni1 and Co81-Si7-Mo4-B3-Ni1 have been studied by a variety of experimental techniques such as TEM, EDX, XRD, VSM, resistivity and DSC. Heat treatment of these alloys showed a variety of interesting crystallization reactions. The DSC results provided the primary and secondary crystallization temperatures and the activation energy of crystallization were extracted by the Kissinger analysis. Resistivity measurements identified the time for crystallization while VSM provided the magnetic properties as a function of heat treatment conditions. TEM and EDX were used to identify the morphology, distribution, structure and composition of the crystalline phase. Based on the above results, selected alloys, heat treated at suitable temperatures and times, were evaluated for use in high temperature motors used in defense and for drug delivery in biotechnology applications.

5:30 PM
Magnetic Structure and Hysteresis in Hard Magnetic Nanocrystalline Film: A Computer Simulation Study: *Yongmei M. Jin*[1]; Yu U. Wang[1]; Armen G. Khachaturyan[1]; Andrei Kazaryan[2]; Yunzhi Wang[2]; David E. Laughlin[3]; [1]Rutgers University, Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA; [2]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; [3]Carnegie Mellon University, Matls. Sci. & Eng., Pittsburgh, PA 15213 USA

Three-dimensional micromagnetic simulations are used to study the effect of textures on the magnetic properties of uniaxial nanocrystalline films of hard magnetic materials with arbitrary grain shapes and size distributions. The correlation lengths of these hard magnetic materials, domain wall width and ferromagnetic exchange length, are assumed to be smaller than the typical grain size. The Landau-Lifshitz equations of the magnetization dynamics are employed to describe the distribution of magnetization in ferromagnetic domains, domain evolution during magnetization switching, and the hysteresis curve. The equations are solved numerically in reciprocal space using the Fast Fourier Transform technique. Simulations are performed for films of different grain textures. The results show that the magnetic coupling between grains in thin film significantly affects the morphology of magnetic domains and their response to the applied magnetic field. The greater the deviation of grain uniaxial directions from the film normal, the smaller the coercivity and the remanence magnetization. It is also shown that the remanence magnetization and coercivity respond differently to the variations in film texture and that the magnetic reversal process is a collective process involving groups of grains. In particular, it is found that the grain texture has a more complicated effect on the coercivity than on the remanence magnetization and on the character of topological changes during magnetization reversal process.

Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging: Microstructural Characterization and Evolution

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Srini Chada, Motorola, Department APTC, Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Jud Ready, MicroCoating Technologies, Atlanta, GA 30341 USA; Jin Yu, KAIST, Center for Electronic Packaging Materials 305-701 Korea

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Session Chairs: Sung K. Kang, IBM, T. J. Watson Rsrch. Ctr., Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Dept. of Cheml. Eng., Chungli City 32054 Taiwan

2:00 PM Invited
Damage Accumulation and Failure of Sn-Based Electronic Solder Joints Under Thermomechanical Fatigue: J. G. Lee[1]; *K. N. Subramanian*[1]; T. R. Bieler[1]; [1]Michigan State University, Dept. Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

The residual mechanical properties of thermomechanically fatigued (TMF) Sn-based solder joints initially deteriorate rapidly and tend to stabilize after the first few hundred cycles, in spite of additional surface damage accumulation. Such findings suggest that crack growth, rather than crack nucleation, is the controlling factor in TMF failures. Since several processes, such as creep/stress relaxation, aging, reversed shear, Sn-anisotropy, etc., contribute toward TMF failure, solder joints possessing similar geometry were subjected to these processes. These solder joints were examined by laser confocal microscopy, SEM, Orientation Imaging, and continuous documentation of microscopic damage accumulation. The relative importance of each of these contributing factors to TMF will be discussed in order to form a comprehensive picture consistent with the observed damage accumulation and mechanical behavior. Acknowledgement: Project funded by National Science Foundation under grant No. NSF-DMR-0081796.

2:25 PM
Evolution of Microstructure and Damage in Tin-Lead Solder Under Fast Cyclic Loading: *Yu-Lin Shen*[1]; Koel C.R. Abell[1]; Stephen E. Garrett[1]; [1]University of New Mexico, Dept. of Mechl. Eng., Albuquerque, NM 87131 USA

Accelerated testing of thermomechanical fatigue of solder joints typically employed a cyclic frequency on the order of 0.001 Hz or lower. At such a straining rate the mechanical and metallurgical (diffusion induced coarsening) effects both contribute to damage initiation and eventual failure. In the present study we focus on fast loading of eutectic tin-lead with frequencies of 1 Hz and higher. The primary purposes of conducting fast cyclic tests are (1) to keep the diffusional contribution at minimum so pure mechanical effects can be examined, and (2) to study solder reliability in microelectronics for space and military applications where severe stress environments such as mechanical shock and vibration prevail. As a baseline bulk specimens were first subject to relatively fast deformations of tension, compression and bending. Grain realignment and phase redistribution were observed and characterized by microscopy and microhardness indentation. A micromechanical model is proposed to elucidate the observed microstructural changes and progressive damage. The significance of damage evolution in the form of microscopic heterogeneity and mechanically induced phase coarsening caused by grain boundary sliding is illustrated. High-frequency twin-lap shear tests on solder joints were then performed. Phase coarsening and the associated fracture initiation were observed after only a short time, suggesting that long-term atomic diffusion is not a necessary factor in initiating coarsening and fatigue failure. Evidences of grain boundary sliding in the cyclically sheared small joints were also found. Progressive damage from microvoiding, coalescence of microvoids to form microcracks, to the development of a major crack during fast cyclic shearing will be presented and discussed.

2:45 PM
Microstructure and Thermal Behavior of Sn-Zn-Ag Solders: *Kwang-Lung Lin*[1]; Chia-Ling Shi[1]; [1]National Cheng Kung University, Matls. Sci. & Eng., 1 Ta-Hsuey Rd., Tainan 701 Taiwan

The microstructure and thermal behavior of Sn-Zn-Ag solder were investigated for 8.73~9%Zn and 0~3.0%Ag. SEM analysis shows Ag-Zn compound when the solder contains 0.1%Ag. XRD analysis results indicate that Ag5Zn8 and AgZn3 become prominent when the Ag content is 0.3% and above. Meanwhile, the Zn-rich phase is refined and the Zn orientations gradually diminish upon increase in Ag content. The morphology of the Ag-Zn compound varies from nodular to dendrite structure when the Ag content increases. The growth of the Ag-Zn compounds is accompanied with diminishing of the eutectic structure of the Sn-9Zn solder. DSC investigation reveals that the solidus temperature of these solders exists at around 198°C. A single sharp exothermic peak was found for the solders with Ag content less than 0.5%. Liquidus temperatures were identified with the DSC analysis to vary from 206°C to 215°C when the Ag content ranges from 1.0% to 3.0%.

3:05 PM
Interface Microstructure Between 42 Alloy and Sn-Based Lead-Free Solder: *Chi Won Hwang*[1]; Jung Goo Lee[2]; Hirotaro Mori[2]; Katsuaki Suganuma[1]; [1]Osaka University, ISIR, Mihogaoka8-1, Ibaraki, Osaka 567-0047 Japan; [2]Osaka University, UHVEM, Mihogaoka 7-1, Ibaraki, Osaka 567-0047 Japan

42 alloy (Fe-42wt%Ni) has been widely used as one of the useful electrode materials in electronic devices. The interfacial characteristics between the Sn-base solder and 42 alloy, however, have not been fully understood yet. To examine interface microstructure, three types of lead-free solder, Sn, Sn-3.5Ag, and Sn-3Ag-0.5Cu, were used as lead-free solders. After joining, cross-sectioned samples against soldered layer were prepared and investigated by TEM, SEM, and EPMA. As an interfacial reaction layer between solder and 42 alloy, FeSn2 phase was developed in a thickness of about 100 nm. Ni from 42 alloy dissolved in the molten solders and it formed Ni3Sn4 phase with Sn. Microstructure of interface was also evaluated in detail.

3:25 PM
Characterization and Microstructure Evolution of Thermomechanically Fatigued Dual Single Shear Lap Sn-Ag Solder Using Orientation Imaging Microscopy: *A. U. Telang*[1]; T. R. Bieler[1]; J. G. Lee[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

Single shear lap specimens having a solder joint area of 1 mm^2 and thickness of 80-160 μm on copper and nickel substrates were

TUESDAY PM

thermomechanically (TMF) cycled from -15 to 150°C in order to mimic solder joint deformation history of surface mount components. SEM studies reveal that shear bands, surface relief effects and other surface morphological features develop even after tens of TMF cycles. Orientation imaging (OIM) studies of various regions of specimens that underwent 0, 20, 100, 500, and 1000 TMF cycles were carried out to determine how the microstructure and crystallographic orientations evolve during TMF. These results were compared to those of unconstrained single shear lap specimens to ascertain the substantial role of reversed shear deformation and the anisotropy of Sn that lead to failure of the solder joints in surface mount components. Supported by National Science Foundation under grant NSF DMR-0081796.

3:45 PM Break

3:55 PM Invited
A Strategy for Producing Adaptive Lead-Free Solder Joints Via Shape-Memory Alloy Reinforcement of Solder Alloys: *Indranath Dutta*[1]; Bhaskar S. Majumdar[2]; Sunglak Choi[1]; William Wright[1]; [1]Naval Postgratuate School, Dept. of Mechl. Eng., 700 Dyer Rd., Code ME/DU, Monterey, CA 93943 USA; [2]New Mexico Tech, Dept. of Matls. Sci. & Eng., Socorro, NM 87801 USA

Microelectronic solder joints are typically exposed to aggressive thermo-mechanical cycling (TMC) conditions during service. During TMC, strain localization occurs near solder/bond pad interfaces, where large inelastic shear strains accumulate, eventually causing low-cycle fatigue failure of the joint. In this paper, a new methodology to mitigate the effects of strain localization within the joint are discussed, wherein the solder alloy is reinforced with Ni-Ti based shape memory alloy (SMA) whiskers with a martensite-to-austenite (MÆA) transformation temperature of 80-100∞C are used to reinforce a Sn-4.7%Ag-1%Cu solder. In this scheme, the SMA reinforcement undergoes MÆA transformation near the high temperature end of the TMC, placing the solder matrix next to the solder/bond pad interfaces in reverse shear, enabling strain redistribution and homogenization, and thereby enhancing joint life. The conceptual mechanics of this adaptive scheme will be presented, along with issues relating to the fabrication of SMA reinforced lead-free solders. Some experimental results delineating the role of SMA reinforcement on the overall joint behavior will also be presented. Supported by the Army Research Office. The assistance of Dr. Treliant Fang of Form Factor, Inc. is gratefully acknowledged.

4:20 PM
Effect of Cooling Rate on the Microstructure of Ag-Cu-Sn: Leonid Snugovsky[1]; Polina Snugovsky[2]; *Doug D. Perovic*[1]; John Rutter[1]; [1]University of Toronto, Dept. of Matls. Sci. & Eng., Wallberg Bldg., 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]Celestica, Matls. Lab., STC, 844 Don Mills Rd., Toronto, Ontario M3C 1V7 Canada

Samples of four Pb-free compositions in the Ag-Cu-Sn system were cooled at various rates and examined metallographically to evaluate microstructural effects. For Ag and Cu contents less than that of the ternary eutectic composition, faster cooling rates produced greater amounts of primary tin dendrites with interdendritic eutectic containing as much as 15 vol.% of compound phases. For Ag and Cu contents greater than that of the ternary eutectic composition, smaller amounts of primary Sn dendrites and increasing amounts of ternary eutectic microstructure were observed. In some samples, binary Ag3Sn-Sn eutectic was also observed. It was found that the Sn phase contained up to 1 wt. % Ag in solution, in the absence of Cu. It is believed there occurs an effective kinetic shift in the eutectic point resulting from the greater undercooling required to drive freezing of the faceting compound phases at the same speed as the nonfaceting Sn phase.

4:40 PM Invited
Microstructure Characterization in the Sn-Ag Solder Joints Between Stud Bumps and Metal Pads: Mu-Seob Shin[1]; *Young-Ho Kim*[1]; Won-Chul Do[2]; Sun-Ho Ha[2]; [1]Hanyang University, Dept. of Matls. Eng., 17 Haengdang-dong, Seongdong-gu, Seoul 133-791 Korea; [2]Amkor Technology Korea, R&D Ctr., Seongsu-dong 2-ga, Seongdong-gu, Seoul 133-120 Korea

The microstructure of the flip chip solder joints fabricated using stud bumps and Pb-free solders were characterized as functions of reflow cycles and aging time. The Au or Cu stud bumps formed on Al pads in Si dies were aligned to corresponding metal pads in the substrates, which had been printed with Sn-3.5Ag pastes. The solder joints were made using reflow. In the solder joints fabricated using Au stud bumps, Au-Sn intermetallics spread over in the whole joints and the solder remained randomly as an island shape due to the fast dissolution of Au. The microstructure of the solder joints did not changed significantly even after multiple reflows. AuSn4 was the main phase after reflow, but AuSn4 and AuSn2 were converted to AuSn during aging. In the solder joints fabricated using Cu stud bumps, the Cu6Sn5 was formed only in the Cu interface and the solder was the main phase.

5:00 PM
The Effects of Cooling Rate and Aging Temperature on Microstructure Evolution in Sn-Ag/Cu and Sn-Ag-Cu/Cu Joints: *George Piotrowski*[1]; Jason J. Williams[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA

The morphology, thickness and evolution of the intermetallic compounds at the solder/metallization interface has a significant effect on the mechanical behavior of the overall solder joint. Thus, it is important to understand the roles that cooling rate and thermal aging have on the growth of the intermetallic compounds in the solder joints. Sn-3.5Ag and Sn-3.5Ag-0.7Cu solder/Cu ìsandwichî joints were reflowed and cooled in one of three different media (water, air, furnace) to obtain a controlled cooling rate. After cooling to room temperature, the solder joints were sectioned and aged at 100°C, 140°C, or 175°C for times ranging from 0h to 1000h. The thickness and morphology of the two intermetallic compounds present at the solder/metallization interface, Cu_6Sn_5 and Cu_3Sn, were examined. It will be shown that the cooling rate has a significant effect on the initial morphology and growth rate of the intermetallic compounds. It will also be shown that the rate of the total intermetallic growth, as well as Cu_6Sn_5 and Cu_3Sn, can be explained in terms of bulk and grain boundary diffusion. Research supported by the National Science Foundation (Program Manager: Dr. K.L. Murty, contract# DMR-0092530).

5:20 PM
A Case Study of Sn Whisker Growth Phenomenon: *Ker-Chang Hsieh*[1]; Shin-Jen Jack Wong[1]; Elton Yu[2]; C. C. Chen[2]; [1]National Sun Yat-Sen University, Inst. of Matls. & Eng., Kaohsiung 80424 Taiwan; [2]Philips Electronic Building Elements Industries (Taiwan), Ltd., Tech. Dev. Div., 10, Chin 5th Rd., N.E.P.Z., PO Box 35-48, Kaohsiung Taiwan

It is well understood that the root cause for whisker formation is the internal stress. The process factors such as plating current density, Sn layer thickness and intermetallic growth morphology could contribute to internal stress. Internal stress measured by XRD decreased as Sn whisker growth in a series of samples under low temperature aging storage. The local structures of Sn grain size, intermetallic and whisker root were examined by using focused ion beam (FIB). The preliminary results of this study are: (I) Plating current density will affect whisker formation. (II) Process flow especially thermal treatment step, will influence the whisker growth. (III) Moisture and electric current will affect whisker formation.

Magnesium Technology 2003: Magnesium Casting Properties and Welding

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, LMD-Magnesium Committee, International Magnesium Association, MPMD-Solidification Committee
Program Organizers: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

Tuesday PM — Room: 2
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Alan Luo, General Motors, Matls. & Processes Lab., Warren, MI 48090-9055 USA; G. Cohen, Nuclear Research Center Negev, Beer Sheva 84190 Israel

2:00 PM
Electrical and Thermal Conductivity Measurements on Commercial Magnesium Alloys: *Sayavur I. Bakhtiyarov*[1]; Ruel A. Overfelt[1]; Sorin G. Teodorescu[1]; [1]Auburn University, Mechl. Eng., 202 Ross Hall, Auburn, AL 36849-5341 USA

The worldwide production of magnesium increased from 10 tonnes per annum in 1900 to 350,000 tonnes per annum during the 1990s. The need for magnesium alloys has increased rapidly because of their light weight (r=1.738 g◊cm-3 at 200C) and rigidity. Poor corrosion resistance of these alloys triggered the development of new corrosion resistant alloys with improved tensile strength and augmented stiffness. The amount of each alloying constituent (up to the solid solubility limit) strongly affects the physical and mechanical properties of magnesium. The distinctive physico-chemical properties of magnesium and its alloys require special techniques to accurately measure their thermophysical properties. A rotational inductive measurement technique has been modified and used to determine the electrical resistivity of magnesium alloy AE42 over a wide range of temperature (including the molten alloy). In addition, the thermal conductivity of the alloy was estimated from these data using the Wiedemann-Franz-Lorenz law.

2:25 PM

Modelling of the Thermo-Physical and Physical Properties for Solidification of Mg-Alloys: *Nigel Saunders*[1]; Xiuqing Li[2]; Alfred Peter Miodownik[1]; Jean-Philippe Schille[2]; [1]Thermotech, Ltd., Surrey Tech. Ctr., The Surrey Rsrch. Park, Guildford, Surrey GU2 7YG UK; [2]Sente Software, Ltd., Surrey Tech. Ctr., The Surrey Rsrch. Park, Guildford, Surrey GU2 7YG UK

The thermo-physical and physical properties of the liquid and solid phases are critical components in casting simulations. Such properties include the fraction solid transformed, enthalpy release, thermal conductivity, volume and density all as a function of temperature. Due to the difficulty in experimentally determining such properties at solidification temperatures, little information exists for multi-component alloys. As part of the development of a new computer programme for modelling of materials properties (JMatPro) extensive work has been carried out on the development of sound, physically based models for these properties. Extensive results will presented for Mg-based alloys.

2:50 PM

Developments in Lost Foam Casting of Magnesium: *Michael Marlatt*[1]; David Weiss[2]; John N. Hryn[3]; [1]wfv/Roeperwerk, Plymouth, WI USA; [2]Eck Industries, Inc., Manitowoc, WI 54221-0967 USA; [3]Argonne National Laboratory, Argonne, IL 60439-4815 USA

Preliminary work was conducted in the casting of magnesium using the lost foam casting process. The lost foam or expendable pattern casting (EPC) process is capable of making extremely complicated part shapes at acceptable soundness levels and with low manufacturing costs. Standard test shapes were used to determine the ability of the magnesium to fill the mold and to assess the types of defects encountered. This paper will briefly explain how this project evolved including the developmental strategies formed, the products selected, the casting trials performed, and the casting results.

3:15 PM Break

3:30 PM Invited

Friction Stir Welding of Magnesium Alloys: Richard Johnson[1]; *Philip L. Threadgill*[1]; [1]TWI, Ltd., Friction & Forge Processes Tech. Grp., Granta Park, Great Abington, Cambridge, Cambridgeshire CB1 6AL UK

Most magnesium alloys are relatively easy to weld, but avoiding some porosity in fusion welds is always difficult. This can be a particular problem in pressure die cast components, where small gas bubbles may be entrapped under very high pressure. This leads to a process instability during fusion welding, and poor quality welds. Solid state processes, in particular friction welding, can circumvent this problem, as no fusion occurs in normal circumstances. Furthermore, the parts being welded are also at a high pressure, limiting the ability of the trapped gas to expand. Of particular interest is the friction stir welding process, first developed at TWI in 1991, which has now been demonstrated as a powerful technique for welding magnesium alloys, in both cast and wrought forms. The process can also be used to join dissimilar magnesium alloys, and significant progress has also been made on joining aluminium alloys to magnesium alloys, a combination of particular interest to the automotive sector. This paper will summarise recent studies at TWI and elsewhere on friction welding of magnesium, with special emphasis on friction stir welding. The advantages and disadvantages of these approaches will be highlighted. The paper includes contributions on process data, microstructures and mechanical properties.

3:55 PM Invited

Laser Welding of Magnesium Alloys: *Kenneth Gordon Watkins*[1]; [1]University of Liverpool, Laser Grp., Dept. of Eng., Brownlow St., Liverpool L69 3GH UK

There is increasing current interest in the use of magnesium alloys in the automobile industry since the low density (and hence high specific strength) of these alloys may play a part in meeting directives aimed at reducing overall vehicle mass with subsequent reduction in the emission of greenhouse gases such as carbon dioxide. However, if the usefulness of these alloys is to be realised in applications of this type, successful joining methods are required. In the achievement of weight reduction based on the use of steel and aluminium alloys, it has been found that a tailored blank strategy based on laser welding has been very successful. This points to the necessity of increasing effort in the adaptation of laser beam welding techniques for the case of magnesium alloys. This paper reviews the joining of magnesium alloys by laser welding. It considers the influence of laser beam process parameters (laser power, welding speed, assist gas type and flow rate) on the weldability of these alloys, concentrating on factors such as elimination of porosity and the achievement of weld ductility and strength. Where appropriate, these factors are related to the metallurgy of the alloys concerned. The joining by laser welding of magnesium alloys to dissimilar alloys (particularly aluminium alloys) is also considered.

4:20 PM

Welding and Weldability of AZ31B by Gas Tungsten Arc and Laser Beam Welding Processes: *Sri Lathabai*[1]; Ken Barton[1]; Damien Harris[1]; Peter Lloyd[1]; Alasdair McLean[1]; [1]CSIRO, Mfg. & Infrastruct. Tech., PO Box 4, Woodville, S. Australia 5086 Australia

Welding will play an important role in the fabrication of modular lightweight structures based on magnesium alloy die castings, extrusion profiles and wrought products. Minimisation of rejection rates during fabrication requires that satisfactory weldability be established for a particular combination of materials and welding procedures. In this paper, we present the results of a study to quantify the weldability of wrought alloy AZ31B by gas tungsten arc (GTA) and laser beam (LB) welding processes. The susceptibility to weld metal solidification cracking was evaluated using the Varestraint and Circular Patch weldability tests. Operating windows of welding parameters for crack-free and porosity-free GTA and LB welding were identified, based on which welding procedures were developed for sheet and plate AZ31B. The microstructure and mechanical properties of welded test plates were assessed, leading to a better understanding of microstructural development and structure-property relationships in GTA and LB weldments in AZ31B.

4:45 PM

Application of Welding Technologies for Joining of Mg Alloys: Microstructure and Mechanical Properties: *Adin Stern*[1]; Abraham Munitz[2]; Gabrial Kohn[3]; [1]Ben Gurion University, Dept. of Matls. Eng., Beer-Sheva 84105 Israel; [2]Nuclear Research Center Negev, PO Box 9001, Beer Sheva 84190 Israel; [3]Rotem Industries, Rotem Industl. Park, PO Box 9046, Beer-Sheva 84190 Israel

Five welding technologies were investigated as a solution to the challenges of joining Mg alloys: Gas Tungsten Arc Welding, Electron Beam Welding, Magnetic Pulse Welds, Laser Assisted Friction Stir Welding and Resistance Spot Welding. The main influencing parameters have been analyzed using the joint strength and microstructure to evaluate the welding processes. The advantages and difficulties for each technique are described and discussed. The microstructural changes that occurred during welding were examined by optical microscopy and under the scanning electron microscope. Backscattered electron imaging giving pronounced compositional contrast was used, in combination with energy dispersive and wavelength dispersive spectroscopy microanalysis. Mechanical properties were determined by standard tensile tests on small-scale specimens and microhardness. The experimental results obtained in this study show that defect free welds could be obtained in magnesium alloys using the aforementioned welding techniques. These techniques hold good promises for industrial applications where reliable high quality joining methods are required. The present paper describes a part of a broader research program aimed at gaining a better understanding of the relationship between microstructure, properties and the welding technique, in Mg alloys welded joints.

Magnesium Solidification, Casting and Welding –Jt. Session with Magnesium Technology 2003: Magnesium Casting and Solidification and Simulations

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, LMD-Magnesium Committee, International Magnesium Association, MPMD-Solidification Committee

Program Organizers: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

Tuesday PM — Room: 2
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Gerald S. Cole, Consultant, USA; David Henry StJohn, University of Queensland, CAST Div. of Matls. Eng., Brisbane, QLD 4072 Australia

See Tuesday AM Magnesium Technology 2003: Magnesium Casting and Solidification and Simulations symposium for abstracts.

Materials Lifetime Science and Engineering - II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; D. Gary Harlow, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015-3085 USA; Dwaine L. Klarstrom, Haynes International, Inc., Kokomo, IN 46904-9013 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Robert P. Wei, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015 USA

Tuesday PM — Room: 18
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Raymond A. Buchanan, University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; Robert P. Wei, Lehigh University, Mechl. Eng. & Mech., Bethlehem, PA 18015 USA

2:00 PM Invited
Adhesion and Reliability in Thin-Film Microelectronic Device Structures: *Reinhold H. Dauskardt*[1]; [1]Stanford University, Dept. of Matls. Sci. & Eng., Stanford, CA 94305-2205 USA

The mechanical behavior and resistance to delamination profoundly influences the mechanical integrity of a wide range of thin-film device structures, particularly during chemical mechanical planarization (CMP) operations where fragile low-k brittle dielectrics are exposed to aqueous environments and additional mechanical loads. Such behavior is considered in a range of representative thin-film structures containing glass, polymer and metal layers frequently encountered in interconnect structures. Three classes of low-k dielectrics are considered: modified silica containing organic, inorganic/organic polymer hybrid, and organics involving aromatic hydrocarbon polymers which are shown to behave in a ductile manner. Interface fracture studies are described to characterize both critical adhesion values and subcritical debonding behavior under selected loading and environmental conditions typically encountered during CMP. Multi-scale models are developed to explain the effect of a range of salient interface proper ties on delamination and fracture behavior. These include interface morphology and chemistry, ductile Cu or dielectric layer thickness, elastic properties and thickness of the barrier layer, environmental species in CMP environments, and loading mode. Finally, the effect of more complex patterned thin-film structures are examined.

2:30 PM Invited
Fracture, Fatigue and Structural Reliability of Bulk Metallic Glass Alloys: Hydrogen and Environmental Effects: *Reinhold H. Dauskardt*[1]; [1]Stanford University, Dept. of Matls. Sci. & Eng., Stanford, CA 94305-2205 USA

The recent development of bulk metallic glass alloys offers the potential for a versatile new class of structural metallic materials with a range of superior mechanical and corrosion resistance properties. Availability of these materials in bulk form has also greatly facilitated the investigation of fundamental deformation, fracture and fatigue behavior compared to previous studies that were limited to thin metallic ribbons. This presentation will review the underlying deformation and fracture behavior in Zr-based bulk metallic glasses and their environmental sensitivity. Specifically, hydrogen and moisture are well-known embrittling species in a wide range of crystalline metals and their effects on the underlying kinetic flow processes and resulting fracture and fatigue behavior will be examined. Microstructure and thermal behavior of the metallic glass were examined using positron annihilation, high-resolution electron microscopy, X-ray diffraction, X-ray photoelectron spectroscopy and differential scanning calorimetry. The microstructural origin of selected environmental interactions in the amorphous microstructure will be identified. It will be demonstrated that hydrogen retards the fundamental molecular rearrangement process responsible for plastic flow in the amorphous metals. The sluggish molecular rearrangement process is believed to be responsible for increased flow stress and degraded toughness and fatigue behavior. The implications for reliability and structural integrity of bulk metallic glasses will be discussed.

3:00 PM
Determining Worst-Case Fatigue Thresholds for Grain Bridging Ceramics: *Jamie J. Kruzic*[1]; Rowland M. Cannon[1]; Robert O. Ritchie[1]; [1]Lawrence Berkeley National Laboratory, Matls. Scis. Div., 1 Cyclotron Rd., MS 62-203, Berkeley, CA 94720 USA

While many ceramics achieve increased toughness via grain bridging, they also suffer from cyclic fatigue failure due to degradation of the bridging zone and additionally should be susceptible to crack size effects when crack sizes become smaller than the bridging zone length. In order to investigate such crack size effects, a coarse grained (~18 micron) alumina was chosen as a model material. Crack-growth properties for both long (> 2 mm) and short (< 2 mm) cracks emanating from notches were investigated, with observed short crack growth rates exceeding those for corresponding long cracks at the same applied stress intensity range. Such results are rationalized by considering the reduced bridging zone for short relative to long fatigue cracks; indeed, after quantifying the effects of grain bridging and comparing the results in terms of an effective, bridging removed, stress intensity, a worst-case fatigue threshold is determined below which neither long nor short cracks appear to propagate. Since for structural applications crack sizes must remain small in ceramics components due to their inherent low toughness, such methodologies for predicting worst-case fatigue thresholds will be paramount for enabling fatigue lifetime predictions in grain bridging ceramics.

3:20 PM
Localized Deformation Around Nanoindentations and the Effects of Hydrogen on Dislocation Cross-Slip: *Kevin Andrew Nibur*[1]; [1]Washington State University, MSE, Pullman, WA 99164 USA

The ability to understand mechanical deformation on the nanometer to micrometer scale is important for developing accurate dislocation models. Atomic force microscopy (AFM) and orientation imaging microscopy (OIM) have been used to study dislocation cross-slip behavior around nanoindentations. Slip steps occurring in the pile-up region around an indentation have been characterized to determine the active slip systems resulting in each step. The frequency and magnitude of each step can be determined. These experimental results can be useful for dislocation dynamics models, since indentations can be selectively placed to either include or exclude interactions with grain boundaries, particles or other defects. This technique has been used to study hydrogen embrittlement in FCC and BCC materials to study the effect of hydrogen on dislocation cross-slip. Results indicate that hydrogen does not greatly affect the frequency of cross-slip, but does decrease the number of dislocations in a given slip step.

3:40 PM Invited
Rotating-Beam Fatigue Properties and Effect of Additional Ni Element in Bulk Amorphous Zr50Cu40Al10 Alloys: *Yoshihiko Yokoyama*[1]; Kenzo Fukaura[1]; Akihisa Inoue[2]; [1]Himeji Institute of Technology, Matls. Sci. & Eng., Shosha 2167, Himeji, Hyogo Pref. 671-2201 Japan; [2]Tohoku University, Inst. for Matls. Rsrch., Katahira 2-1-1, Aoba, Sendai, Miyagi Pref. 980-8577 Japan

In order to evaluate the effect of Ni element on fatigue strength, W^hler curves of Zr50Cu40Al10 and Zr50Cu30Ni10Al10 bulk glassy alloys were examined with rotating-beam fatigue test. We also observed fatigue feature surfaces of Zr50Cu40Al10 and Zr50Cu30Ni10Al10 bulk glassy alloys to estimate the effect of adding Ni element, which can improve the oxygen embrittlement in Zr-based bulk amorphous alloy. As the result, fatigue limit is increased from 250 MPa to 500 MPa by adding 10 at% Ni instead of Cu. Furthermore, by adding Ni element, fatigue-fractured region is limited inside of semi-circle region on the fractured surface. Besides, Zr50Cu40Al10 bulk glassy alloy shows wide fatigue-fractured region with striation like mark up to 90% under the low applied stress (close to fatigue limit) condition. Consequently, the prevent oxidization effect of Ni element is promotes fatigue limit in Zr50Cu40Al10 bulk glassy alloys.

4:10 PM Invited

Mechanisms for Fatigue Crack Initiation and Early Propagation: *Tongguang Zhai*[1]; [1]University of Kentucky, Dept. of Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY USA

In this paper, a model for fatigue crack initiation has been proposed, based on the experimental results that there is always a net irreversible slip in one direction of the active slip in a metallic material. Such irreversible slip leads to a local stress which gradually breaks the material in the surface with the aid of environmental effects. The model appears to be able to explain the observed fatigue phenomena in Al, Cu, Cu-Al alloy and Mg single crystals. The paper has also discussed a crystallographic model for short crack growth across grain boundaries in a metallic material, especially those with strong tendency for planar slip. The main factor that controls the growth behavior of a short crack is the twist angle of crack deflection at the grain boundary plane. The short crack always chooses the path that forms the minimum twist angle in a new grain with the crack plane in the previous grain. These proposed models for crack initiation and early propagation could lead to more precise life prediction of a metallic material.

4:40 PM Invited

Analysis of Plastic Deformation Behavior During Equal Channel Angular Pressing: *Yong Liu*[1]; Zhihong Tang[1]; Baiyun Huang[1]; Kechao Zhou[1]; [1]Central South University, The State Key Lab. for Powder Metall., Changsha 410083 China

Equal channel angular pressing (ECAP) is a novel method for producing very high plastic strain, and, subsequently, ultrafine grains in materials, with no change in the billetís shape. However it is very important to understand the deformation behavior of the materials and their relationships with the friction condition and tooling configuration during the ECAP process before it can be exploited in the engineering field. In this study, the plastic deformation behavior of pure Al during the ECAP process with a die corner angle (120∫) was investigated using the commercial two-dimensional rigid-plastic finite element software (DEFORM2D). It has been found that the ECAP process was characterized by five steps based on the analysis of the calculated load-ram displacement curve (fast increase, slow increase, fast increase, steady and finally decreasing stages). The inhomogeneous strain distribution within the billets was analyzed by separating the billets into a front transient zone, an end transient zone, an outerless sheared zone, and the remaining shearing deformation zone, which is also proved by the experiment using the billets with scribed grids. The homogeneity of the strain distribution in the middle of billet is strongly dependent upon the material flow rate distribution and the stress state in the deformation zone. During the ECAP process, the stress state transits from the compressive stress to tensile stress in the inner part of the billets, while the stress state transits from the compressive stress, then tensile stress, and to compressive stress in the outer part of the billets, which, in part, attributes to forming inhomogeneous deformation. Due to the faster flow of the outer part compared with the inner part within the main deforming zone, the lesser shear zone in the outer part of the billets occurs. The inhomogeneity of deformation still occurs even under the condition of a free friction between the samples and the dies. However, if using a round die instead of a corner die, the material flow rate in the main deformation zone is nearly homogenous because of the inexistence of the tensile component in the outer part of the billets. Consequently, the homogeneity of deformation is greatly enhanced.

5:10 PM

Evaluating the Induced Strain During Equal Channel Angular Processing: *Grigoreta Mihaela Stoica*[1]; Peter K. Liaw[1]; [1]University of Tennessee, Matl. Sci. & Eng., 323 Dougherty Eng. Bldg., Knoxville, TN 37996-2200 USA

Grain refinement using Equal Channel Angular Processing (ECAP) has become well known in improving the mechanical properties of metals and composites. Any plasticity modeling of the microstructure changes during ECAP requires reliable macroscopic strain and strain rate data (and this is also consequential for comparing with other processes). The evaluation of the shear strain induced during one pass and/or multipass ECAP is still under debate, as well as the definition of the equivalent strain and strain rate. The present analysis is aimed at reconsidering the slip-line field (SLF) modeling in order to achieve a better description of the experimentally observed plastic deformation zone (PDZ) for ECAP severe plastic deformation. Strain and strain rate deduced using SLF theory are compared with finite element modeling (FEM) results for perfect-plastic and work-hardened materials. The SLF approach was used to describe the non-homogeneous deformation near the outer corner of the ECAP die by introducing the split of the shear plane into two cylindrical surfaces.

5:30 PM

Molecular Processes in Creep and Durability of a Loaded Oriented Crystalline Polymer: *Ulmas Gafurov*[1]; [1]Institute of Nuclear Physics, Tashkent 702132 Uzbekistan

Molecular models of chain slippage and rupture, interrelation of these molecular processes and durability for a loaded high-oriented amorphous-crystalline polymer is suggested. It is taken into account complex interaction between slippage and rupture of interconnecting polymer chains. According to the molecular model in polymer sample creep, chain rupture accumulation and polymer fracture the leading molecular process is mechanically stimulation of thermo-fluctuation slippage of interconnecting molecular chains. It is valid for flexible chain polymers at least. The activation energy value for the chain slippage process and consequently mechanical durability (fracture process) depend on intermolecular interaction and consequently on temperature and molecular chain flexibility.

Materials Processing Fundamentals: Solidification and Forming Processes

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee, EPD-Process Fundamentals Committee

Program Organizers: Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA; Princewill N. Anyalebechi, Grand Valley State University, L. V. Eberhard Center, Grand Rapids, MI 49504-6495 USA

Tuesday PM Room: 1A
March 4, 2003 Location: San Diego Convention Center

Session Chair: Adam C. Powell, Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Cambridge, MA 02139-4307 USA

2:30 PM

Directional Solidification of Commercial Brass: Alicia Esther Ares[2]; Rubens Caram[3]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Scis., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]CONICET/University of Florida, Dept. of Matls. Sci. & Eng., 131 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA; [3]State University of Campinas, Matls. Eng. Dept., Campinas, Sao Paulo CP 6122 Brasil

The columnar-to-equiaxed transition (CET) was observed in brass samples directionally solidified from the chill zone in different solidification conditions. The transition occurs when the gradient in the liquid ahead of the columnar dendrites reaches values between -2.23∞C/cm and 1.05∞C/cm and the growth velocities reach values between 0.13 cm/s and 0.34 cm/s. The microstructure obtained is analyzed taking into account the characteristics of the alloy and the temperature profiles at the solidification interface. The variation of the primary and secondary dendritic spacings along the length of the sample is analyzed and the values experimentally measured are compared with results predicted by different theoretical models.

2:50 PM

The Effect of Solidification Parameters on Dendrite Spacing in Unidirectional Solidification: Alicia Esther Ares[2]; Rubens Caram[3]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Sci., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]CONICET/University of Florida, Dept. of Matls. Sci. & Eng., 131 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA; [3]State University of Campinas, Matls. Eng. Dept., Campinas, Sao Paulo CP 6122 Brasil

Experiments were carried out in which the conditions of dendritic growth are known, as well as the velocity and direction of solidification, in alloy systems such as Al-Cu, Al-Mg, Al-Zn, Al-Li, Al-Si, Al-Si-Cu, Cu-Zn, Pb-Sn, Sn-Pb and Steel. From these experiments it was determined that the primary dendritic spacing is a function of the temperature gradient, G, and the growth velocity, V, while the secondary and tertiary dendritic spacing are a function of the local solidification time, tSL. In the present work, we investigated the relation proposed by Hunt, Kurz and Fisher and by An and Liu in such alloy systems with dendritic growth, besides that, we correlated secondary and tertiary measured dendritic spacings with the equations proposed by Kattamis et. al., Allen and Hunt, Grugel and Feurer for the same alloy systems.

3:10 PM
Large Refractory Metal Single Crystals Grown by Plasma-Induction Zone Melting: *Viktor A. Shapovalov*[1]; [1]E.O. Paton Electric Welding Institute, 11 Bozhenko Str., Kiev 03680 Ukraine

The expanding applications of refractory metal single crystals has placed increasing demands on their properties and performance, especially concerning their enlargement and structural perfection. Due to their high melting point, the production of tungsten and molybdenum single-crystals is technically challenging. The growth of high quality crystals is critically dependent on the management of temperature profiles at the liquid-solid interface and the thermal fields in the solidified crystal. At the E.O. Paton Electric Welding Institute, a process for large crystal growth (a variant of the Verneuil method) has been developed. Special equipment for the production of large single crystal slabs of high purity and crystallographic perfection have been designed and installed. The crystal perfection is improved with the application of two independent heat sources (plasma-arc and induction) and process parameters have been optimized (principally, the ratio of plasma arc to induction heating power) in order to manage the thermal state during crystal growth and cooling. These parameters have been found to be the main factors controlling the production of high quality crystals. The plasma-arc source creates a local liquid pool of optimum form on the horizontally oriented seed crystal for the melting of the consumable materials (rods) for growth of the crystal in the shape of a slab. The choice of seed crystal orientation allows the control of material performance through the orientation dependence of properties in single crystals. The induction heater acts as a second independent heat source and acts to limit thermal losses as the slab grows. It effectively reduces the residual strains present in the crystals. Using the method described, single crystals of sizes up to 20 X 140 X 170 mm3 have been produced. Metallography and x-ray diffraction topography have been used for macro- and microstructural characterization of the single crystal slabs. Additionally, the large size of these crystals offers unique opportunities for measuring physical and mechanical properties.

3:30 PM
Evolution of Recrystallization Texture in Drawn OFHC Copper Wire: *Daudi R. Waryoba*[1]; Peter N. Kalu[1]; [1]National High Magnetic Field Laboratory, Matls. Sci. & Tech., 1800 E. Paul Dirac, Tallahassee, FL 32310 USA; [1]Florida State University, Mechl. Eng., National High Magnetic Field Lab., 1800 E. Paul Dirac, Tallahassee, FL 32310 USA

Oxygen free high conducting (OFHC) copper rod was drawn, at room temperature, to a true strain of 2.31 and annealed at various temperatures between 200∞C and 900∞C. Local orientations of the microstructures were measured by means of electron backscattered diffraction (EBSD) technique, from which inverse pole figures were calculated. The results of this study show that the starting, as-drawn, microtexture of the wire was a duplex <111>+<100> fiber texture. For annealing temperatures between 200∞C and 600∞C, a <111>+<112>+<100> fiber texture was formed. However, the intensity of <112> and <100> components were observed to increase at the expense of the <111> component. For annealing temperature between 600∞C and 800∞C, the intensity <111> component was observed to increase at the expense of the <100> and <112>. At 900∞C, the microtexture of the wire was essentially <111> fiber texture. It was concluded that the wire recrystallization texture of OFHC copper develops from the deformation texture. The volume fractions of the <111> and <100> depend on the annealing temperature. At low annealing temperature, the <100> component is enhanced due to depletion of the <111> component. At higher temperature, the <100> is unstable and is consumed by the <111> orientation. The <112> orientation is a transition component between the two major constituents. Thus, contrary to previous results that associated the <112> recrystallization texture with secondary recrystallization, the results of this study show that the <112> can also be formed at medium temperatures well before secondary recrystallization.

3:50 PM Break

4:10 PM
Effect of Oxygen Content and Processing on Ti-6Al-4V Ti Alloy Combat Vehicle Components: *Mustafa Guclu*[1]; Ibrahim Ucok[1]; Joseph R. Pickens[1]; [1]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA

Titanium (Ti) alloys are of interest to Army ground vehicle designers because of their unique combination of ballistic, corrosion and mechanical properties, such as high specific yield strength, fracture toughness and fatigue resistance. However, widespread use of Ti alloys has been limited by their relatively high cost compared to steels and aluminum alloys. Track components are good candidates for the application of titanium because the high specific strength and good fracture toughness of Ti alloys can be exploited to reduce the weight of moving parts. The use of single-melt Ti-6Al-4V combined with near-net shape processing technologies such as casting and forging can reduce fabrication cost even further. The Combat Vehicle Research (CVR) program at Concurrent Technologies Corporation (CTC) deals with cost reduction issues in fabrication of titanium components by using single melt titanium and simulating single melt titanium in manufacturing. In this study, Ti-6-4 track blocks were fabricated by rammed graphite, centrifugal investment casting, and open and closed die forging. The effects of oxygen level and processing method on mechanical properties were examined and compared to a standard Ti-6Al-4V alloy. Fabricated track blocks will be field-tested on the Marines Advanced Amphibious Assault Vehicle. The data presented in this paper will be useful to designers for assessing the viability of low-cost Ti-6Al-4V to reduce weight for combat vehicles. This work was conducted by Concurrent Technologies Corporation (CTC) for the Tank-automotive and Armaments Research Development & Engineering Center (TARDEC) through the National Center for Excellence in Metalworking Technology (NCEMT). CTC operates the NCEMT under Contract No. N00014-00-C-0544 to the US Navy as part of the US Navy Manufacturing Technology Program.

4:40 PM
Microstructure and Deformation of Microalloyed Steels in the Two-Phase Region: *Janusz Majta*[1]; Anna K. Zurek[2]; [1]University Mining & Metallurgy, Metall. & Matls. Sci., Mickiewicza 30, Krakow 30-059 Poland; [2]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

Traditionally conducted hot working processes with in depth knowledge and control of most important metallurgical phenomena are still very beneficial for achievement of high quality products for steels as long as the microalloyed elements are used. Recently developed technology of metal forming processes in the two-phase and ferrite regions, made it possible to enter new mechanical property characterization, controlled structure inhomogeneity and finally new implementations. To obtain these possibilities the computer control process of the full production technology is definitely needed. Thermomechanical processing strongly affects the microstructure components and the resultant mechanical properties of microalloyed steels. Therefore the influence of the process parameters on the microstructure and mechanical properties of niobium and boron treated steels has been investigated. The phenomena that have been taken into account are deformation temperature, amount of accumulated strain (both, in the austenite and ferrite phase), cooling rate during and after transformation, and dissolution of precipitates. For studying the microstructure development products that were formed in hot and intercritical conditions, rolling and compression tests were performed. The corresponding microstructures were investigated by means of TEM and optical microscopy. The obtained relationships, generalizing the problem, should optimize the technological parameters of the rolling, forging processes, considering obtaining the required distributions as well as average values of mechanical properties of final product. To reach the objective, the constitutive equations are proposed for modeling the effects of the hot-deformation process occurring in the austenite and two-phase region on the texture development of the resulting product. These models link advanced, finite element approaches simulating metal flow and heat transfer during hot-plastic deformation with constitutive equations describing microstructural processes, phase transformation and mechanical properties. Attention has so far been given to the calculation of the amount of transformed austenite and changes in temperature distributions.

5:00 PM
Effect of an Heterogeneous Distribution of Dissolving Particles on the Formation of Banded Grain Structure in Wrought Alloys: *SÈbastien Coste*[1]; Eric Andrieu[1]; Julitte Huez[2]; Olivier Brucelle[2]; [1]ENSIACET, CIRIMAT, 118 rte. de Narbonne, Cedex 4, Toulouse

31077 France; [2]Aubert & Duval Fortech, Service MÈtallurgie, 75 Blvd. de la LibÈration, Pamiers 09102 France

Alloy 718 is known to be sensitive to the interdendritic segregation forming during the ingot solidification. The occurrence of banded grain structures under heat treating conditions close to 1000∞C in relation with interdendritic segregation is often reported. In order to better understand this microstructural evolution, an extensive experimental program has been carried out. Finally, models based on selective dissolution of δ phase are proposed to account for these evolutions. The approach is detailed here after. In the first part of the present work, a grain growth simulation by Monte-Carlo method is proposed to illustrate the grain structure evolution in a banded particle distribution. However, the study of grain growth has shown that the banded structure can disappear under specifics heat treating conditions. Thus, in the second part of the study, a δ phase solutionning model is developed to point out the evolution of the δ fraction according to the local chemical composition and the heat treatment testing conditions.

5:20 PM

Mutual Effects of Electro Slag Remelting and Open Die Radial Forging on the Properties of V-Microalloyed Steel: Taha Mohamed Taha Mattar[1]; *Taher Ahmed El-Bitar*[1]; [1]CMRDI, Metal Forming, PO Box 87, Helwan, Cairo 11421 Egypt

Both ESR and forging processes have a pronounced effect on the steel properties. ESR process improves the properties of steel. The metal forming processes are not only used for dimensional purposes but also for enhancing the mechanical properties of steel. The present work aims at studying the mutual effects of both ESR and open die radial forging process on the mechanical properties and structure of V-microalloyed steel. In this study V-HSLA steel consumable electrodes were produced in an EAF. These electrodes were electro-slag remelted. The produced ingots were subjected to open die radial forging. Forging has been carried out to different amounts of reduction in cross-sectional area. The forged bars were subjected to both mechanical testing and microstructure investigations. The obtained data showed pronounced improvements in the microstructure and mechanical properties of produced steel.

Materials Processing Under the Influence of Electrical and Magnetic Fields - IV

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Joanna R. Groza, University of California-Davis, Chemical Engineering Material Science Department, Davis, CA 95616 USA; George S. Dulikravich, The University of Texas at Arlington, Multidisciplinary Analysis, Inverse Design, and Optimization (MAIDO) Program, Department of Mechanical and Aerospace Engineering, Arlington, TX 76019 USA; Nagy H. El-Kaddah, University of Alabama, Department of Metallurgical & Materials Engineering, Tuscaloosa, AL 35487-0202 USA; James W. Evans, University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA; Zuhair Munir, University of California, College of Engineering, Davis, CA 95616-5294 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Tuesday PM — Room: 14A
March 4, 2003 — Location: San Diego Convention Center

Session Chair: Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

2:30 PM Workshop

3:15 PM Workshop on the Influence of External Fields on Materials Processing: Schedule TBA

4:00 PM Workshop on Electrical and Magnetic Field Effects in Materials Processing Invited speaker: Dr. K. L. Murti (NSF)

4:45 PM Break

Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction: Damage Interrogation

Sponsored by: Structural Materials Division,

Program Organizers: James M. Larsen, US Air Force, Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA; Leo Christodoulou, Defense Advanced Research Agency, Arlington, VA 22203-1714 USA; William J. Hardman, Naval Air Systems Command, Propulsion and Power Directorate (AIR 4.4.2), Patuxent River, MD 20670-1534 USA; Andrew J. Hess, Naval Air Systems Command, Propulsion and Power Directorate, Patuxent River, MD 20670-1534 USA; J. Wayne Jones, University of Michigan, College of Engineering, Department of Materials, Science and Engineering, Ann Arbor, MI 48109 USA; Stephan M. Russ, Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA

Tuesday PM — Room: 16A
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: William J. Hardman, Naval Air Systems Command, Propulsion & Power Direct., Patuxent River, MD 20670-1534 USA; Reji John, Air Force Research Laboratory, AFRL/MLLMN, WPAFB, OH 45433-7817 USA

2:00 PM

Nonlinear Acoustics and X-Ray Diffraction for Evaluation of the Fatigue Damage-State in Ti-6Al-4V: *Shamachary Sathish*[1]; Jerome Frouin[1]; [1]University of Dayton Research Institute, 300 College Park, Dayton, OH 45469-0127 USA

Nonlinear acoustics and x-ray diffraction techniques have been used to study the changes in the local properties in fatigue-fractured samples of Ti-6Al-4V. Large changes in the nonlinear acoustic parameter and the x-ray diffraction peak width have been observed as the fracture end is approached. The role of changing microstructure and the dislocation density due to cyclic loading will be utilized to explain the experimental observations. Application of the experimental methods for the detection of the onset of fatigue damage and its implication on the prognostication of fatigue cracks are discussed.

2:30 PM Invited

Material Characterization and Life Prediction for Critical Components Through Application of Photon Induced Positron Annihilation (PIPA): *Douglas Akers*[1]; [1]Positron Systems, Inc., 6151 N. Discovery Way, Boise, ID 83713 USA

A new patented technology, Photon Induced Positron Annihilation (PIPA) has been developed that provides a revolutionary damage prognosis capability to nondestructively detect all types of failure mechanisms such as fatigue, creep, and radiation induced damage, at levels less than 1% change in the atomic lattice structure. Further, PIPA can accurately assess existing defect levels and predict the remaining life of various metallic, composite, and polymeric materials. PIPA has successfully detected microstructure fatigue mechanisms and crack growth phenomena in numerous materials, such as aluminum and super nickel alloys, stainless steel and titanium. This technology has numerous applications in the power generation and aerospace industries from the assessment of high temperature creep in steam piping to fatigue and compressive stress release characterization on aircraft jet engine fan disks and turbine blades. Additionally, PIPA has demonstrated the capability to detect damage in 2nd layer materials, without disassembly; demonstrating in situ interrogation capability.

2:55 PM

Scattered Light Scanning for Fatigue Damage Precursor Detection on Turbine Components: *James C. Earthman*[1]; [1]University of California-Irvine, Cheml. Eng. & Matl. Sci., Eng. Tower 916, Irvine, CA 92697-2575 USA

A technique for in-situ laser light scanning (LLS) was developed to monitor surface damage on nickel-base superalloy turbine component under low-cycle fatigue conditions. This technique characterizes the surface state using the defect frequency parameter which was designed to minimize memory requirements and data processing time. As a result, the present technique is capable of scanning speeds that are substantially greater than those achieved with image processing methods. Sections of Inconel 718 turbine rotors were tested using a servo-hydraulic MTS machine at ambient temperature under load control conditions. The fatigue damage was monitored by scanning a laser beam along the rotor section in situ and during periodic interruptions of the cyclic loading. Acetate replicas of the gage section surface were also made to examine the surface mrophology using SEM. Comparisons of the results demonstrate the capabilities of the present light scanning technique for characterizing fatigue damage precursors on the surface of turbine components. In particular, a rapid rise in the mean defect frequency is shown to correspond to surface relief features that correspond to grain boundaries that intersect the surface in the areas of greatest stress. The presence of this surface relief can be attributed to the presence of relatively soft precipitate free zones along the grain boundaries that preferentially deform under fatigue loading conditions leading to the formation of microcracks.

3:20 PM Break

3:50 PM
Nondestructively Estimated Damage Degradation in Ceramic Matrix Composites: *Chris Deemer*[1]; Benjamin J. Wheeler[1]; William A. Ellingson[1]; [1]Argonne National Laboratory, Energy Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA

In ceramic matrix composites (CMC), the ability to quantify damage caused by unpredictable loading has been a goal for some time. Recently, an acousto-ultrasonic technique to nondestructively measure a damage parameter for monitoring matrix cracking in CMC materials has been developed. Data has been obtained on damaged Melt Infiltrated (MI) SiC/SiC and oxide/oxide CMC materials. Damage has been induced by using tension-tension cyclic fatigue, monotonic tensile loading, as well as thermal cycling and impact testing. Details of the NDE method and review the results are presented. Work supported by US DOE/EERE/OIT-CFCC Program under contract W-31-109-Eng-38.

4:15 PM
Ball Indentation for Nondestructive Monitoring of Thermal Aging of Cast Stainless-Steel (CF-8) and Alloy 625: *K. L. Murty*[1]; M. D. Mathew[2]; [1]North Carolina State University, 2500 Stinson Dr., PO Box 7909, Raleigh, NC 27695-7909 USA; [2]Indira Gandhi Center for Atomic Research, Kalpakkam 603102 India

Monitoring the progressive changes in the mechanical properties of high temperature materials in-service is important for ensuring the structural integrity of the components and also for possible extension of their useful service life. In this paper, we describe an innovative automated technique based on ball indentation that can be used for laboratory and field applications in order to measure the mechanical and fracture properties of materials along with typical results from laboratory studies on engineering materials. Thermal aging effects are considered in CF-8 stainless steel and a superalloy 625. Ball indentation test results are compared with those obtained from traditional tensile and fracture tests, and are correlated with the microstructural modifications due to thermal aging. Plausible extensions for in-service applications will be described. K.L. Murty is currently Program Director, Metals Research, DMR, National Science Foundation.

4:40 PM
Piezoelectric In-Situ Transmission Electron Microscopy Observations of Fatigue Damage Accumulation in Constrained Metallic Thin Films: X. Tan[1]; P. Liu[1]; *J. K. Shang*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA

A piezoelectric in-situ transmission electron microscopy (TEM) technique has been developed to observe damage mechanism in constrained metallic thin films under cyclic loading. The technique was based on the piezoelectric actuation of a multilayered structure in which a metallic thin film was sandwiched between a piezoelectric actuator and a silicon substrate. An alternating electric field with a static offset was applied on the piezoelectric actuator to drive the crack growth in thin metallic layer while the sample was imaged in TEM. The technique was demonstrated on solder thin films where cavitation was found to be the dominant fatigue damage mechanism.

5:05 PM
Mechanism-Based Models for the Prediction of Fatigue in Si MEMS Structures: Seyed Allameh[1]; M. Huang[1]; P. Shrotriya[2]; Y. Cao[1]; Z. Suo[1]; *W. O. Soboyejo*[1]; [1]Princeton University, Princeton Matls. Inst., D404 E. Quad., Princeton, NJ 08544 USA; [2]Brown University, Div. of Eng., Providence, RI 02912 USA

This paper presents mechanism-based models for the prediction of fatigue in silicon micro-electro-mechanical systems (MEMS) structures. Following a brief review of the experimental evidence of fatigue, the paper identifies the important role of the topical SiO2 layer on fatigue in silicon MEMS structures. Mechanism-based models are then used to model the different stages of environmentally-assisted fatigue under cyclic and static loading. These include: activation volume-based models for the prediction of surface roughening and crack nucleation; fatigue crack growth and stress corrosion cracking models for the prediction of crack growth, and bi-layer fracture models for the modeling of final failure conditions. The implications of the results are then discussed for the design of reliable Si MEMS structures.

Measurement and Interpretation of Internal/ Residual Stresses: Processing

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Shaping and Forming Committee
Program Organizers: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Bimal K. Kad, University of California, Ames Laboratory, La Jolla, CA 92093-0085 USA

Tuesday PM	Room: 17B
March 4, 2003	Location: San Diego Convention Center

Session Chairs: Tom Holden, Los Alamos National Laboratory, Eng. Sci. Applic. Design Eng., Los Alamos, NM 87545 USA; Camden R. Hubbard, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37830-6064 USA

2:00 PM
Residual Stress Relaxation in Fatigue of Mechanically Surface Treated Materials: *I. Altenberger*[2]; U. Noster[2]; R. K. Nalla[1]; B. Scholtes[2]; R. O. Ritchie[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]Institut fuer Werkstofftechnik, Un. Kassel, Kassel 34125 Germany

A necessary prerequisite for the effectiveness of mechanical surface treatments in enhancing the fatigue properties of a material is the mechanical and thermal stability of near-surface residual stresses or microstructures. Only stable residual stresses influence fatigue strength/ life and are usually regarded as local mean stresses. Additionally, near-surface microstructures, such as those associated with increased dislocation densities, strongly influence fatigue performance of mechanically surface treated materials; however, the interactions between the residual stresses and near-surface microstructures are rarely investigated. It is the objective of this paper to clarify the role of fatigue loading parameters, such as the stress amplitude and plastic strain amplitude as well as that of the surface treatment induced microstructures, in the relaxation of macro- and micro residual stresses during mechanical and thermal cycling. Characteristic examples of fatigue-induced residual stress relaxation in various metallic engineering alloys, including steels, titanium and magnesium alloys, are presented and recommendations for optimizing the stability of the residual stresses are proposed.

2:30 PM Invited
An Investigation of Springback Stresses in Sheet Metal Forming: *Thomas Gnaeupel-Herold*[1]; Henry Prask[1]; [1]NIST, Ctr. for Neutron Rsrch., 100 Bureau Dr., Stop 8562, Gaithersburg, MD 20899 USA

Springback is the shape change of sheet metal after forming and removal from the die. In order to achieve sufficiently small product tolerances, conventional die design involves expensive, time-consuming trial-and-error methods which represent major challenges in the cost-effective manufacturing of lightweight and fuel efficient vehicles. In the Springback Predictability Project there is a joint effort underway involving car companies, material suppliers and government agencies to develop a 3D computer model to predict stress, strain and fracture in sheet metal with the goal of a significant reduction in tool design time. The necessary data for understanding the forming process is being collected. Springback is an effect of residual stresses that are caused by different levels of accumulated plastic strain through the thickness of sheet metal. Upon release from the die, these stresses create a bending moment which is essentially the integrated through-thickness stress. The computer model is benchmarked by these stresses which are, in turn, benchmarked by the bending moment that can be measured my mechanical means. In a simplified approach, both modeling and measurement of springback stresses is focused on deep drawn cups after die removal and trimming of top and bottom of the cup. Through-thickness axial and hoop stresses were measured on Al-6022 and steel for wall thicknesses ranging from 0.9 mm to >3 mm and for different locations axially and around the cup circumference by means of synchrotron radiation and neutron diffraction. Spatial resolutions varied, depending on thickness, from 0.05 mm up to 0.5 mm. It was found that the through-thickness hoop stresses deviate distinctively from the symmetric S-profile sometimes assumed in simulations. We also obtained results indicating substantial stress variations in the axial direction. An update on the status of the predictability project will be given.

3:00 PM

Mapping Complex Residual Stresses in a Large, Cold Worked, Aluminum Forging: *Michael B. Prime*[1]; [1]Los Alamos National Laboratory, Eng. Scis. & Applic. Div., MS P946, Los Alamos, NM 87545 USA

Distortion in high-strength aluminum forgings has been an extremely costly problem for aerospace manufacturers. The distortion is caused when residual stresses are released as the part is machined to its final dimensions. The problem occurs because the complex cold compression process that is used to relieve quenching residual stresses in the forgings is not sufficiently effective. In this study, the residual stresses before and after cold compression of a large (107x158x718 mm) 7050-T74 forging were mapped using the contour method. The results show a spatially periodic variation of stresses that results from the periodic nature of the cold work. The results compare favorably with a finite element prediction of the stresses. The reasons why the stress relief process is not successful, compared to the stretching process used for plate, is discussed. Also discussed is why the residual stress measurements probably would not have been possible with any method other than the contour method.

3:20 PM

Modeling and Measurement of Temper Stresses in Bulk Metallic Glasses: *Cahit Can Aydiner*[1]; Ersan Ustundag[1]; Michael B. Prime[2]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Eng. Scis. & Appl. Div., Los Alamos, NM 87545 USA

New multi-component metallic alloys with exceptional glass forming ability have recently been developed at Caltech. These alloys allow the processing of large amorphous specimens. The possibility of formation of thermal-tempering-induced residual stresses during the processing of these bulk metallic glass specimens was investigated via several models. In particular, the viscoelastic nature of the material was considered. The crack compliance method was then used to measure the residual stress profiles. The measured profiles were roughly parabolic suggesting that thermal tempering was the dominant residual stress generation mechanism. It was shown that material properties and processing conditions exert strong influence on the final residual stress values.

3:40 PM

Microstrain Evolution During Creep of a Polycrystalline Superalloy: S. Ma[1]; D. Brown[2]; B. Clausen[2]; M. Bourke[2]; B. S. Majumdar[1]; [1]New Mexico Tech, Matls. Dept., Socorro, NM 87801 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

The creep resistance of superalloys containing a high volume fraction of gí phase is strongly influenced by lattice misfit, and by constraints imposed on the narrow g channels by the primarily non-deforming gí precipitates. In order to probe deformation behavior at the g/gí level, we have conducted creep experiments at 900C on a polycrystalline superalloy CM247LC. A pulsed neutron diffraction source was used to capture the insitu *d*//bsb/*hkl*//esb/ and appropriate procedures were developed to deconvolute the phase strains. The elastic misfit was negligible at room temperature, but increased to a significant negative value at 900 C, consistent with estimates based on thermal strain calculations. During creep, the evolution of microstrains was a function of grain orientation, and could be categorized in terms of location within the stereographic triangle. For example, grains with the loading axis close to the [001] orientation exhibited a monotonic increase in elastic microstrain in the gí phase as a function of creep time. In contrast, the corresponding g-phase exhibited a *decrease* in microstrain during the tertiary creep stage. We rationalize this latter behavior in terms of a build up of interface dislocations in the vertical g channels. While such interface dislocations have sometimes been observed in the past, this work likely provides the first data validating stresses arising from such a dislocation network. These stresses are important, because microstructural rafting, which is a major reason for loss of creep resistance at high temperatures, is driven by unequal strain energy in the horizontal and vertical g channels. This work was supported through contract UCDRD-10012. We thank the SMARTS LANSCE center for the neutron diffraction experiments

4:00 PM Break

4:15 PM

Process Optimization Through Measurement and Monitoring of Residual Stresses Under Industrial Conditions: *Prabir K. Chaudhury*[1]; [1]Intercontinental Manufacturing, 1200 N Glenbrook Dr., Garland, TX 75040 USA

Residual stresses play a very important role in manufacturing processes of structural parts. Monitoring and control of the residual stresses induced by various industrial metalworking processes are important for reducing cost of many structural aerospace parts. This investigation focuses on a commercial aircraft part to demonstrate the process optimization for reducing residual stresses through measurement of residual stresses by machining and x-ray diffraction techniques. First, the heat treatment process was optimized to reduce residual stresses without significantly sacrificing mechanical properties. Then various methods of residual stress relieving was conducted and the residual stresses were monitored to determine their effectiveness. Distortion measurement after machining an area of a forged and heat treated aluminum part was primarily used as a measure of residual stresses. Some of the distortion measurments were compared and verified with data from x-ray diffraction measurements.

4:35 PM

Residual Stresses in Components Made by the Laser-Engineered Net Shaping (LENS™) Process: *Partha Rangaswamy*[1]; Thomas M. Holden[1]; Ronald B. Rogge[2]; Michelle L. Griffith[3]; Michael B. Prime[1]; [1]Los Alamos National Laboratory, Eng. Sci. Applic., Design Eng., Los Alamos, NM 87545 USA; [2]National Research Council of Canada, Neutron Prog. for Matls. Rsrch., Chalk River Labs., Chalk River, Ontario K0J 1J0 Canada; [3]Sandia National Laboratory, Albuquerque, NM 87185 USA

During manufacturing of components using the Laser Engineered Net Shaping (LENSô) process the introduction of residual stresses is a problem associated with cracking for volumes greater than 100 cm^3. The origin is attributed to thermal transients encountered during solidification. In the absence of predictive models measurements are imperative. Preliminary measurements using a Laser Holographic Drilling Technique (holographic) on 316 stainless steel specimens have shown yield level tensile residual stresses. However, the holographic technique is restricted to near surface measurements (<1 mm), accordingly we used neutron diffraction to probe greater depths (25-50 mm). Residual stresses were measured in LENSô samples (316 Stainless Steel) having simple geometrical shapes. Using the L3 spectrometer at Chalk River Laboratories (Canada), stresses were mapped spatially with sampling volumes ranging from 2 to 10 mm^3 in three orthogonal directions. The neutron (core) and holographic (surface) results will be compared and discussed in the contest of growth direction during the LENS process. These results will also be compared against residual stresses obtained using contour method, which provides spatial distribution normal to the plane of sectioning.

4:55 PM Invited

Shot-Peen Residual Stress Relaxation and its Application in Life Management of Turbine Engine Components: *Reji John*[1]; James M. Larsen[1]; Mark P. Blodgett[2]; Stephan M. Russ[1]; Jeffrey Finch[1]; Bryan Sanbongi[2]; [1]US Air Force Reseach Laboratory, AFRL/MLLMN, Matls. & Mfg. Direct., WPAFB, OH 45419 USA; [2]US Air Force Research Laboratory, AFRL/MLLP, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA

The US Air Force has initiated a technology development initiative known as Engine Rotor Life Extension (ERLE), which has the goal of extending the useful lifetime of major, fracture-critical components in gas turbine engines, without increasing the fleet-wide risk of component failure. Current damage-tolerance based life management practices do not explicitly account for the beneficial effects of shot-peen residual stresses. This presentation will critically assess the role of residual stresses in the durability of representative turbine engine materials and identify opportunities for improvement in life prediction methods. Recent studies have shown that residual stresses in engine components relax during service. Typically, this relaxation effect occurs across the entire residual stress profile. Hence, reliable NDE techniques are required to measure the residual stress profile during inspections. This presentation will discuss the life prediction and NDE issues to be considered prior to incorporation of residual stresses in engine component life management practices.

5:15 PM

The Effects of Shot Peening Surface Coverage on the Residual Stress of Fretting Fatigued Ti-6Al-4V Samples: *Sonia Aixa Martinez*[1]; Shamachary Sathish[2]; Mark P. Blodgett[1]; Shankar Mall[3]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth

St., Ste. 1, WPAFB, OH 45433-7817 USA; [2]University of Dayton Research Institute, Structl. Integrity & Non Destructive Evaluation, 300 College Park, Dayton, OH 45469-0127 USA; [3]Air Force Institute of Technology, Eng. Dept., WPAFB, OH 45433-7765 USA

Residual stresses generated by shot peening are compressive in nature. The depth of these stresses is a function of the peening intensity. Increasing the peening intensity generates a deeper layer of compressive stresses, this is desirable for crack growth resistance and fatigue life. Optimum residual stress distribution can be obtained if there is an understanding of the effect of shot peening parameters. The effect of shot peening surfaces under fretting wear is of importance in this research. Fretting is a contact loading that modifies the surface of the component and can lead to relaxation of the compressive residual stresses and loss of matter. In this investigation, the effects of fretting fatigue on the residual stress distribution and fatigue life of Ti-6Al-4V shot peened samples have been studied. Experiments are conducted in dry conditions for samples made with different surface coverages. X-ray diffraction has been used as a method for measuring surface residual stress.

Microstructural Processes in Irradiated Materials: Multiscale Modeling II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)

Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

Tuesday PM	Room: 11A
March 4, 2003	Location: San Diego Convention Center

Session Chairs: Yuri Osetsky, The University of Liverpool, Dept. of Eng., Brownlow Hill, Liverpool L69 3GH UK; Charlotte Becquart, Universite de Lille I, Lab. de Metallurgie Physique et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

2:00 PM

1-D to 3-D Diffusion Reaction Kinetics of Cascade Induced SIA Clusters in MetalsñImplications for Void Nucleation and Growth: *Helmut Trinkaus*[1]; Bachu N. Singh[2]; [1]ESS Project, Forschungszentrum J¸lich, J¸lich D-52425 Germany; [2]Risoe National Laboratory, Matls. Rsrch. Dept., Roskilde 4000 Danemark

It has been shown recently that some features in the microstructural evolution occurring in metals under cascade damage can be rationalised in terms of intra-cascade clustering of vacancies and self-interstitial atoms (SIAs), differences in the thermal stability and mobility of the resulting clusters and one-dimensional (1-D) glide of SIA clusters. We discuss consequences of deviations of SIA cluster diffusion from strictly 1-D, resulting in diffusion reaction kinetics between the 1-D and 3-D limiting cases. Analytical solutions for the partial sink strengths of voids and dislocations for the annihilation of SIA clusters in such general reaction kinetics are presented. An intermediate kinetics is identified where the sink strength increases continuously with increasing disturbance from low values for 1-D to high values for 3-D. The implications of such changes in the SIA cluster reaction kinetics, particularly for enhanced swelling near grain boundaries and saturation of void growth, are discussed.

2:45 PM

Meaning, Relationship and Influence of Interaction Radii Used in Rate Theory and Kinetic Monte Carlo Modelling of the Clustering of Point Defects: Christophe Domain[3]; Charlotte S. Becquart[2]; *Alain Barbu*[1]; [1]CEA, LSI, Ecole Polytechnique, Palaiseau F-91128 France; [2]LMPGM, UMR 8517, UniversitÈ de Lille 1, Villeneuve díAscq F-59655 France; [3]EDF R&D, MMC, Moret sur Loing F-77250 France

One of the key parameter in modelling the nucleation and growth of point defect clusters in rate theory as well as in lattice Kinetic Monte Carlo (KMC) is the interaction radius (recombination or clustering radius). Depending upon the model used, the meaning of this radius is rather different. In lattice KMC, defects migrate from a lattice site to a nearest neighbour site through jumps characterised by jump frequencies, the interaction radius is the critical discrete distance (which can depend on the relative crystalline direction) below which an instantaneous reaction occurs. In rate theory, the defects migration is no more described in terms of jumps but by diffusion coefficients which are more global quantities. As a consequence, the physical meaning of the capture radius is not straightforward. The reason is that it takes into account the different paths that the defects can follow to get close to each other at the atomic level. This point will be illustrated by KMC and rate theory simulations of electron irradiations in pure Fe. These simulations will be compared to experiments carried out with 2.5 MeV electrons.

3:05 PM

Ab Initio Contribution to the Study Interaction of Point Defects with Interstitial Impurities (C, N) in Fe: Christophe Domain[2]; *Charlotte S. Becquart*[1]; [1]UniversitÈ de Lille I, LMPGM, UMR 8517, Batiment C6, Villeneuve díAscq, CÈdex 59655 France; [2]EDF-R&D, MMC, Les RenardiËres, Moret sur Loing F-77818 France

The critical importance of interstitial impurities in bcc metals has been observed over many years and is now well established. Under irradiation, the defects produced appear to serve as trapping centres for these interstitial impurities and as a consequence the mechanical properties of the materials are affected. We have investigated, by ab initio calculations based on the density functional theory, the interactions of C and N atoms with point defects in Fe. The structures and relative stabilities of the different configurations as well as their formation and binding energies have been determined. The consequences of the results are discussed.

3:25 PM Break

3:55 PM

Sink Strengths Revisited: N. V. Doan[1]; G. Martin[1]; J.-L. Bocquet[1]; *J. Dalla Torre*[1]; [1]CEA Saclay, DMN/SRMP, Bat. 520, Gif-sur-Yvette 91191 France

The rate theory of irradiation effects in crystalline solids rests on a set of two ordinary differential equations which, for each type of point defect (vacancy and self-interstitial), describe the balance between the production of defects and their annihilation. The elimination rate on point defect sinks is proportional to the defect concentration times the defect diffusion coefficient times a geometrical factor, the ìsink strengthî. We propose a new criterion to define the sink strength: the value of the sink strength should give the correct value for the partitioning of the defect annihilation between mutual recombination and elimination on sinks. Depending on the irradiation conditions, the correcting factor on individual sink strengths may be large (several orders of magnitude). When several types of sinks compete, the sink strengths, as defined in this work, are additive, at variance with the classical ones.

4:15 PM

Modeling Structural Metastability of Irradiated Thin Films: Paolo M. Ossi[1]; *Alessio Lamperti*[1]; [1]Politecnico, Dip. Ingegneria Nucleare, via Ponzio, 34/3, Milano 20133 Italy

The experimentally observed structure evolution in irradiated binary compounds, both metallic and non-metallic, where dense collision cascades form, is modelled by the Segregation-Charge Transfer (SCT) atomistic model. The cascade space and time evolution produces non-equilibrium compositional and electronic density profiles at the cascade-crystalline matrix interface. This is due to short-range interface migration of one film constituent. System relaxation to metastable equilibrium is simulated by charge transfer reactions (CTR), each involving a couple of dissimilar atoms of the initial compound (IC), that generate an effective compound (EC) dimer. By SCT model we calculate the energy cost to insert an EC dimer into the matrix, the formation enthalpy difference between corresponding EC and IC, and the local volume change the film undergoes when a CTR results in ion formation. For the above structure stability parameters threshold values provide a qualitative separation between amorphised and crystalline compounds, both metallic and non-metallic.

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - IV

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Alloy Phases Committee

Program Organizers: Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Michael R. Notis, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18015 USA; Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA

Tuesday PM — Room: 12
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: Hyuck Mo Lee, Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., Taejon 305-701 Korea; Sinn-Wen Chen, National Tsing Hua University, Dept. of Cheml. Eng., Hsinchu 300 Taiwan

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2:00 PM Invited
Morphology Transition of Interfacial Intermetallic Compound at Sn-3.5Ag/Ni Interface: Jong Hoon Kim[1]; *Hyuck Mo Lee*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., Kusung-Dong 373-1, Yusung-Gu, Taejon 305-701 Korea

The morphology transition of Ni3Sn4 grains that formed at the interface between liquid Sn-3.5Ag (in wt.%) solder and Ni substrate has been observed at 650°C. This morphology transition of Ni3Sn4 is caused by decrease of enthalpy of formation of Ni3Sn4 phase and explained well through Jacksonís parameter. The solder joint strength with increase of soldering temperature decreased rapidly at around 6.5 micrometer thick IMC. However the solder joint strength with increase of soldering time decreased slowly without drop of solder joint strength although the thickness of IMC is larger than 6.5 micrometer. The drop of solder joint strength with increase of soldering temperature appears to be due to a large width of IMC grains.

2:20 PM Invited
The Morphology and Kinetics of Solid-State Intermetallic Compound Layer Growth Between Copper and Sn-Ag-XCu Solders (X = 0, 0.6, 4.0): *Paul Thomas Vianco*[1]; Jerome A. Rejent[1]; Paul Hlava[1]; [1]Sandia National Laboratories, MS0889, PO Box 5800, Albuquerque, NM 87185-0889 USA

The morphology and kinetics of interfacial, solid-state intermetallic compound layer growth are a function of compositions of the solder and base metal members of the couple. These properties were examined for Sn-based solders containing 0.6Cu (wt.%) or 4.0Cu, which were applied to 100Cu base metal by the hot solder dipping process. Aging was performed for time periods of 1-400 days and at temperatures of 70-205∞C. The 100Sn/Cu couples provided the baseline. The interface reaction kinetics were determined, based upon Arrhenius format. Electron microprobe analysis (EMPA) identified Cu3Sn and Cu6Sn5 sub-layers, the extents of which were dependent upon the particular solder composition. Understanding the mechanism that varies the Cu3Sn/Cu6Sn5 ratio is required for computational model development for these systems. Sandia is a multi-program laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Dept. of Energy under contract DE-AC04-94AL85000.

2:40 PM Invited
Reduced Melting Temperatures in Nanometer Length Scale Eutectic Alloys: Elisa Pueschel[1]; Richard Chromik[1]; Mitchell Johnson[1]; Natalya Chernova[1]; Scott Oppenheimer[1]; *Eric J. Cotts*[1]; [1]SUNY Binghamton, Physics & Matls. Sci., Sci. 2, PO Box 6016, Binghamton, NY 13902-6016 USA

Melting temperatures in eutectic alloys were examined as a function of relevant length scales. Thin film composites were fabricated by physical vapor deposition. Average compositions of these multilayered structures were near eutectic, or far from eutectic. The structure of the composites was examined by x-ray diffraction analysis and electron microscopy. Melting behavior was studied using differential scanning calorimetry. Reduced eutectic melting temperatures were observed in a number of systems, including Al-Sn and Au-Si. Support of the National Science Foundation, DMR 9902783 is gratefully acknowledged.

3:00 PM
Sputtered Copper Films with Insoluble Elements for Cu Metallization: A Thermal Annealing Study: Chon-Hsin Lin[1]; *J. P. Chu*[1]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2 Pei-Ning Rd., Keelung 202 Taiwan

Copper is an attractive material for metallization in microelectronics, owing to its low resistivity and high reliability against electromigration compared with Al and its alloys. However, Cu diffuses readily into Si and SiO2, resulting in the formation of copper silicide compounds at low temperatures. This work was directed towards the study of thermal stability, microstructure and electrical properties of Cu films containing dilute insoluble W or C. Cu-11.3at.%C and Cu-2.3at.%W films deposited on Si(100) substrate by R.F. magnetron sputtering were annealed at temperatures ranging from 200 to 800∞C. As-deposited Cu-C and Cu-W films consisted of non-equilibrium solid solutions of C in Cu with nanocrystalline microstructures. X-ray diffraction results show that Cu4Si is formed at 530∞C that is better than pure Cu film, which appears at 400∞C. This result is consistent with transmission and scanning electron microscopy observations and implies that the amount C and W could inhibit formation of Cu4Si compound. With the insoluble W for retardation effect, the film also maintains its fine structure after annealing up to 400∞C. On the contrary, the pure Cu film has suffered adverse recrystallization and reaction with Si substrate at 400∞C. Thermal stability of Cu-C and Cu-W films will be presented and discussed in the light of the results obtained.

3:15 PM
Electromigration in SnAg3.8Cu0.7 Solder Lines: *K. C. Lin*[1]; Ying-Chao Hsu[1]; T. L. Shao[1]; C. Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Eng., 1001 Ta Hsueh Rd., Hsinchu 300 Taiwan

Due to the excellent thermo-mechanical property, SnAg3.8Cu0.7 solder will be one of the most promise lead-free solders in microelectronic packaging industry. This study investigated the electromigration damage on lead-free SnAg3.8Cu0.7 solder in V-groove lines. V-groove lines were obtained on (001) Si wafers by anisotropic etching. Metallurgy layers of Cr/Cu/Au were deposited by e-beam evaporation. Nickel film were electroplated to form electrodes. V-grooves were finally filled with SnAg3.8Cu0.7 solder by reflowing the solder into the lines. Electromigration damage were investigated by applying current through the v-groove lines under a current density of $2*10^4$ A/cm2 at 100°C. Compounds of (Cu, Ni)6Sn5 were observed at the interface between the solder and the Ni electrode after reflow. After current stressing for seven days, copper content in (Cu, Ni)6Sn5 compound decreased. Also, Ni atoms were carried into the solder and reacted with tin atoms to form Ni3Sn4. Whiskers and hillocks were observed at the anode side, and voids were observed at the cathode side.

3:30 PM Break

3:50 PM Invited
Intermetallic Formation and Growth Around Metallic Particles in Sn-Based Solders: H. Rhee[1]; K. C. Chen[2]; J. G. Lee[1]; *K. N. Subramanian*[1]; [1]Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA; [2]California Polytechnic State University, Matls. Eng. Dept., San Luis Obispo, CA 93407 USA

Incorporation of metallic particulate reinforcements in Sn-base solder has resulted in intermetallic compounds (IMC) formation along the interfaces and with different morphologies. For instance, ìsunflowerî and ìblockyî faceted shapes have been observed around Ni particle reinforcements incorporated in eutectic Sn-Ag solder matrix. The amount of heat input during the heating stage above the melting temperature of solder has been found to be play a significant role in determining the IMC morphology shape in the Ni particulate reinforced solders. In order to identify and understand the operative mechanisms, similar studies were carried out with Cu and Ag particulate reinforcements in same solder matrix. Solidification and ageing effects are to be discussed. Acknowledgement: Project funded by National Science Foundation under grant No. NSF DMR 0081796.

4:10 PM Invited
Phase Stability and Elastic Properties of Intermetallics Relevant to Electronic Packaging: *Gautam Ghosh*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

Due to high thermodynamic driving force for interfacial reactions coupled with high mobility of the constituents, intermetallics are formed in solder joints both during fabrication of the devices as well in service.

TUESDAY PM

Depending on the solder chemistry and the metallization scheme, one or more intermetallics may form at the interface. Multicomponent interdiffusion may also cause phase transformation of intermetallic(s). Besides thermodynamic stability of the intermetallics, to understand the processing-structure-property relationship it is important to determine their elastic properties as a first step. Because, in modern electronic packages the intermetallics constitute a significant fraction of the solder joints. We will present the results of a systematic study of the phase stability, elastic properties and fracture toughness of Sn-base intermetallics. The intermetallics considered are Ag_3Sn, $AuSn_4$, Cu_3Sn, Cu_6Sn_5 and Ni_3Sn_4. Results include both recent experiments as well as theoretical modeling.

4:30 PM Invited

Interfaces in Lead-Free Soldering and Conductive Adhesive Joining: *Katsuaki Suganuma*[1]; Keun-Soo Kim[1]; Chi-Won Hwang[1]; Munenori Yamashita[1]; [1]Osaka University, Inst. of Scientific & Industl. Rsrch., Mihogaoka 8-1, Ibaraki, Osaka 567-0047 Japan

Structural integrity of circuits is greatly dependent on interfacial microstructure. In this paper, the current status on various interfaces appearing both in lead-free soldering and in conductive adhesive joining is briefly reviewed. This paper will present the recent data on interfaces in SMT primarily by TEM. Most of Sn alloys involving pure Sn, Sn-Ag, Sn-Bi or their ternary alloys form two intermetallic compounds, Cu6Sn5 and Cu3Sn. The former is much thicker than the latter and their interface integrity is strongly influenced by the Cu6Sn5 layer. The Sn-Zn alloy only forms different intermetallic compounds at the interface with Cu. Plating such as Ni also influences interface integrity. Conductive adhesives, i.e., Ag-epoxy, react with a metal substrate at elevated temperatures. The heat-resistance of the interface is greatly influenced by the presence of Sn plating on a substrate due to the preferential diffusion of Sn to the conductive adhesive layer. From those reaction mechanisms, an ideal interface structure will be discussed.

4:50 PM

Coarsening of the Ternary Eutectic in the Tin-Silver-Copper System: *Sarah Louise Allen*[1]; Michael R. Notis[1]; Richard R. Chromik[1]; Richard P. Vinci[1]; [1]Lehigh University, Dept. of Matls. Sci. & Eng., Whitaker Lab., 5 E. Packer Ave., Bethlehem, PA 18015 USA

The response of materials used as microelectronics solders to different thermal conditions is of great interest as changes in microstructure, for example coarsening, can have a significant effect on the mechanical properties and hence the reliability of the solder joint. The eutectic tin-silver-copper lead-free solder alloy has been isothermally annealed at a number of different temperatures and at different times in order to ascertain how the ternary eutectic structure coarsens. Cast and directionally solidified alloys at the ternary eutectic composition have been examined by SEM and x-ray mapping to study the changes in microstructure, and quantitative image analysis has been used to obtain the effective activation energy of the coarsening process. It has been observed that the two intermetallic phases present in the eutectic, Cu_6Sn_5 and Ag_3Sn, coarsen at different rates and both migrate to grain boundaries during the annealing process.

5:05 PM Invited

Effects of Intermetallic Morphology at Metallic Particle/Solder Interface on Dynamic Mechanical Properties of Sn-Ag Solder Joints: *H. Rhee*[1]; K. C. Chen[2]; J. G. Lee[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA; [2]California Polytechnic State University, Matls. Eng. Dept., San Luis Obispo, CA 93407 USA

Dynamic thermal/mechanical analysis (DTMA) using a Solids Analyzer and creep tests with a miniature frame were carried out to investigate the effects of morphology of intermetallic compound reinforcements on mechanical properties of Sn-Ag solder joints under simulated service conditions. These intermetallic reinforcements with different morphologies resulted around metallic particles added to the solder depending on reflow profile used. Such particle reinforced composite solders were prepared by mechanically dispersing Cu, Ni or Ag particles into the eutectic Sn-3.5Ag solder paste. The morphology, size and distribution of the intermetallic phases at particulate/solder interface of the solder joints were also characterized metallographically. As a result, a better understanding of the role of the morphology of the reinforcing phases on the mechanical properties of the composite solder joints is obtained. Acknowledgement: Work supported by National Science Foundation under grant NSF DMR-0081796.

Science and Technology of Magnetic and Electronic Nanostructures: Semiconductor Nanostructures

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee

Program Organizers: Ramamoorthy Ramesh, University of Maryland, Department of Materials and Nuclear Engineering, College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Tuesday PM — Room: 15A
March 4, 2003 — Location: San Diego Convention Center

Session Chair: Darrell G. Schlom, Pennsylvania State University, Dept. of Matls. Sci. & Eng., University Park, PA 16802 USA

2:00 PM

Effects of Microstructures of Mixed III-V Layers on Electronic and Optical Properties: *Subhash Mahajan*[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., PO Box 876006, Tempe, AZ 85287-6006 USA

It will be demonstrated that atomic species in mixed III-V layers, residing on the same sub-lattice and differing in their covalent tetrahedral radii, are not randomly distributed within the layers. Two types of deviations from randomness are observed: (1) phase separation, and (2) atomic ordering. Both of these microstructural features evolve at or near surfaces during the layer growth. Phase separation reduces carrier mobility and ordering affects band gap. In addition, it will be shown that the reliability of light emitting devices containing mixed layers is enhanced because non-radiative recombination glide and climb is difficult in the presence of phase separation and ordering.

2:45 PM

Morphological Evolution of Compositionally Modulated Nanowires: *Timothy D. Sands*[1]; [1]Purdue University, Sch. of Matls. Eng. & Sch. of Electl. & Compu. Eng., MSEE Bldg., Northwestern Ave., W. Lafayette, IN 47907-1289 USA

Whereas equilibrium considerations may present theoretical limits to heterostructure design in thin films, the equilibrium state represents a more practical design limitation for nanowires. The relatively rapid evolution toward local equilibrium in a nanowire heterostructure can be utilized to an engineering advantage in the design of new device materials. In this talk, modeling of local equilibrium states uniquely accessible in the nanowire format is highlighted. In the case of longitudinal lattice-mismatched heterostructures, it will be shown that elastic relaxation normal to the nanowire axis expands the range of lattice-mismatched materials that may be grown coherently in mechanical equilibrium. In a second example, it will be demonstrated that composition modulation along an as-grown nanowire can be used to direct the morphological evolution toward the fabrication of monosized capsules, spheres or periodic constrictions with spatial periods less than the minimum wavelength for Rayleigh breakup.

3:30 PM

Preparation and Properties Nanocrystals and Nanocrystal Superlattices: Building with Artificial Atoms: *Christopher B. Murray*[1]; [1]IBM Corporation, T. J. Watson Rsrch. Ctr., 1101 Kitchawan Rd., Rte. 134, PO Box 218, Yorktown Heights, NY 10598 USA

Synthetic chemistry allows to production nanometer scale structures which are uniform size to + or - one lattice constant while controlling crystal shape, structure and surface passivation. We combine a high temperature solution phase synthesis with size selective processing techniques to produce organically passivated magnetic and nanocrystals with size distributions less than 5%. These nanocrystals then form the basis for a combined structural and magnetic study of the evolution nanocrystal properties with size. These monodisperse nanocrystals self-organize during controlled evaporation to produce 2D and 3D superlattices (colloidal crystals, opals). The nanocrystals resemble ìartificial atomsî sitting on regular close-packed superlattice sites, each separated by a selected organic spacer. The inter-particle spacing can be varied from intimate contact up to ~40≈ separation. The superlattices retain and enhance many of the desirable mesoscopic properties of individual nanocrystals and permit the first systematic investigation of new collective phenomena. Our goal is to study the properties of both the dispersed nanocrystals and assemblies as all major structural parameters are varied (composition, size, and spacing). Procedures have been developed for Co, Ni, and FePt magnetic nanocrystals as well as for CdSe and PbSe semiconductor quantum dots. Recent explorations of magnetic recording the transport phenomena in magnetic nanocrystal superlattices will be discussed as well as optical studies of the semiconductor nanosctructures. Progress in the development of techniques to pattern nanocrystal superlattices, which will be essential to the fabrication of devices incorporating these molecular-scale building blocks will also be highlighted.

4:15 PM
Metal/Semiconductor Nanowire Nanocontacts: *Suzanne Mohney*[1]; Ahmad Mohammad[1]; Soham Dey[1]; K. K. Lew[1]; Joan Redwing[1]; Marco Cabassi[1]; Theresa Mayer[2]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Eng., 109 Steidle Bldg., University Park, PA 16802 USA; [2]Pennsylvania State University, Dept. of Electl. Eng., University Park, PA 16802 USA

Semiconductor nanowires (NWs) are presently the focus of intense research owing to their potential for use in both interconnects and nanoelectronic devices. Ohmic contacts to the NWs will be required for many of these devices to realize their full potential. However, the restricted geometry of the nanocontacts can be expected to influence current transport as well as interfacial reactions between the metal and the semiconductor. Our group has recently fabricated reacted metal silicide/Si NWs in porous alumina membranes using electrodeposition for the metal NW segments and vapor-liquid-solid (VLS) growth for the Si segments. In this presentation we will discuss an overview of our fabrication method and the results of an investigation into the structural and electronic properties of these nanostructures.

Surface Engineering in Materials Science - II: Coatings and Properties

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA; A. Agarwal, Plasma Processes, Inc., Huntsville, AL 25811-1558 USA; Narendra B. Dahotre, University of Tennessee-Knoxville, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; C. Suryanarayana, University of Central Florida, Mechanical, Materials & Aerospace Engineering, Orlando, FL 32816 USA

Tuesday PM — Room: 7A
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: C. Suryanarayana, University of Central Florida, Mechl., Orlando, FL 32816 USA; J. DeHosson, University of Groningen, Dept. of Applied Physics, Groningen 9747 AG Netherlands

2:00 PM Cancelled
Reaction Enthalpies as Selection Criteria for Metal Coatings: *Newton Eng Kin Ooi*[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287 USA

2:20 PM
Surface Morphology and Roughness Evaluation of Ni-P-W Coatings: *Fan-Bean Wu*[1]; Jenq-Gong Duh[1]; [1]National Tsing Hua University, Matls. Sci. & Eng., 429R, 4th Eng. Bldg., Sec. II, Guang-Fu Rd. #101, Hsinchu 300 Taiwan

Surface morphologies of the electroless plated Ni-P and Ni-P-W coatings were investigated by Atomic Force Microscopy (AFM). Both coatings exhibited nodular feature with an averaged nodule size of 2-3 mm. The roughness of the coatings evaluated from the AFM image was highly dependent on the acquisition area. The roughness was as low as 2 nm with a scan area of 0.25 mm2, while 24 nm was determined to be a reliable data under sufficient surveying area wider than 25 mm2. The sine profile was adopted as a model of nodular feature to simulate the roughness calculation. The roughness variation reduced significantly for acquisition region larger than one wavelength. The critical acquisition region of one wavelength, corresponding to twice of a nodule diameter, was then determined for a nodular surface on the electroless plating.

2:40 PM
Tribological Performance of Titanium Carbide & Gr Co-Deposited Thin Films: *Frank Mark Kustas*[1]; Jennifer Sinchak[2]; Jinhui Zhou[2]; Brajendra Mishra[2]; [1]Engineered Coatings, Inc., PO Box 4702, 15422 Winterleaf Ct., Parker, CO 80134 USA; [2]Colorado School of Mines, Adv. Coating & Surface Eng. Lab., 1500 Illinois, Golden, CO 80401 USA

Thin films of titanium carbide (TiC) and graphite (Gr) were fabricated using unbalanced magnetron co-deposition using either a titanium (Ti) or TiC target with Gr strips. The intent was to fabricate composite films with a ceramic matrix for high hardness and wear resistance and a solid lubricant for low friction. Structural and composition investigations were performed using X-ray diffraction and X-ray photoelectron spectroscopy, respectively. Coating properties, scratch adhesion, cracking from indentation with a diamond indenter, nanohardness, galling resistance, and simulated fretting resistance were measured. Films prepared from targets with more Gr had a larger quantity of carbon (C)-C and C-hydrogen (H) bonding (amorphous (a)-C). Nanohardness measurements showed reduced hardness for films with a higher percentage of the a-C constituent. Cracking resistance appears to be higher for films prepared from the TiC & Gr compared to films deposited from the Ti & Gr target.

3:00 PM Break

3:20 PM
Cathodic Deposition of Hydroxyapatite without H2 Evolution: *Zhongwei Zhao*[1]; [1]Central South University, Col. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

Cathodic electrodeposition is a talent process for preparing hydroxyapatite (HAp) coatings on titanium. But dense and adhesive film can not be obtained by transitional method owing to the fact that vigorously evolution of H2 bubble, a product of the reaction 2H2O+2e=2OH-+H2?™, obstruct the nucleation and growth of calcium phosphate crystal. Commonly, thermal treatment of the product at high temperature is necessary for increasing the strength of bioactive film. When H2O2 is added to the electrolyte, there is no H2 evolution on cathode since the dominating electrochemical reaction changed into H2O2+2e=2OH-. The HAp coatings deposited became dense and adhesive.

3:40 PM
Plasma Assisted Coatings on Porous Materials: *Sharmila M. Mukhopadhyay*[1]; Pratik Joshi[1]; Saswati Datta[2]; John MacDaniel[2]; [1]Wright State University, Mechl. & Matls. Eng., 3640 Colonel Glen Hwy., Dayton, OH 45435 USA; [2]Procter & Gamble, CETL, West Chester, OH 45069 USA

A five-layer stack of hydrophilic (water absorbent) filter paper was taken as the model porous solid that was made hydrophobic (water repellent) by plasma treatment. Influence of process parameters and monomer chemistry on coating effectiveness and plasma permeation was investigated. Water-drop-absorption time can be used as the initial measure of coating effectiveness. For a quantitative understanding, X-ray Photoelectron Spectroscopy (XPS) was used to analyze the surface chemistry of each surface in the stack and correlated with the water contact angle of that surface. It is clear that by adjusting plasma parameters, it is possible to create a porous film that is hydrophobic on one side and hydrophilic on the other. The precise control of coating penetration into inner layers depends mainly on treatment time and monomer chemistry, rather than on plasma pressure and power. Four types of plasma from different fluorine-containing monomers (TDFO, PDFOA, PFDD and PFMCH) have been compared. All are equally effective in coating the ìexternalî surfaces of the stack, but the extent of permeation of the plasma into the inner layers varies with monomer structure. PDFOA produces more penetrating plasma compared to other molecules.

4:00 PM
Oxidation Behaviors of Fe-Mn-Al Alloys with Electroless Nickel and Aluminizing Coatings: Jyh-Wei Lee[1]; *Jenq-Gong Duh*[2]; [1]Tung Nan Institute of Technology, Dept. of Mechl. Eng., #152, Sec. 3, Pei-Shen Rd., Taipei County 222 Taiwan; [2]National Tsing Hua University, Dept. of Matls. Sci., Rm. 429, Eng. Bldg. #4, 101, Sec. 2, Kuang-Fu Rd., Hsin-Chu 300 Taiwan

Surface modification is a technique to introduce foreign materials onto the surface of engineering component to improve its service behavior and to increase its lifetime. Aluminization and electroless nickel coating techniques were employed and combined to deposit iron-aluminide, electroless nickel and nickel-aluminide on the surface of Fe-31Mn-5.9Al-0.95C alloy, respectively. Phases and microstructure of individual coating layer were investigated with X-ray diffractometer and electron probe microanalyzer. High temperature oxidation behaviors of coatings were evaluated by the 800°C oxidation test. Owing to the spallation of nickel oxide layer, the oxidation performance of electroless nickel coated alloy was not sufficient. However, weight gains of iron-aluminide and nickel-aluminide coated alloys were very low due to the formation of protective Al2O3 layers on surfaces. It is concluded that the oxidation resistance of Fe-Mn-Al alloy is greatly improved by the iron-aluminide and nickel-aluminide coatings on the surface derived from aluminization and electroless nickel coating combinations.

4:20 PM

Development of Surface Engineered Coatings for Dies Used in Material Processing: *Dalong Zhong*[1]; Stacey Carrera[1]; Arron Michael Peters[1]; Olympia Salas[1]; Brajendra Mishra[1]; John Moore[1]; [1]Advanced Coatings and Surface Engineering Laboratory, Colorado Sch. of Mines, 1500 Illinois St., Golden, CO 80401 USA

This paper will describe the development of surface engineered coatings for die materials that have applications in glass molding, aluminum pressure die-casting and metal stamping. The design concept involves the development of a ìcoating systemî that has: (a) a ìworking layerî that offers a non-wetting, wear and oxidation resistant surface that interfaces with the material to be formed (i.e., liquid glass or aluminum, solid metal); (b) an intermediate multi-layer or compositionally graded layer that will help minimize the thermal and residual stresses that are generated during the process or shot cycling; and (c) a thin (50-100 nm) adhesion layer that improves the adhesion of the coating system to the die surface (substrate). Finite element modeling (FEM) has been used to identify suitable candidate intermediate layers. In each case, a multi-layer coating architecture has been developed. Glass molding dies and forming tools operate in air at elevated temperatures (e.g. 600-1000∞C), and are subjected to chemically active molten glass and thermal cyclic operations. Similarly, aluminum pressure die casting dies operate typically between 150-6000C in air and need to be non-wetting with molten aluminum, while metal stamping dies need to exhibit low lubricity. This work is directed to identify optimized coating systems for dies used in glass molding, aluminum pressure die casting and metal stamping applications in an effort to improve their performance and reliability. In each case, nanostructured, multi-layer thin films have been deposited using magnetron sputtering, and functionally graded coating architectures have been developed based on these nanocomposite thin films.

TUESDAY PM

Terence E. Mitchell Symposium on the Magic of Materials: Structures and Properties: Phase Transformations and Defect Dynamics - II & Phase Transformations and Defect Dynamics - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Amit Misra, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Harriet Kung, Los Alamos National Laboratory, Materials Science & Technology Division, Los Alamos, NM 87545 USA; Stuart A. Maloy, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Michael Nastasi, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Ricardo Schwarz, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Tuesday PM   Room: 10
March 4, 2003   Location: San Diego Convention Center

Session Chairs: Ricardo Schwarz, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Ivar Reimanis, Colorado School of Mines, Metallurgl. & Matls. Eng. Dept., Golden, CO 80401 USA

2:00 PM Invited

Dislocation Model of Martensite Transformation: Steven Celotto[1]; Robert C. Pond[1]; John P. Hirth[2]; [1]University of Liverpool, Brownlow Hill, Liverpool L69 3BX UK; [2]114 Ramsey Canyon Rd., Hereford, AZ 85615 USA

Current understanding of martensitic transformations is based on the phenomenological theory of martensitic crystallography (PTMC). The object of the present work is to develop a defect model of the interface consistent with a diffusionless transformation accompanying its motion. As a necessary step towards this goal we have considered the properties of single arrays of disconnections, and compared their crystallographic attributes with predictions based on the PTMC for identical transformations. In the defect model, the lattice parameters determine the topological properties of disconnections (Burgers vector, b, and step height, h). In turn this determines the disconnection spacing, since these accommodate coherency strains on the terraces, and the habit plane orientation through the defectsí step height. Also components of b perpendicular to the terraces give rise to a rigid body rotation of the crystals away from their reference orientation. Experimental observations obtained using TEM will be shown which corroborate the defect model.

2:30 PM Invited

Martensitic Transformations in Platinum Modified Nickel Aluminide Bond Coats for Thermal Barrier Coatings: *Kevin J. Hemker*[1]; [1]Johns Hopkins University, Dept. of Mechl. Eng., Baltimore, MD 21218 USA

Thermal barrier coatings used in commercial gas turbine engines insulate the engine components from the hot gas stream. There are four main layers in a thermal barrier system: the superalloy substrate, a nickel aluminide bond coat, a thermally grown oxide (TGO) that grows on the top of the bond coat, and the ceramic top coat. Failure of the TBC is caused by spallation, which is related to the development of stresses and strains in the multi-layered system during thermal cycling. Traditionally, the two main source of stress have been considered to come from: (i) the thermal expansion misfit upon cooling and (ii) the growth of the TGO during service. Recent microstructural observations have highlighted the importance of a martensitic transformation of the bond coat that also plays an important role in governing the underlying properties of the TBC. Results of TEM, micro-probe, X-ray diffraction and microsample tensile testing will be presented and used to characterize the transformation of the bond coat from its original B2 structure to a Ni-rich L10 martensite. The attendant transformation strain and variations in the mechanical properties of the bond coat and will be shown to play an important role in determining the life of the multilayered TBC system.

3:00 PM Invited

Phase Transformations and Phase Stability in the Pu-Ga System: *Jeremy N. Mitchell*[1]; Marius Stan[2]; Daniel S. Schwartz[1]; Carl J. Boehlert[3]; Thomas G. Zocco[1]; [1]Los Alamos National Laboratory, Nucl. Matls. Tech. Div., MS G721, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [3]Alfred University, Dept. of Ceram. Eng. & Matls. Sci., 2 Pine St., Alfred, NY 14802 USA

From room temperature to melting, Pu undergoes five phase transformations that result in a maximum volume change of 20%. The fcc δ phase is stabilized at room temperature by adding small amounts of Ga. Gallium-stabilized δ Pu transforms martensitically to α¥ Pu (monoclinic), but only at low temperatures (~-100∞C). Concurrent to the early work on Pu-Ga in the U.S., Russian scientists developed a phase diagram that shows α Pu + Pu_3Ga stable at room temperature. In this paper, we will present recent experimental results that explore the onset of the δ to α¥ transformation and the thermodynamics of the various phase transformations. We will also present results of phase diagram modeling that incorporate these and previous data. Preliminary results on an internally inconsistent database suggest that α Pu + Pu_3Ga is stable at room temperature thermodynamically, but that the kinetics of the reaction δ Pu to α Pu + Pu_3Ga are extremely sluggish.

3:30 PM Break

4:00 PM Invited

Modeling of Amorphization in Network Structures: *Linn W. Hobbs*[1]; Xianglong Yuan[1]; [1]Massachusetts Institute of Technology, Rm. 13-4054, 77 Massachusetts Ave., Cambridge, MA 02139-4307 USA

The network structures of silicon, silica, silicon nitride and silicon carbide have in common corner-sharing tetrahedra, significant excess volume over close-packed structures, and the requisite structural freedom to be amorphizable by displacive radiation. Topology is important in such tetrahedrally-connected structures and both governs the range of configurations that can be adopted and may be used to characterize them. The principal tool is evaluation of a local unit known as the local cluster based on irreducible ring content. We have used a powerful combination of topology and molecular dynamics simulations to investigate point defect configurations in silicon and silicon carbide, structures of amorphized silicas, and a chemical disorder-driven glass transition in silicon carbide. Some notable findings discussed are that conventional pair-correlations derived from diffraction data cannot distinguish between well-connected silica structures with vastly different topologies, that silicon carbide is amorphizable only above a chemical disorder threshold, and that defects in amorphizing systems can be identified and reliably quantified by topology far after any fiducial reference lattice is lost.

4:30 PM Invited
Nanoscale Probing of Ferroelectric Domain Wall Structure and Dynamics: *Venkatraman Gopalan*[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., 253 MRL, University Park, PA 16802 USA

This talk will explore the local structure and dynamics of individual domain walls in ferroelectrics. Many new fascinating and unexpected discoveries relating to the presence of wide regions of strain, optical birefringence, and local fields at ferroelectric domain walls were made by Gopalan and Mitchell about 6 years ago. These are forcing us today to revisit our fundamental understanding of the role of atomic defects in ferroelectrics.

5:00 PM Invited
Pressure Effects on Flow, Fracture, and Processing of NiAl: *John J. Lewandowski*[1]; Robert Margevicius[2]; Joseph D. Rigney[3]; [1]Case Western Reserve University, Dept. Matls. Sci. & Eng., Cleveland, OH 44106 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [3]GE Aircraft Engines, Evandale, OH USA

The effects of changes in the dislocation substructure on the strength and toughness of stoichiometric polycrystaline NiAl have been determined. The dislocation substructure was introduced via either exposure to high hydrostatic pressure, or via thermomechanical processing by hydrostatic extrusion of polycrystalline NiAl at low temperature. The material tested exhibited a sharp yield point in annealed material. Pressurization removed the yield point without measurably affecting the ductility. Testing with superimposed pressure significantly increased the ductility and changed the fracture mode. Hydrostatic extrusion increased both the strength and toughness due to the beneficial substructure produced. Subsequent annealing experiments were conducted in order to determine the effects of removal of the beneficial dislocation substructure on the resulting strength and toughness.

5:30 PM
Friction Stir Processing for Energy Efficient Surface Modification: *Glenn J. Grant*[1]; Richard W. Davies[1]; Darrell R. Herling[1]; [1]Pacific Northwest National Laboratory, Matls. Procg. & Performance, 902 Battelle Blvd. P8-35, Richland, WA 99352 USA

Friction Stir Processing (FSP) is a process by which a spinning, non-consumable tool is plunged into a material and translated across the surface, leaving behind a severely plastically deformed region. This is a solid-state process that can be applied to a wide range of materials to potentially improved wear, corrosion, or mechanical properties of the near surface region. FSP also has the potential to modify near surface microstructure or composition to create functionally graded structures, without multiple process steps. Research at the Pacific Northwest National Laboratory has focused on using FSP in two main areas: 1) to create refined grain structures to modify the near surface regions of castings and extrusions for improved microstructure, and 2) to use FSP to stir ceramic particulate into the surface of aluminum alloys to create robust and thick reinforced region for wear applications. The Friction Stir Process has the potential to consume less energy, and produce fewer emissions than many current material processing technologies.

The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research: Iron and Iron Alloys

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Japan Institute of Metals, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Julia R. Weertman, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Katherine T. Faber, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Morris E. Fine, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Wayne King, Lawrence Livermore National Laboratory, San Ramon, CA 94583-2496 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Ben Mori, Tokyo 168-0081 Japan

Tuesday PM — Room: 9
March 4, 2003 — Location: San Diego Convention Center

Session Chairs: K. Maruyama, Tohoku University, Dept. Matls. Sci., Sendai 980-8579 Japan; C. G. Park, Pohang University of Science & Technology, Ctr. for Adv. Aeros. Matls., Pohang 790-784 Korea

2:00 PM
Structure of a Stress-Induced Martensite Plate and its Formation Stress: *T. Mori*[1]; Edward Oliver[1]; Mark Daymond[1]; Philip Withers[2]; [1]Rutherford Appleton Laboratory, ISIS Fac., Bldg. R3, Chilton, Didcot OX11 0QX UK; [2]The University of Manchester, Manchester Matls. Sci. Ctr., Manchester M13 9PL UK

A twinned martensite is usually assumed to form with a structure containing fixed fractions of two Bain Correspondence Variants, such that the elastic energy vanishes. Contrary to this assumption, the present work demonstrates that the structure and, consequently, the average transformation strain of a martensite plate depends on external and internal stress. The total energy, consisting of chemical energy, elastic energy and the potential energy of external stress, is minimized under the conditions of constant external stress and temperature. The minimum condition gives the stress required to form the corresponding macroscopic strain due to transformation. The face-centered cubic to face-centered tetragonal transformation in Fe-Pd is used to demonstrate the method. It is shown that the external stress required to initiate martensitic transformation is lower than that predicted using the fixed structure assumption.

2:20 PM
HVEM Observation of Microstructures in Fe-Mn-Si Based Shape Memory Alloys After High Speed Deformation: *A. Sato*[1]; H. Oonishi[1]; Y. Yamaguchi[1]; S. Kumai[1]; T. Maruyama[2]; H. Kubo[3]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., 4259 Nagatsuta, Midori-ku, Yokohama 226-8502 Japan; [2]Awaji-Sangyo Company, Ltd., M&C Bldg., 2-3-13 Kanda-ogawa-cho, Chiyoda-ku, Tokyo 101-0052 Japan; [3]Kantoh Polytechnic College, 612-1 Mitake, Yokokura Oyama-shi, Tochigi-ken 323-0810 Japan

Fe-Mn-Si based shape memory alloys deformed by a super high-speed roller were examined by HVEM after various heat treatments and mechanical tests. It has been revealed that the strong iron-based shape memory alloy can be rolled down to 50% in thickness reduction by use of Cu plates covering a foil specimen. Microstructures of as rolled and annealed specimens were examined with special interest in the refinement of crystal structure. Application of a 2-1/2 D method enabled us to visualize microscopic distribution of sub-grains and distribution of misorientations that play an important role in strengthening of these alloys. Effects of introduction of second phase particles on the strengthening has also been studied for the purpose of improving the shape memory property by structural refinement.

2:40 PM
Thermography of Fatigue Damage: *Bing Yang*[1]; Peter K. Liaw[1]; Hsin Wang[2]; Liang Jiang[1]; Jiunn Yuan Huang[3]; Roang Ching Kuo[3]; J. G. Huang[4]; Doug Fieldon[1]; [1]University of Tennessee-Knoxville, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Institute of Nuclear Energy Research (INER), PO Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan 325 Taiwan; [4]Taiwan Power Company, Nucl. Oper. Dept., Taipei 100 Taiwan

An infrared (IR) thermography technique, as a nondestructive evaluation technique, was applied to investigate the fatigue damage of Reactor Pressure Vessel (RPV) Steels during 0.5 Hz, 20 Hz and 1,000 Hz fatigue testing. Five stages of temperature profiles were observed: an initial decrease of the average specimen temperature, a followed temperature hump, an equilibrium temperature region, an abrupt increase of the temperature, and a drop of temperature following specimen failure. The relationship among the temperature and fatigue behavior is discussed. Both thermodynamics and heat-transfer theories are applied to model the observed temperature variation during fatigue. The predicted and measured temperature evolutions during fatigue were found to be in good agreement. Both close from-solution and finite-element methods have been attempted to predict the temperature evolution along the specimen gage-length. The shear-band evolution of the plate specimen during fatigue has been observed and analyzed by thermography.

3:00 PM
Phase Instability and Corrosion of Alloy 22 as a High-Level Nuclear Waste Container Material: *Yi-Ming Pan*[1]; Darrell S. Dunn[1]; Gustavo A. Cragnolino[1]; [1]Southwest Research Institute, Center for Nuclear Waste Regulatory Analyses(CNWRA), 6220 Culebra Rd., San Antonio, TX 78238 USA

Alloy 22 is currently proposed by the U.S. Department of Energy (DOE) for use as the outer container of the waste package for the disposal of high-level nuclear waste. DOE will need to address the extent to which phase instability resulting from fabrication processes (i.e., welding and postweld treatments) could limit the lifetime of the Alloy 22 waste packages. In this context, recent work involved the study of alloy specimens in mill-annealed condition after thermal exposure at 870°C [1,598°F] for periods of up to 30 minutes. The effect of metallurgical stability on localized corrosion susceptibility was evaluated using corrosion tests and analytical electron microscopy measurements. Results obtained from this study indicate that thermal exposure at 870°C [1,598°F] for only 5 minutes resulted in the formation of topologically close-packed (TCP) phases at grain boundaries; however, no significant alloy depletion was detected in the matrix adjacent to the precipitates nor in the grain-boundary regions between precipitates. Nevertheless, precipitation of TCP phases was found to promote localized corrosion along grain boundaries and decrease repassivation potential in chloride-containing solutions. This abstract is an independent product of the CNWRA and does not necessarily reflect the views or regulatory position of the U.S. Nuclear Regulatory Commission.

3:20 PM

TEM Investigation of Deformation Mechanism of Iron Processed by Severe Plastic Deformation: *B. Q. Han*[1]; W. A. Chiou[1]; F. A. Mohamed[1]; E. J. Lavernia[1]; [1]University of California-Irvine, Dept. of Cheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

To understand the deformation mechanisms of nanostructured materials, the investigation of grain refinement of pure Fe was performed via severe plastic deformation (equal-channel angular pressing) at room temperature. The microstructural evolution during pressing and the deformation behavior pure Fe were investigated by transmission electron microscopy, and tensile and compressive tests, respectively. The initial grain size of annealed Fe was about 200 mm. It reduced dramatically during pressing and down to about 200-400 nm after 8 passes. In the tension test, a drop in the stress-strain curve following the occurrence of maximum strength was observed in processed Fe, which is different from strain hardening in annealed Fe. In compression test, an initial strain-hardening region followed by a no strain-hardening region was observed in ultrafine grained Fe. The strengthening mechanisms and work-softening behavior were discussed with the evolution of dislocations.

3:40 PM

The Use of EELS Elemental Mapping to Study Small Precipitates in Steels: Xinyi Y. Wang[1]; *Michael L. Jenkins*[1]; John M. Titchmarsh[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

The precipitation processes in an age-hardenable maraging steel with composition 12-Cr-9Ni-4Mo-2Cu (wt%) have been investigated using energy-filtered transmission electron microscopy and conventional high-resolution imaging and diffraction techniques. It proved possible to use EELS elemental mapping to identify and obtain information on the composition of precipitates as small as 1.5 nm. A number of different precipitate types were identified, including Cu-rich precipitates with the bcc and 9R structures, ω-Fe_7Mo_2 and η-Ni_3(Ti, Mo), and the formation sequences of these precipitates during ageing were clarified. Ni-rich precipitates tended to form adjacent to existing Cu-rich precipitates, consistent with an earlier suggestion that Cu-rich precipitates act as nucleation sites for Ni_3(Ti, Mo) precipitates. The ability of EELS elemental mapping to image Cu-rich precipitates at sizes where they are coherent with the matrix and difficult to image using conventional methods is an important factor in our ongoing investigations of embrittlement of ferritic steels under irradiation.

4:00 PM

Interaction of Coherent Nanoscale Precipitates with Screw Dislocations to Lower the Peierls Stress in Low Carbon Steels: *Morris E. Fine*[1]; Anthony Tongen[2]; Michael S. Gagliano[3]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; [2]Trinity International University, Dept. of Math./Compu. Info. Sys., 2065 Half Day Rd., Deerfield, IL 60015 USA; [3]Ondeo-Nalco, Metall., One Ondeo-Nalco Ctr., Naperville, IL 60563 USA

Several years ago Mike Meshii was interested in solid solution softening that occurs in iron and certain other metals at certain temperature and strain rate ranges. The screw dislocations in iron and other body-centered cubic metals have a high Peierls stress. Solute atoms were thought to interact with screw dislocations to locally lower the Peierls stress making it easier for them to escape the Peierls valley and produce softening. This concept was based on an early paper by Hans Weertman. Recently a low carbon steel with nanoscale precipitates was found to have a remarkably high Charpy impact fracture energy at cryogenic temperatures. The nanoscale precipitates are thought to interact with screw dislocations reducing the Peierls stress. The present paper applies some of the theoretical treatments developed to explain solid solution softening to the interaction of misfitting coherent nanoscale precipitates with screw dislocations. The activation energy for plastic flow by screw dislocations is reduced. The effects of such interactions on the flow stress-temperature-strain rate relations and the ductile to brittle transformation temperature will be discussed.

4:20 PM

Characterisation of Stress Corrosion Cracks in Stainless Steels and Nickel-Based Alloys: Sergio Lozano-Perez[1]; *Michael L. Jenkins*[1]; John M. Titchmarsh[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

The potential application of TEM for studying the morphology and chemistry of intergranular stress corrosion cracks in components fabricated from stainless steels and nickel-base alloys has been limited by difficulties in sample preparation. We have developed a novel focussed ion beam method, which has proven to be ideal for preparing cross sectional TEM specimens through such cracks. Individual cracks can be pre-selected following characterisation of the crack geometry and grain misorientation by SEM and EBSD, and the site from which the specimen is taken can be chosen with a precision of nanometres. Detailed and novel observations of the microstructure and chemistry of crack flanks and tips will be described which provide new insight into mechanisms of intergranular stress corrosion cracking.

4:40 PM

TEM Studies of Non-Equilibrium Microstructure Evolution and Phase Transformation of Fe-Cr-B-Ni-Mo Spray Coatings: *H. W. Jin*[1]; C. G. Park[1]; M. C. Kim[2]; [1]Pohang University of Science & Technology, Ctr. for Adv. Aeros. Matls., Dept. Matls. Sci. & Eng., Pohang 790-784 Korea; [2]Research Institute of Industrial Science and Technology, Pohang 790-600 Korea

The non-equilibrium microstructure evolution has been investigated by using TEM in thermally sprayed Fe-Cr-B-Ni-Mo coatings. The spray coated layer was composed of $(Cr,Fe)_2B$ boride particles and α-(Fe,Cr) matrix phase containing equiaxed nanocrystallites. The dissolution of $(Cr,Fe)_2B$ followed by the super-saturation of boron within the matrix, during the thermal spraying, could result in a brick-wall type matrix structure through the homogeneous volume nucleation of nanocrystalline α-(Fe,Cr) inside the sprayed splats. Thermal stability of these metastable phases and consequent microstructure development have also been investigated by in-situ TEM heating experiments. The sequences of thermal decomposition of the nanocrystalline matrix phase, upon exposure to high temperatures above 670K, to the final intragranular precipitation of Cr_2B borides within the coarsened α-(Fe,Cr) grains will be presented.

5:00 PM

Music, Materials, and Metaphysics: Novel Applications of Transmission Electron Microscopy: *L. E. Murr*[1]; [1]University of Texas at El Paso, Metallurgl. & Matls. Eng., 500 W. University Ave., El Paso, TX 79968-0520 USA

This paper summarizes a number of interesting and often related case studies. Nearly twenty years ago on a Caribbean cruise I vowed to find out how Caribbean steel drums create their chromatic tones. I was convinced that some intricate dislocation structures were involved. Five years ago my students and I cut notes out of steel drums and thinned them down for transmission electron microscope (TEM) examination. Some interesting examples of that work will be presented. In a study of defects in copper rod and very fine wires drawn from the rod, observations of what we called void lobed defects created by copper mist in the wire casting process drew the ire of the industry. Some interesting techniques for observing defect structures in 15-20 µm diameter wires by TEM are illustrated. Recent observations of dynamic recrystallization and associated microstructures in a range of high strain/high-strain-rate phenomena such as friction-stir welded metals and metal shaped charges provide some common processing mechanisms. Some interesting defects were observed in lattice imaging of shock consolidated $YBa_2Cu_3O_7$ superconducting monoliths some years ago and these defects in the {100} planes resemble the Pepsi-ColaÆ logo. Finally, recent efforts to collect nanoparticles in the air suggest considerable potential for respiratory health effects and demonstrate rather interesting microstructural issues about this ultra-fine particle regime.

The MPMD Fourth Global Innovations Symposium: Energy Efficient Manufacturing Processes: Innovative Sheet Forming and Net-Shape Processing

Sponsored by: Materials Processing and Manufacturing Division, *Program Organizers:* Toni G. Marechaux, National Research Council, National Materials Advisory Board, Washington, DC 20418 USA; Iver E. Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Chris Cockrill, DOE Seattle Regional Office, Seattle, WA USA

Tuesday PM　　Room: 5A
March 4, 2003　　Location: San Diego Convention Center

Session Chairs: Glenn Daehn, Ohio State University, Columbus, OH 43210 USA; Mark T. Smith, Pacific Northwest National Laboratory, Matls. Sci. & Eng., Richland, WA 99352 USA

2:00 PM Welcome

2:05 PM Invited
Electromagnetically Assisted Stamping: *Glenn S. Daehn*[1]; Jianhui Shang[1]; Vincent J. Vohnout[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Electromagnetic forming is a technique where a capacitor is charged to a voltage on the order of a few thousand volts and this energy is run through an acutator near a conductive workpiece. The rapidly changing current in the coil induces a rapidly changing magnetic field that induces eddy currents in the workpiece. In the end a significant repulsion between the workpiece and coil can be attained. Actuators of this type can be embedded in traditional stamping dies. This can produce many advantageous features. In particular strain can be produced exactly where it is needed in a part, giving the process designer unprecidented freedom. This has the practical advantages of 1) enabling the production of part shapes with high strength materials that may otherwise be impossible, 2) fewer press operations may be required to make a given part, and 3) it may be possible to completely eliminate the use of lubricants from the stamping plants. All three of these advantages have significant positive environmental and energy payoffs.

2:30 PM
Inverse Analysis of Aluminum Tubes Hydroformed Under Free Conditions and Within a Conical Die: *Ba Nghiep Nguyen*[1]; Kenneth I. Johnson[1]; Glenn J. Grant[1]; Mohammad A. Khaleel[1]; [1]Pacific Northwest National Laboratory, Richland, WA 99352 USA

In order to reduce the very costly trial-and-error practices, numerical methods predicting how the material can deform, and the hydroforming parameters are essential. This paper presents an inverse approach to tube hydroforming to efficiently predict the thickness, strain and pressure distributions for a given deformed configuration of aluminum AA6061-T4 tubes under free hydroforming conditions or within a conical die. The analysis employs a membrane finite element formulation within the framework of the deformation theory of plasticity and Hillś criterion to describe the plastic flow. Experiments on hydroforming of Aluminum tubes using a conical die or under free hydroforming conditions were also conducted to validate the model. Good comparisons of results have been found for the deformed configurations in which the bending effect is small.

2:55 PM
A Comparison of Tube-Hydroforming Experiments and Model Results Using a Numerical Process Control Method: *K. I. Johnson*[1]; B. N. Nguyen[1]; G. J. Grant[1]; M. A. Khaleel[1]; [1]Pacific Northwest National Laboratory, PO Box 999, Richland, WA 99352

The incorporation of process control logic within finite element simulations has the potential to speed the introduction of lightweight materials, such as hydroformed aluminum tubing, into automotive structures. This paper compares the deformations and strains observed in tube-hydroforming experiments with companion finite element simulations that were used to predict the optimum load paths for maximum tube deformations without wrinkling. The tests were conducted using extruded AA-6061-T4 tubes that were formed in both a conical die shape and in a free-hydroforming configuration. A numerical process control method was incorporated in the finite element simulation to predict the increments in the axial end-feed and internal pressure loads to give a constant ratio of axial-to-hoop plastic strain based on a controlled increment in the maximum equivalent plastic strain. The process controller uses the deformation theory to describe the plastic flow and the forming limit diagram to predict the onset of rupture. The end-feed and pressure loads predicted by the models were applied in the tests, and the resulting strains, deformations, and loads to failure were measured for comparison with the model predictions. The hydroforming tests show that the controller accurately predicted the end-feed and pressure load paths that gave stable deformations to the onset of wrinkling in the tube. The finite element model also gave reasonably accurate predictions of the deformed shapes and thinning that were observed in the experiments.

3:20 PM Break

3:40 PM Invited
Design of Magnetic Pulse Welding Processes: *R. Douglas Everhart*[1]; [1]Advanced Computational and Engineering Services, LLC, 13603 Fernlace Ct., Pickerington, OH 43147 USA

Magnetic pulse welding is an impact welding technique that is solid-state in nature. It consists of accelerating a work-piece up to a high velocity (typically 300 to 1200 meters/second) using a magnetic pulse. The high-velocity work-piece strikes a stationary work-piece and a metallurgical impact bond is formed. The process is identical to explosive welding (which has been used for decades) with the exception that the metal is driven with a magnetic pulse, rather than explosives. Design of a quality magnetic pulse welding process involves the consideration of several factors and is a multi-step endeavor. The design goal defines the desired impact conditions (high-velocity work-piece speed and attitude) to produce a quality weld. The characteristics of the magnetic pulse power supply have a strong influence on the welding process. Coil durability (and therefore replacement) is the number one factor for considering the economic advantage of this process over another. Finally, the work-piece tooling and set-up must be designed.

4:05 PM Invited
Clean and Efficient Metal and Powder Forming Through Magnetic Pressing Technology: *Duane Charles Newman*[1]; [1]IAP Research, Inc., 2763 Culver Ave., Dayton, OH 45429 USA

This paper will address the use of magnetic pressing technology to form metals and produce net shape component from powders. Magnetic pressing refers to a suit of processes that includes electromagnetic forming, Dynamic Magnetic Compaction (DMC) and magnetic pulse welding. The paper describes several applications of the magnetic pressing technology and discusses details of high volume production systems developed by IAP. The benefits of using magnetic pressing technology and their impact on energy efficiency will be highlighted.

4:30 PM Invited
Energy Efficient Production of Sheet Material Using Radiant Arc-Lamp Heating: *Evan K. Ohriner*[1]; John Rivard[1]; Craig A. Blue[1]; Adrian Sabau[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6083, Oak Ridge, TN 37831-6083 USA

Radiant arc-lamp heating offers the potential for direct production of melted sheet products and foil. A focussed 300 kW arc lamp provides incident radiant heat at a flux of up to 3 kW/ cm2. The melting of sheet materials of metals, intermetallic alloys and composite materials has been achieved. The process offers the advantages of: 1) high efficiency of conversion of electrical energy to absorbed incident energy, 2) the ability to transmit radiant energy through a quartz window enabling environmental isolation of processed material, and 3) processing of large areas of material at rates of the order of 10 cm2/s. Current research efforts include control of grain structure and texture in the solidified sheet and numerical modeling of melting and solidification sheet materials.

Universities Servicing Education, Research and Technology Internationally for the Aluminium and Light Metals Industries - I

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: Subodh K. Das, Secat, Inc., Coldstream Research Campus, Lexington, KY 40511 USA

Tuesday PM　　Room: 5B
March 4, 2003　　Location: San Diego Convention Center

Session Chairs: Sara Dillich, US Department of Energy, Office of Industl. Tech., Washington, DC 20585-0121 USA; Subodh K. Das, Secat Inc., Coldstream Rsrch. Campus, Lexington, KY 40511 USA

2:00 PM
Measurements of Bubble Dispersion and Other Bubble Parameters in a Gas Fluxing Unit at Alcoa Using a Capacitance Probe: *James W. Evans*[1]; Neeta Mittal[1]; Autumn Fjeld[1]; D. Corleen Chesonis[2]; [1]University of California, Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]Alcoa Inc., Alcoa Techl. Ctr., Alcoa, PA 15069 USA

A capacitance probe has been developed at Berkeley that is intended to measure the frequency with which bubbles pass a point in liquid aluminum. The probe is comprised of an inner conducting wire (in some cases two wires) that is separated from the liquid aluminum by an alumina sheath. It works by detecting the change in capacitance, between the wire and the aluminum, which occurs when the aluminum moves back from the sheath as a bubble passes. Because only alumina is in contact with the melt, the probe is robust and can survive several hours in the metal. The software necessary for processing the signals from the probe is described. The probe has been used to measure bubbles in a fluxing unit at the Alcoa Technical Center and the difficulties in doing so are described. Results are presented and their relevance to modeling and optimization of fluxing units discussed. Research cofunded by DOE Energy Efficiency and Renewable Energy.

2:20 PM
Surface Behavior of Aluminum Alloys Deformed Under Various Processing Conditions: *Wojciech Z. Misiolek*[1]; William H. Van Geertruyden[1]; Paul T. Wang[2]; [1]Lehigh University, Inst. for Metal Forming, 5 E. Packer Ave., Bethlehem, PA 18015 USA; [2]Alcoa Technical Center, Process Tech. Div., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

The fundamentals of surface defect formation in extruded and rolled 6xxx aluminum alloys are not yet completely understood. These defects include peripheral coarse grain (PCG) structure, spangled surfaces, surface tearing, heat checking and pick-up. The objective of this paper is to understand the origins and mechanisms of the formation of surface imperfections for peripheral coarse grain (PCG) structure as the first step to understand surface behavior of aluminum alloys deformed under various processing conditions. This paper will include results of compression, rolling, and laboratory scale extrusion tests, in which deformation parameters are closely controlled. The deformed material is characterized to understand the influence of processing conditions and alloy chemistry on surface defect formation using traditional metallography and electron backscatter diffraction techniques. The goal is to use the results to generate a material response model that can predict these imperfections and help prevent their occurrence.

2:40 PM
Microstructure and Texture Evolution of Continuous Cast and Direct Chill Cast AA 5052 Aluminum Alloy During Annealing: *Jiantao Liu*[1]; *Xiangming Cheng*[2]; Tongguang Zhai[1]; James G. Morris[1]; [1]University of Kentucky, Dept. of Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506-0046 USA; [2]SECAT, Inc., 1505 Bull Lea Rd., Lexington, KY 40511 USA

In an effort to understand the differences in recrystallization behavior between continuous cast (CC) and direct chill (DC) cast aluminum alloys that potentially lead to differences in formability, investigation was performed on the recrystallization microstructures and textures of industrially produced hot bands of CC and DC cast AA 5052 aluminum alloy (Al-2.4%Mg) during subsequent annealing procedures. Macrotextures, determined by X-ray diffraction pole figures, were analyzed using three-dimensional orientation distribution functions (ODFs). The electron backscatter diffraction (EBSD) technique was adopted to study microtextures and mesotextures. The effects of annealing temperature, annealing time and cold rolling reduction on texture evolution during annealing process were studied. Both microstructures and textures during annealing are compared between the CC and DC materials.

3:00 PM
Reduction of Annealing Times: Development of a Model for Recrystallization in Hot Deformed Aluminum: *Anthony D. Rollett*[1]; Mohammed H. Alvi[1]; Hasso Weiland[2]; Jaakko Suni[2]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Wean Hall 4315, Pittsburgh, PA 15213 USA; [2]Alcoa Inc., Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

The kinetics of recrystallization are being studied with respect to crystallographic orientation (texture) in hot deformed aluminum. Two alloys, 1005 and 5005 and included in the study which is aimed at reducing annealing times for the processing of aluminum alloy sheet and plate. Results will be described for the kinetics and microstructural characteristics of recrystallization after hot deformation. When coupled with conventional methods such as hardness measurement, use of automated electron back-scatter diffraction (EBSD) in the scanning electron microscope (SEM) affords a powerful tool for quantifying the variation in behavior as a function of orientation. Results will be described from annealing of as hot-rolled plate, and material subjected to hot deformation under a variety of temperatures and strain rates.

3:20 PM
Development of Integrated Methodology for Thermomechanical Processing of Aluminum Alloys: *Ben Q. Li*[1]; David Field[1]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Sloan 207, Pullman, WA 99164 USA

This seminar discusses a research project on the development of a finite element-based integrated mechanical and microstructural model for process understanding and design sensitivity analyses for thermomechanical processing of aluminum alloys and the validation of the integrated model through bench-mark experiments. The research effort is being carried out in collaboration with researchers at Alcoa. We have made significant progress since it started last August. In essence, a finite element model has been developed, which has incorporated both the total formulation and rate formulation for large nonlinear deformation associated with thermomechanical processing of aluminum alloys. The finite element model development is based on our in-house finite element code, which will provide flexibilities required to integrate with complex constitutive relations describing the microstructure development during large deformation processes. Numerical simulations have been carried out for single crystal plasticity and poly-grain crystal plasticity for aluminum alloys. Channel die experiments have been carried out with aim to develop a reliable constitutive relation that describes the microstructure evolution during plastic deformation and to validate the finite element-based model predictions. Modeling strategies, numerical simulations and experimental measurements as well as the comparison of numerical results and experiments will be presented.

3:40 PM Break

3:50 PM
Microstructural Processes Occurring During Hot Deformation of AA 7055: *Blythe E. Gore*[1]; Henry A. Padilla[2]; Ian M. Robertson[1]; Armand J. Beaudoin[2]; Jonathan A. Dantzig[2]; Hasso Weiland[3]; [1]University of Illinois at Urbana-Champaign, Matls. Sci. & Eng., 1304 W. Green St., MC-246, Urbana, IL 61801 USA; [2]University of Illinois at Urbana-Champaign, Mechl. & Industl. Eng., 1206 W. Green St., MC-244, Urbana, IL 61801 USA; [3]Alcoa Inc., Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Ctr., PA 15069 USA

In hot working of 7xxx series aluminum alloys, the thermomechanical processing window is limited by problems such as hot shortness and by property requirements such as fatigue and fracture toughness. Industrial rolling conditions are such that stress must be sustained near the melting point of secondary phases, thus making it necessary to understand the underlying precipitation and deformation mechanisms involved to predict material response during processing. Evolution of precipitates and microstructural changes during high temperature in-situ TEM deformation have been studied and interactions of dislocations with grain boundaries and particles have been observed. By correlation of these findings with mechanical properties, a material description for hot deformation of AA 7055 has been formulated. This information is being used to develop a two-phase constitutive model for hot deformation processes.

4:10 PM
Process Systems Tools for Design and Optimization of Carbothermic Reduction Processes: *Dimitrios I. Gerogiorgis*[1]; B. Erik Ydstie[1]; Marshall Bruno[2]; Kai Johansen[3]; [1]Carnegie Mellon University, Dept. of Cheml. Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]Alcoa Inc., Next Generation Al. Processes, Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069-0001 USA; [3]Elkem ASA Research, PO Box 8040 Vaagsbygd, Kristiansand, N-4602 Norway

Carbothermic reduction is a nontraditional alternative process for aluminium production, based on the endothermic chemical reduction reaction occurring between aluminium oxide and carbon. This process has potential for drastic reduction of fixed and operation costs of the investment. Furthermore, it is environmentally benign and in principle significantly more energy-efficient, as the costly electrolytic ionization is effectively avoided by the direct chemical reduction pathway. This method is identified as a potential alternative to Hall-HÈroult electrochemical reduction by several studies, but its complexity still poses remarkable technical obstacles for implementation. Interdisciplinary collaboration is crucial in combining academic and industrial expertise: thus, a collaborative team has been formed among Carnegie Mellon University, ALCOA and ELKEM, in order to create an integrated modeling environment for design, control and economic analy-

TUESDAY PM

sis. It is important to underline that this systematic process development procedure is the standard in the petrochemical industry. Nevertheless, it is much less followed in the metallurgical industry, because of its traditional nature and the complexity of high-temperature metallurgical processes. The philosophy of this collaboration is to advance the carbothermic program more efficiently by introducing the use of Process Systems Engineering theory and Computer Aided Design tools. The main advantage of the systems approach to conceptual design is that process alternatives can be rapidly evaluated for economic feasibility at little cost, using detailed computer simulations. The availability of models and tools in a CAD environment thus allows us to speed up design. Furthermore, a sensitivity analysis can evaluate the importance of uncertain process parameters and provide sound guidelines for efficient experiment design, thus economizing on expenditure. Thermodynamics, kinetics, computational fluid dynamics, thermophysical material properties, large scale numerical solution and optimization strategies and economic evaluation methods are all facets of our modeling in order to evaluate the feasibility of various reactor configurations. The strategic goal here is to design a modeling environment for design of carbothermic reactors: A number of complementary objectives need to be pursued to advance towards this major goal: the creation of a framework for information storage, modeling, design and economic analysis, the development of steady state and dynamic, total (ODE) and distributed (PDE) process models, the efficient integration of commercial process simulation tools with physical property databases and the identification of sensitive variables (useful for pilot plant design and experiment design) are some of these objectives that can drastically expedite development, reducing costs and time. Presently distributed and newly acquired knowledge from ongoing work must also be integrated into a common modeling framework for rapid evaluation, optimization and experiment design.

4:30 PM

Aluminum Electrorefining in Ionic Liquids via Near Room Temperature Electrolysis: *Venkat Kamavaram*[1]; Ramana G. Reddy[2]; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., A129 Bevill Bldg., Tuscaloosa, AL 35487-0202 USA; [2]The University of Alabama, Dept. of Metallurgl. & Matls. Eng. & Ctr. for Green Mfg., 27th Ave., A129 Bevill Bldg., Tuscaloosa, AL 35487-0202 USA

Electrorefining of aluminum alloys in ionic liquids via near room temperature electrolysis was investigated. The applicability of different ionic liquids such as 1-methyl-3-butylimidazolium chloride (C_4mimCl) + $AlCl_3$ and 1-methyl-3-hexylimidazolium chloride (C_6mimCl) + $AlCl_3$ in the electrorefining process was studied. In the present study, electrorefining of two different alloys Al-356 and Al-360 alloy was performed. The effect of experimental parameters such as cell voltage, concentration of the electrolyte and temperature was studied for both the electrolytes. The physicochemical properties such as viscosities, densities, decomposition temperatures and electrochemical windows of these electrolytes were determined. The electrorefining process has the advantages of low energy consumption, low temperature and no pollutant emissions compared to the current industrial processes.

4:50 PM

A Network for Aluminum Research: *Rung Tien Bui*[1]; [1]Universite du Quebec a Chicoutimi, NSERC-ALCAN Industl. Rsrch. Chair in Proc. Eng., Chicoutimi, Quebec G7H 2B1 Canada

REGAL, a province-wide network of Quebec institutions of higher learning, R&D agencies and technology users has been set up to carry out research in aluminum fabrication and transformation technologies, and to help channel the expertises and the resources into large projects with more efficiency in terms of generating synergy and fundings, cutting-edge research, qualified personnel training, and technology transfers. This paper aims at presenting this unique experience in collaborative, mutipartite, university-based R&D. In addition to discussing the research program, the paper covers topics like dispersed inter-institutional collaboration and management, resources sharing, search for new fundings, joint graduate training, intellectual properties. Examples are drawn from activities conducted jointly with major aluminum industries worldwide.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Slags and Fluxes

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee: See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday PM	Room: Solana
March 4, 2003	Location: San Diego Marriott Hotel

Session Chairs: Kazuki Morita, The University of Tokyo, Dept. Metall., Tokyo 113-8656 Japan; Florian Kongoli, FLOGEN Technologies Inc., Matls. Tech. Dept., Montreal, Quebec H3S 2C3 Canada

2:30 PM Keynote

Slags and Fluxes in Pyrometallurgical Processes: *Florian Kongoli*[1]; [1]FLOGEN Technologies, Inc., Matls. Tech. Dept., 5757 Decelles Ave., Ste. 511, Montreal, Quebec H3S 2C3 Canada

Slags and fluxes are important interexchanged dimensions of pyrometallurgical processes. They can increase the efficiency of the smelting, converting and refining processes and improve the quality of the final products. A good slag, in one hand, should have appropriate physicochemical properties such as low liquidus temperature, optimal viscosity, maximum ability to attract undesirable elements, minimum potential of attracting valuable elements, etc. A good flux, on the other hand, when properly used, can considerably improve the physicochemical properties of the slag since by modifying its chemical composition it can decrease the liquidus temperature, improve viscosity etc. Consequently, the choice of a good slag and the corresponding fluxing strategy has become indispensable in most of industrial processes. However, the properties of multicomponent slags as well as the effect of several fluxes used in practice today are known only empirically. Sometimes they have been globally asserted without taking into account the characteristics of individual processes, the positioning of the initial slag composition or the particularities of certain laboratory procedures used to assert these effects. In the todayís reality of frequent changes in the composition of the raw materials and that of the fluxes themselves, in the existing or new developing technologies, the quantification of the physicochemical properties of the multicomponent slags and the effect of fluxes becomes indispensable. This paper reviews the work carried out from the authorís group during the last 15 years on the quantification of the physicochemical properties of multicomponent slags and on the effect of fluxes in several smelting and converting processes in close relation to individual characteristics of these processes. Several examples have also been given in order to demonstrate the fact that when taken outside the context some fluxes can become in fact anti-fluxes.

3:05 PM

Phase Diagram of CaO-FeOx-Cu2O Slag Under Copper Saturation: *Yoichi Takeda*[1]; [1]Iwate University, Fac. of Eng., Ueda 4-3-5, Morioka 020-8551 Japan

The CaO-FeOx-Cu2O slag system has been practically applied to continuous converting of copper, and also has potential for refining of copper. Liquidus lines of the slag system under copper saturation and phase stability diagram for the existing compounds are presented. Slag and copper metal were melted in a magnesia crucible. Liquidus composition was determined by chemical analysis of slag sample saturated solid phase. The temperatures of invariant equilibria were investigated by thermal analyses. Oxygen potentials on the liqudus lines, the univariant and invariant equilibria were extrapolated from the relation between slag composition and oxygen potential, that relation was confirmed in separate experimental work.

3:30 PM Invited

Some Aspects of Calcium Ferrite Slags: *Sharif Jahanshahi*[1]; [1]CSIRO Minerals, Clayton, Saint-Petersburg, Victoria 3168 Australia

Calcium ferrite slags are often associated with Professor Yazawa due to his pioneering work on thermodynamics of this type of melts. Over the past years CSIRO has invested some effort in studying physico-chemical properties of such melts to fill in some of the gaps in our knowledge and explore opportunities for application of the understanding developed. These investigations cover thermodynamics, transport properties of calcium ferrite based slags as well as the kinetics of reactions of such slags with gases or refractories. The present paper provides an overview of some of these studies and highlights some of the interesting behavior of such melts.

4:00 PM

A Counter-Flow Batch Process for Refining Copper Using Sodium Carbonate Slags: *Hiroshi Hashimoto*[1]; Mitsuru Tanahashi[1]; Hong Yong Sohn[2]; Chikabumi Yamauchi[3]; Kunihiko Takeda[1]; [1]Nagoya University, Dept. of Matls. Sci. & Eng., Grad. Sch. of Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan; [2]University of Utah, Dept. of Metallurgl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA; [3]Chubu University, Dept. of Mechl. Eng., Sch. of Eng., 1200 Matsumoto-cho, Kasugai, Aichi 487-8501 Japan

For the application of sodium carbonate slag treatment to product high purity copper on an industrial scale, the counter-flow batch operation, which can reduce the amount of utilized slag, was proposed from a viewpoint of a post-treatment of slag. Based on the distribution ratios of several impurities between the slag and molten copper determined previously, the effectiveness of this operation is discussed using a mathematical model and confirmed experimentally. According to the model, the slag consumption to remove Sb to a required level in case of the counter-flow batch operation is calculated to be around 30% of that in case of a ordinary batch operation, where pure sodium carbonate is used as a slag at every stage.

4:25 PM Break

4:35 PM Invited

Dissolution Mechanism and Solubility of Chlorine in the Oxide Melts: *Kazuki Morita*[1]; Taro Hirosumim[1]; Makoto Miwa[1]; [1]The University of Tokyo, Dept. Metall., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656 Japan

Ecological problems have recently been treated in iron- and steel-making processes, as is seen in the development of waste plastic injection into a blast furnace in Japan. However, we have to develop more effective and more extensive utilization of such a high temperature mass production system for the waste management. At present, waste plastic containing chlorine such as polyvinyl chloride (PVC) cannot be treated due to the problem of dioxine and HCl generation as well as the erosion of refractories. In order to develop the waste treatment system for the materials containing chlorine, its behavior in high temperature furnaces must be predicted and the evaluation of chlorine gas absorption into slags becomes essential, which is also very important for the practical incineration process. However, no data are available regarding the thermodynamic properties of chlorine in molten slags, because it has not been treated as impurities in molten iron such as S, P and N. In the present study, thermodynamic properties of chlorine in the $CaO-SiO_2-Al_2O_3$, $Na_2O-SiO_2-Al_2O_3$, $CaO-SiO_2-Al_2O_3-Na_2O$, $CaO-SiO_2-FeO$ and $CaO-SiO_2-Al_2O_3-FeO$ slags have been investigated in the present study. The experiments were carried out using gas-slag equilibrium, controlling both PO_2 and PCl_2 simultaneously ($PO_2=10^{-6}-10^{-19}$ atm, $PCl_2=10^{-6}-10^{-13}$ atm at 1673-1748K), and the solubility of chlorine in the 40mass%CaO-40mass%SiO_2-20mass%Al_2O_3 slag was found to vary in proportion to $PO_2^{-1/4}$ and $PCl_2^{1/2}$. Accordingly, the chloride capacity (C_{Cl-}), which represents the ability of slags to absorb chlorine, has been defined; The C_{Cl-} values were observed to increase with increasing slag basicity and temperature, and showed a reasonable relationship with that of $C_{S^{2-}}$.

5:05 PM Invited

Selection of Slag Composition and Structure for Autogenous Smelting Process to Produce White Metal Using the Basic Developments of Prof. A. Yazawa: *V. M. Paretsky*[1]; A. V. Tarasov[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals ìGintsvetmetî, 13 Acad. Korolyov St., Moscow 129515 Russia

The basic work conducted by Prof. A. Yazawa has demonstrated that in order to ensure single-stage white metal production by autogenous smelting of copper sulfide raw materials, it is most favorable to use highly basic slags, in particular oxide systems CaO-FeO-Fe2O3-SiO2. The main advantage of this system is its homogeneity at normal smelting temperatures (1200-1400∞C) and within the common ranges of calcium-to-iron ratios under high partial oxygen pressures. Ferrite-calcite slags proposed by Prof. A. Yazawa and his followers found practical use for the first time in the well-known Mitsubishi process. In order to provide a more rigorous substantiation of the selection of the slag composition for the autogenous smelting to produce white metal, special studies have been conducted in the Gintsvetmet Institute to investigate the composition and structure of slags produced on a semi-commercial scale when testing the KFP and FBP processes for white metal smelting, as well as the slag obtained in a full-scale KFP furnace at the Almalyk copper smelter. These studies were conducted using mineralogical microscopy techniques, a Cameca microprobe and nuclear gamma-resonance (NGR) spectroscopy. Studies were carried out using solidified and quenched slags with different cooling rates: 103∞C/sec (bar sample) and 10^6∞C/sec (superfast quenching by spinning-disc method). In the latter case, the structure of liquid slag was preserved (super-cooled liquid) permitting more definite assessment of the true structure of slag in molten state. The NGR-spectra obtained have indicated that: ∑ bar sample (quenching rate of 10^3∞C/sec) characterizes an intermediate state between liquid and solidified slag (60% fyalite and 40% vitreous phase); ∑ two types of positions occupied by Fe2+ ions have been identified: tetrahedral (A-positions) and octahedral (B-positions); it should be pointed out that Fe2+ ions occupy preferably positions in octahedral vacancies, while Ca2+ ions occupy only octahedral vacancies and Si4+ ions only tetrahedral vacancies; ∑ with an increase in the Ca2+/Fe2+ ratio the proportion of Fe2+ ions in A-positions increases and that in B-positions decreases resulting in a lower probability of replacement of Fe2+ ions with Cu2+ ions which are located only in B-positions; ∑ an increase in the SiO2 concentration in slag results in occupation of A-positions with Si4+ ions leaving vacant B-positions for Fe2+ ions, and as a consequence, increasing the probability of copper dissolution in slag; ∑ the total proportion of Fe2+ ions in molten slag increases with a higher concentration of CaO in slag. The investigations conducted have demonstrated why production of high-grade matte or white metal is associated with lower loss of copper in slag in a series: silicate slag/silicate-calcium slag/ferrite-calcium slag.

5:35 PM Invited

Ferrous Calcium Silicate Slags in Direct to Blister Flash Smelting: *Asmo Vartiainen*[1]; Ilkka V. Kojo[2]; Cesar AcuÒa Rojas[3]; [1]Outokumpu Research Oy, Pori 28101 Finland; [2]Outokumpu Technology, Espoo 02201 Finland; [3]Codelco, Chuquicamata Chile

Background of Direct-to-Blister Flash Smelting is discussed; especially slag chemistry relating to copper solubility, slag fluidity and impurity behavior. In December, 2000, Mini-pilot Flash Smelting test runs were carried out at Outokumpu Research Oy to produce blister copper from Chilean concentrate and white metal and from a mixture of them. Tested slag types were iron silicate slag, ferrous calcium silicate slag and calcium ferrite slag with different slag compositions and different concentrate/white metal ratios. The test runs were successful. The fluxing effect of copper oxide was clearly demonstrated and ferrous calcium silicate slags remained fluid on a very wide liquidus range. The distribution of arsenic between ferrous calcium silicate slag and blister copper is much higher than between iron silicate slag and blister copper. This successful test run with the new slag type offer new possibilities in Direct-to-Blister process design.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: Copper I

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee: See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday PM	Room: Santa Rosa
March 4, 2003	Location: San Diego Marriott Hotel

Session Chairs: Ron M. Sweetin, Mitsubishi Materials Corporation, Oakville, Ontario L6J 4B2 Canada; Mineo Hayashi, PT. Smelting, Gresik E. Java 61151 Indonesia; Motoo Goto, Port Kembla Copper Pty. Ltd., Port Kembla, NSW 2505 Australia

2:30 PM Keynote

Recent Advances in Modern Continuous Converting: *Motoo Goto*[1]; *Mineo Hayashi*[2]; [1]Port Kembla Copper Pty., Ltd., Military Rd., Port Kembla, NSW 2505 Australia; [2]PT. Smelting, Desa Roomo, Kecamatan Manyar, Gresik, E. Java 61151 Indonesia

Today, Pierce-Smith converters, coupled together with various types of smelting units, are used in most of the worldís smelters. However, due to the ìbatchî processing nature, and problems associated with fugitive emissions during the transfer of melt and blowing stages, this arrangement will unlikely be adopted in future smelters. Modern day alternative processes include the Mitsubishi Process, Outokumpu Flash Smelting and Flash Converting Process, the Noranda Process, and the Ausmelt Process, but only the Mitsubishi Process is a ìtruly continuousî smelting and converting operation, producing a constant flow of blister without tapping, and is economically proven by long term commercial operations. This paper reviews recent advances in modern continuous converting technologies, with a special section devoted to operation of the ìfirst standalone Mitsubishi C-furnaceî coupled together with a Noranda Reactor, at Port Kembla Copper Pty. Ltd. in Australia.

3:00 PM

Development of New Copper Continuous Converter: *Yasuo Ojima*[1]; *Yasuhiro Kondo*[1]; *Kazunori Kawanaka*[1]; Keisuke Yamamoto[1]; [1]Sumitomo Metal Mining Company, Ltd., Niihama Rsrch. Labs., 17-5 Isoura-cho, Otu 145-1 Funaya Saijo, Niihama, Ehime 792-0002 Japan

New copper continuous converting process was investigated to evaluate the possibility to be applied for the commercial operation. The tests were performed with scaling up from the laboratories crucible experiments to the pilot plant test. Bath smelting process with using top submerged lance technology was applied in these tests. Furnace shape was modified to the lengthwise cylindrical shaped one with having the settling area. The furnace monitor was put in the settling area to check inside furnace. FeOx-SiO2, FeOx-CaO and FeOx-CaO-SiO2 based slag system were used for this test to check their efficiency. The optimum slag chemistry to have good metallurgical results in this pilot test was discussed and FeOx-CaO system with adding some SiO2 was turned out to be preferable, which has not been practically used. In this pilot plant test feeding rate of 1 t/H of solid grain matte was charged consecutively for two weeks and continuous converting could be done successively. This process is supported to overcome the problems of existing processes and be easily applied for existing plants.

3:25 PM Invited

New Approach for the Optimization of Copper Concentrates Flash Combustion by the Control of Blends: *Roberto Parra*[1]; Roberto Parada[2]; Marcelo RodrÌguez[1]; [1]Universidad de ConcepciÛn, Dept. of Metallurgl. Eng., Edmundo Larenas 270, ConcepciÛn Chile; [2]CompaÒÌa Minera Disputada de Las Condes, Chagres Smelter, Pedro de Valdivia 291, Santiago Chile

The classic criteria in the preparation of the charge for the smelting of copper concentrates in a flash furnace consider the mass and energy balance from the chemical and mineralogy composition of the concentrates. The operation parameters fixed with this method donít assure the stability of the operation and critical problems of slag quality and dust formation occur very often. This paper present the results of a laboratory test that allows the prediction of the behavior of concentrates blends in view to the minimization of dust formation and magnetite control in the slag. The combustion of different copper concentrates and blends were done in a drop tube furnace. The results show that the magnetite and dust formation donít have a linear behavior with the weight proportion of the blend and some optimal blends of 2 or 3 types of concentrates can be identified. From the reasonable hypothesis that the qualitative behavior of blends in the drop tube represents the real behavior in the burner, the preparation of the charge with this approach could help to solve those operational problems.

3:50 PM

Effect of Magnetic Field on the Rate of Slag Reduction in an Electric Furnace: *Victor Montenegro*[1]; Andrzej Warczok[1]; Tashiharu Fujisawa[2]; Gabriel Riveros[1]; [1]Universidad de Chile, Av. Tupper 2069, Santiago 2777 Chile; [2]Research Center for Advanced Waste and Emission Management, Furo-Cho, Chikusa-ku, Nagoya 464-8603 Japan

Smelting of copper concentrate in Teniente Converter produces highly oxidized slag, containing from 4 to 10% Cu and from 15 to 25% of Fe3O4. Copper recovery requires magnetite reduction to a level of 5%, determining the degree of cuprous oxide co-reduction and liberation of copper matte inclusions. Rate of slag reduction is controlled by the mass transfer to the reaction interface. Slag stirring affects the rate of reduction and enhances coalescence of mechanically entrained copper matte inclusions. Slag reduction and sedimentation in AC electric furnace is commonly used method of slag cleaning. Utilization of DC electric furnace creates beneficial conditions introducing slag electrolysis on top of chemical reductions with carbon of coke and electrodes. Application of external magnetic field, interacting with direct current in an electric furnace, induces magnetohydrodynamic phenomena, such as intensive stirring and electromagnetic buoyancy force acting on inclusions. Laboratory scale research in simulated DC electric furnace with superimposed magnetic field, show a significant acceleration of the rate of slag reduction and copper removal. Continuous measurement of gas flow and composition allowed for determination of the reduction rate along the time. Application of external magnetic field in reduction of industrial slag in a crucible simulated DC electric furnace results in the increase of reduction rate from 7 to 15 times, comparing with the identical conditions in AC electric furnace. Interaction of non-homogeneous electric field with crossed permanent magnetic field induces vigorous slag stirring enhancing mass transfer and accelerating the rate of slag reduction. Additional participation of slag electrolysis in magnetite and cuprous oxide reduction as well as acceleration of inclusions settling by electromagnetic buoyancy force affect in slag cleaning intensification.

4:15 PM Break

4:25 PM Invited

Ausmelt Technology, Flexible, Low Cost Technology for Copper Production in the 21st Century: *Joseph Sofra*[1]; Robert Matusewicz[1]; [1]Ausmelt, Ltd., 12 Kitchen Rd., Dandenong, Melbourne, Victoria 3175 Australia

It has become increasingly evident that sustainable metals production in the 21st century requires producers to balance the needs of cost effective, high value production with that of responsible plant operation. This is becoming more difficult as lower grade and more complex feed materials, including recycled scrap and residues, are being processed to reduce production costs and decrease exposure to primary metal market fluctuations. One particular facet of the metals industry facing these challenges in the 21st century are copper producers. The recent steady decline in the copper price has seen the industry cut expenditure, tighten control on income streams and focus on high returns on capital investment. For these reasons copper producers are looking towards flexible technologies that can deliver high value products, with minimal environmental impact at low capital and low sustainable operating costs. Ausmeltís Top Submerged Lancing (TSL) technology for copper production is a low cost, proven technology for the processing of both primary and secondary copper materials. Both the smelting and converting operations introduce significant advantages in environmental performance, improved process efficiencies and high rates of metal recovery. Ausmelt has made significant gains in the past 4 years in establishing a strong market position for its copper technology. New installations in China, South Africa and India as well as existing operations in Zimbabwe and China have demonstrated its effectiveness. This paper reviews the application of Ausmelt Technology for both smelting and converting of primary and secondary sources. It discusses Ausmeltís design and implementation philosophy, and how this philosophy meshes with the needs of the end user.

4:50 PM

Quantification of the Liquidus Surface of ìLime Ferriteî Slags at Several Oxygen Potentials: *Florian Kongoli*[1]; Ian McBow[1]; Akira Yazawa[2]; [1]FLOGEN Technologies, Inc., Matls. Tech. Dept., 5757 Decelles, Ste. 511, Montreal, Quebec H3S 2C3 Canada; [2]Tohoku University, 16-32, Niizaka, Aoba-ku, Sendai 981 Japan

ìLime ferriteî slag with limited silica content has proven to be a valuable choice in the modern processes of copper smelting and converting due to several advantages that this slag offers compared to the classical silicate slags. Nevertheless the liquidus surface of this slag has been experimentally measured only at low oxygen potentials such as in equilibrium with iron or near it and in air. Although most of the smelting and converting processes that use this slag occur at intermediate oxygen potentials, the liquidus surface of this slag is not known at these conditions and the effect of oxygen potential and silica has not been correctly understood. This has brought some confusion in literature as well as in the industrial practice. In this work the liquidus surface of the lime ferrite slags has been quantified by the means of a new thermophysicochemical model and a new type of liquidus surface diagrams which is very convenient for any industrial process that uses lime ferrite slags. These diagrams ca n be easily used to select the lowest liquidus temperature of lime ferrite slags at a minimum cost and can help design several fluxing strategies in copper smelting and converting processes. The effect of the oxygen potential, silica and copper is also quantified and important industrially related conclusions are drawn.

5:15 PM

Direct Copper Production by Cyclone Smelting: *I. Wilkomirsky*[1]; R. Parra[1]; [1]University of ConcepciÛn, Dept. of Metallurgl. Eng., Edmundo Larenas 270, ConcepciÛn 270 Chile

The conventional classical production of blister considers the oxidation of sulfur and iron from copper concentrates in two consecutive steps, although there are no physicochemical reasons why blister copper can not be produced in a single stage. The practical limitations are related to the technology that determines the thermal conditions and the kinetics in different reactors. An analysis was done to determine the conditions required for a cyclonic reactor pilot plant to produce blister copper directly from concentrates. The best metallurgical results were obtained with the cyclonic reactor operating at above 1450∞C.

5:40 PM Invited

The Chilean Copper Metallurgical Industry: An Update: *A. Valenzuela*[1]; J. Palacios[2]; D. Cordero[3]; M. S·nchez[4]; [1]Chilean Copper Comission, Santiago Chile; [2]University of Atacama, CopiapÛ Chile; [3]CODELCO, El Teniente Div., Rancagua Chile; [4]University of ConcepciÛn, ConcepciÛn 270 Chile

In the last years, Chile continue being the top producer of copper, either in the form of concentrates or electrowinning and electrorefining cathodes, the last one obtained from seven smelters, which have carried out several modernization plans either to increase smelting capacity or to achieve environmental regulations established in the Chilean legislation. This paper updates the current situation of the Chilean copper metallurgical industry developed in the last years, regarding technological innovations in smelters, environmental regulations and presenting solutions for management of SO2, particulate matter and arsenic, considering that Chile is the worldís largest copper producer. Also, the authors emphasize the important role played by CODELCO Chile, the more important state owner copper company, developing Chilean technologies, as El Teniente Converter and El Teniente Slag Cleaning Furnace.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Aqueous and Electrochemical Processing III

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee: See Plenary Session for Co-Sponsors

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Tuesday PM	Room: Pacific
March 4, 2003	Location: San Diego Marriott Hotel

Session Chairs: Adam C. Powell, IV, Massachusetts Institute of Technology, Dept. of Matl. Sci. & Eng., Cambridge, MA 02139-4301 USA; Ryosuke O. Suzuki, Kyoto University, Dept. of Energy Sci. & Tech., Kyoto, 606-8501 Japan

2:30 PM Invited

OS ProcessñThermochemical Approach to Reduce Titanium Oxide in the Molten CaCl2: *Ryosuke O. Suzuki*[1]; Katsutoshi Ono[1]; [1]Kyoto University, Dept. of Energy Sci. & Tech., Yoshida-Honmachi, Sakyo-ku, Kyoto 606-8501 Japan

A new cell concept for calciothermic reduction is presented where titanium dioxide is used as the raw material for reduction. It bases on the thermochemical requirement that metallic calcium is needed as the reductant, CaCl2 as the solvent. The thermodynamic phase equilibria and physical properties are analyzed to optimize the reaction and to separate the product. The reduction system consists in a single cell, where both the reduction reaction and the electrolytic reaction for recovery of reducing agent coexist in the same molten calcium chloride bath. TiO2 powder is directly top-charged into themolten CaCl2. A few %Ca dissolves in the melt, and it constitutes the media with a strong reducing power. TiO2 + 2 Ca+ + 2 e- = Ti + 2 Ca2+ + 2 O2- Sufficiently deoxidized titanium metal deposits agglomerate rapidly and form granular sponge, which sink down to the bottom of the cell. Both mechanisms of the halide flux deoxidation and the electrochemical deoxidation work efficiently for these fine precipitates. The reducing agent is in situ recovered by electrolysis of CaO, which is supplied as the by-product of reduction. The molten CaCl2 has a relatively large solubility for CaO. At the anode: C + 2 O2- = CO2 + 4 e- At the cathode: Ca2+ + e- = Ca+ Some cell designs and modifications are proposed for industrial applications.

3:00 PM

Preparation of Uniform Ultra-Fine Nickel Oxalate Particles by Chelating Precipitation: Huang Kai[1]; Chen Huiguang[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Eng., Lushannanlu, Changsha, Hunan 410083 China

Uniform spherical and fiber-like nickel oxalate particles were prepared by chelating precipitation method, which is by adding the oxalate acid into the solution of ammine nickel chloride. The reactive concentration of the ammine nickel chloride and the oxalate acid are the key factors to the morphologies of the final particles, i.e., low reactive concentrations(<0.1 mol) of the two reagents are in favor of the formation of fiber-like particles while high reactive concentrations are in favor of the formation of spherical particles. The feeding methods and the agitation strength all have effects on the particle formation, which experimentally proved the above formation mechanism to be reasonable.

3:25 PM

Anion-Exchange Separation in HCl Media for the Ultra-High Purification of Cobalt: *Tamas Kekesi*[1]; Masahito Uchikoshi[2]; Kouji Mimura[3]; Minoru Isshiki[3]; [1]University of Miskolc, Miskolc, Egyetemvaros 3515 Hungary; [2]Fine Materials Corporation, Tagajo 985-0843 Japan; [3]Tohoku University, Inst. for Adv. Matls. Procg., Sendai 980-8577 Japan

Anion exchange in HCl media is considered an efficient alternative to the combination of the conventional purification methods. Anion-exchange distribution functions have been determined for cobalt and the main impurity elements by the technique of batch equilibration. Results were confirmed by spectrophotometric investigations and elution tests. Based on the new and the available distribution functions, combined with the assessment of thermodynamic stability, a procedure of anion exchange separation has been devised to eliminate virtually all the impurities from the cobalt-chloride solution. Separation of copper has been enhanced by the introduction of a preliminary step under reduced conditions. The rest of the impurities are eliminated in a second anion-exchange step applying rinsing and elution stages under oxidized conditions. The optimum parameters of the procedure have been determined according to performance characteristics (purification ratios, yields and volume efficiencies) derived from the analysis of elution curves obtained with laboratory scale ion-exchange columns. Special computer programs have been developed to facilitate thermodynamic simulation, analytical correction and data processing.

3:50 PM

Morphological Characteristics of Nickel Particles Electrodeposited from Aqueous Solution: *Guo Xueyi*[1]; Zhang Chuanfu[2]; Zhang Duomo[2]; Masazumi Okido[3]; Huang Kai[2]; Li Qihou[2]; [1]The University of Tokyo, Ctr. for Collaborative Rsrch., Komaba 4-6-1, Meguro-Ku, Tokyo 153-8505 Japan; [2]Central South University, Col. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China; [3]Nagoya University, Ctr. for Integrated Rsrch. in Sci. & Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

In this study, the electrolytic nickel powder was deposited from the NiSO4-NH4Cl-NaCl-H3BO3-H2O solution. The morphological characteristics of the nickel particles electrodeposited from this aqueous solution were investigated. The effects of the electrolyte constituents, the parameters for the electro-deposition on the morphology of the nickel particles were addressed. It is concluded that not only the particle size, but also the morphology of the deposited nickel particles strongly depend on the electrolyte constituents and technical conditions. Especially, it is observed that the growth of the particle morphology is strongly related to the duration for electro-deposition. The ultra-fine nickel particles with flake, nodular or dendrite shape may be easily deposited by just controlling the time interval for particle growth. Further, the influences of various conditions on the particle morphology were explained, the growth mechanism of the particle electrodeposited from the solution was elucidated.

4:15 PM Break

4:25 PM Invited

Detailed Mathematical Modeling of Liquid Metal Streamer Formation and Breakup: David Dussault[1]; *Adam C. Powell*[1]; [1]Massachusetts Institute of Technology, Dept. of Matl. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139-4301 USA

A three-dimensional Cahn-Hilliard phase field model is formulated to describe transport-limited electrochemical reactions coupled with fluid flow in a metal reduction cell. When the reaction is limited by mass transfer of metal ions from the electrolyte to the cathode, the metal-electrolyte interface at the cathode exhibits a Mullins-Sekerka instability, leading to the growth of dendrite-like ìstreamersî of liquid metal into the electrolyte, which in turn significantly enhance the apparent mass transfer coefficient there. Model results for iron reduction from ferrous oxide in slag show formation of these streamers, and their breakup due to the instability of the cylindrical liquid-liquid interface. Although the double-layer is not included in this model, the related phenomenon of electrocapillarity is represented by a gradient penalty term which includes the electric potential gradient, such that interfacial energy is a function of the electric field normal to the interface.

4:55 PM Invited

Purification of Leach Solutions by Direct Solvent Extraction: *Chu Yong Cheng*[1]; Mark Urbani[1]; [1]AJ Parker Cooperative Research Centre for Hydrometallurgy/CSIRO Minerals, Australia, Conlon St, Waterford, PO Box 90, Bentley, WA 6982 Australia

The solvent extraction (SX) processes of the four nickel plants in Australia and the Goro SX process in New Caledonia were reviewed. The use of intermediate precipitation, solids/liquid separation and re-leach in the three WA nickel plants and the use of Cyanex 301 for a direct solvent extraction (DSX) process in the Goro process make these processes complicated and costly in capital and operation. The research work carried out by the SX group at the AJ Parker Cooperative Research Centre for Hydrometallurgy/CSIRO Minerals has led to the invention of DSX processes to recover nickel and cobalt from leach solutions. The simplicity of the process flowsheets and the expected savings in capital and operating costs are the major advantages of the new DSX processes over the reviewed processes. By using a new synergistic organic system in semi-continuous tests with a pilot plant leach solution from BHP-Billiton Stainless Steel Materials (after iron precipitation), the metal values (Ni and Co) together with zinc and co pper were separated from the major impurities (Mn, Mg and Ca, together with Cl) in the first SX circuit. The co-extracted manganese, magnesium and calcium were easily scrubbed out. After stripping, the metal values (Ni and Co) together with zinc and copper were concentrated, resulting in a much smaller second SX circuit and equipment in the down stream processes. The extraction and stripping kinetics of the metals with the new synergistic organic solution were very fast. Within 0.5 minutes, the extraction and stripping almost reached steady state. Semi-continuous test work with a synthetic leach solution, a cobalt pilot plant leach solution from Peko Rehabilitation Project Pty Ltd and a synthetic solution to simulate a concentrated solution from BHP-Billiton Stainless Steel Materials showed that manganese, calcium, copper and zinc can be effectively and efficiently separated from nickel, cobalt and magnesium by extraction, scrubbing and stripping. This led to the invention of another type of DSX process using D2EHPA to recover nickel and cobalt from leach solutions.

5:25 PM

Nontoxic Method of Nickel/Cobalt/Copper Sulfides Precipitation as Rich Concentrates from Diluted Sulphate Solutions: *M. I. Kalashnikova*[1]; Y. M. Shneerson[1]; M. V. Keskinova[1]; V. V. Chetvertakov[1]; [1]Norilsk Nickel RJS, Gipronickel Institute JS, Saint-Petersburg Russia

Results of investigations concerning precipitation of heavy non-ferrous metals from solutions using precipitators based on elemental sulphur and lime with gypsum transfer to a separate product are given.

5:50 PM

The Polymorph Transformations of Antimony White: Xiao Songwen[1]; Yan Xiaohui[2]; Xiao Xiao[1]; [1]Changsha Research Institute of Mining & Metallurgy, Lushannanlu, Changsha, Hunan 410012 China; [2]Hunan Sunrise Nanometter Material Company, Ltd., Yuwanzheng, Changsha, Hunan 410014 China

On ground of the theoretical models of growth unites with coordination polyhedron structure of anion, the polymorph transformation mechanisms of antimony white Sb2O3 in hydro-process was presented. The new findings had been verified in the commercial-scale test, and it showed that a small amount of tratrate ions in the solution was effective for the crystal transformation of antimony white in hydro-process.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Recycling, Waste Treatment and Environmental Issues III

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee: See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday PM — Room: Leucadia
March 4, 2003 — Location: San Diego Marriott Hotel

Session Chairs: Douglas Raymond Swinbourne, RMIT University, Sch. of Civil & Cheml. Eng, Melbourne, Victoria 3001 Australia; Venkoba Ramachandran, Ram Consultants, Scottsdale, AZ 85262 USA

2:30 PM Invited

Treatment of Aqueous Effluents for Recovery and/or Removal of Metals in Non-Ferrous Metalsí IndustryñA Review: *Venkoba Ramachandran*[1]; [1]Ram Consultants, 9650, E. Peregrine Pl., Scottsdale, AZ 85262 USA

In view of the increasing environmental regulations that have been promulgated in the last 30 years, non-ferrous smelters and refineries have learnt to handle effectively the process waste streams that need to be bled from the system for impuritiesí control. The cations of concern are copper, lead, zinc, cadmium, mercury, thallium, nickel, silver, sodium and potassium. Anionic species of arsenic and selenium also need to be addressed. Sulfates and chlorides-not a major problem at present- have come under scrutiny in the recent past. The current paper reviews several treatment options with reference to a)types of waste streams processed b)process chemistry c)discharge options and recovery of treated water and d)sludge disposal. The long term goal of any smelter and/or refinery should be to have zero liquid discharge from the plant. Methods to obtain zero discharge are discussed.

3:00 PM

A Dynamic LCA Model for Assessing the Impact of Lead Free Solder: *M. A. Reuter*[1]; E. Verhoef[1]; A. Scholte[1]; G. Dijkema[1]; [1]Delft University of Technology, Dept. of Appl. Earth Scis., Mijnbouwstraat 120, Delft 2628 RX The Netherlands

While the importance of LCA to support decision-making, in particular to environmental impact assessment, is widely acknowledged, there is also some criticism. LCAs are very data intensive, which means the success of a study is strongly dependent on the availability of good data. Thus may explain why in many studies time dependence is neglected, and production of materials are considered individually. Although this may give rise to significant errors in calculated environmental impact, because of the interconnectedness and dynamics of systems considered. The carbon cycle, for example, is interlinked both via the non-hydrocarbon components of commercial plastics, such as chlorine and heavy metals, and via residues from refining and coals based operations. The production of one material, therefore, often is interrelated to and dependent on the consumption or generation of another. Most of the raw materials for silver production, for example, are not concentrated silver ores, but obtained as by-products of copper, steel and zinc production. When primary production of these material changes, silver production is affected. Similarly, in case outlets for arsenic cease to exist, primary production of copper, lead and zinc must be modified. Similar to this, the recovery of one material can interrelate to the recovery of another. The recycling of copper from messing or the steel from cars also produces zinc. On that account, a dynamic, hierarchical model of a number of interconnected material cycles is developed. The model includes detailed models of the production and waste management stages to connect production or recovery of one material to the production of others. As the interconnection of metal production occurs at process level, modelling of the different production route involves considering the different types of production processes individually. The model is constructed on the basis of mass balances, in which the lower system levels represent increasing technical detail. Consistent with Reuter (1996) split factors, or recoveries, are used to model the processes. Data reconciliation is used on calculate the split factors from literature values on process feed, products and residue composition to ensure data quality. National, European or world production figures on process capacity can be used to calculate the relative contribution of processes. The model is set up dynamically. The continuous generation of new products, by-products, and material wastes result changes of the production network, e.g. the selection resources or suppliers employed, factory locations, innovation of facilities and products. In the model these changes can be considered, e.g. by changing the distribution of material flows over the different processes, or changing the split factors in time. The hierarchical structure allows matching the information needs of the different actors, supporting and/or coordinating decision-making processes in industry, SWM and governments. Municipal waste flows can be estimated from series of production data and assumption on delay of materials in the consumption phase. This allows for the evaluation of solid waste policy options in different dynamic scenarios with respect to the environment and resource depletion, but also the evaluation of the impact of lead-free soldering. The hierarchical structure allows In addition to the absolute environmental impacts, the model can be used to evaluate relative impact of policy, infrastructure or product design on the whole system as well.

3:25 PM

Disposal Treatment of Alternative CFC Gas(C2H2F4) Using Chemical Reaction with Metal Compounds: *Hideki Yamamoto*[1]; Akihiro Kushida[1]; Norihiro Murayama[1]; Junji Shibata[1]; [1]Kansai University, Dept. of Cheml. Eng., Fac. of Eng., 3-3-35, Yamatecho, Suitashi, Osaka 564-8680 Japan

A new technology to make alternative CFC gas (C2H2F4:R-134a) used as refrigerant of an air cooling units or a refrigerators convert directly into a harmless substance have been established and new concept on the disposal treatment of global warming gas was presented. Reaction vessel made of Hastroy steel was applied to examine the chemical reaction of R-134a with metal chloride and metal oxides at the valuable experimental conditions. The effect of the kind of metal compound on the chemical reaction activity for the disposal treatment of R134-a gas was examined in this experiment. In the case of metal chloride(CaCl2, MgCl2), the beginning temperature of these chemical reactions are at 700K-770K and the reaction percentage of solid phase base is about 30-60%. Beginning temperature of the chemical reaction with metal oxide(CaO) is 820K, and the reaction percentage of solid phase base is about 60%. Experimental results verify that the chemical reactions can be take place at substantially lower temperature of 700 K-850K as compared with the combustion treatment method(1300K-1500K). Reaction product is mainly metal fluoride which is a harmless and a valuable chemical material as new resource. The other favorable characteristics are that the continuous treatment is possible at a low temperature under atmospheric pressure. Furthermore this process is compact, easily controllable and safely operable at low running cost. This paper concerns with a new harmless disposal treatment of toxic global warming gas.

3:50 PM

Modelling and Control of Dioxin Formation During Iron Ore Sintering: *Pengfu Tan*[1]; Dieter Neusch¸tz[2]; [1]Portovesme s.r.l., S.P.n.2-Carbonia/Portoscuso, Portoscuso 09010 Italy; [2]Rheinisch-Westf‰lische Technische Hochschule Aachen, Lehrstuhl f¸r Werkstoffchemie, Aachen D-52056 Germany

In order to minimize dioxins and furans formation in iron ore sinter plants and to possibly avoid expensive end-of-pipe removal stages, thermodynamic calculations on the stability of PCDD/Fs have been combined with sinter process modelling by means of kinetic simulations and computational fluid dynamics. Three thermodynamics databases of PCDD/Fs, derived using the Group Additivity approach and two computational molecular modeling methods, MNDO and PM3, respectively, combined with the SGTE database have been used to model the PCDD/F formation and the PCDD/F isomer distributions in iron ore sintering. The results show very similar conditions for the PCDD/F formation using the different databases. The predicted isomer distributions of toxic PCDD/Fs have been compared with measured data from sinter plants, electrical arc furnaces, waste incinerators and wood burning furnaces. The calculated data using the database derived from the MNDO method show the best agreement with the industrial measurements. The thermodynamic calculations also show that the isomer distributions found in combustion processes are equilibrium values, while the concentration ratio of PCDDs and PCDFs in the wind boxes does not represent an equilibrium state. A CFD model of iron ore sintering was developed including the kinetics of 15 relevant chemical reactions with the aim of describing the chemical and the thermal processes of sintering and the gas flow patterns. On the basis of these informations possible pathways for the PCDD/F formation in sinter plants have been identified: Between charging zone and center part of the sinter bed, PCDD/Fs are formed below the hot zone, carried downwards with the gas and condensed in the cold zone. Transported to the discharge end with the solid mixture, they are again released into the gas phase when the flame front approaches the bottom of the bed. In addition, PCDD/Fs are formed in the last wind boxes when the hot offgases cool down and reach the critical temperature range. Consequently, methods to reduce the dioxin formation reactions are proposed including inhibitor addition and off-gas quenching.

4:15 PM Break

4:35 PM Invited

Behaviour of Thallium During Direct Lead SmeltingñA Thermodynamic Viewpoint: *Douglas Raymond Swinbourne*[1]; Akira Yazawa[2]; [1]RMIT University, Sch. of Civil & Cheml. Eng., PO Box 2476V, Melbourne, Victoria 3001 Australia; [2]Tohoku University, 16-32 Niizaka, Sendai 981-0934 Japan

Thallium is a minor element found in lead ores. The behaviour of thallium during lead smelting has become of much more interest recently due to the toxic effect it had on maintenance staff at one particular smelter. In this paper the behaviour of thallium will be examined using computational thermodynamics techniques for the case of direct smelting of lead concentrate by several generic technologies. Such methods have been pioneered by Prof. Yazawa to model many processes in extractive metallurgy. It will be shown that thallium behaviour is strongly influenced by the presence or absence of certain secondary recycle materials in the charge and by the way in which the smelter offgases are handled.

5:05 PM

Environmental Assessment of Imperial Smelting Process Practice in China: *Guo Xueyi*[1]; Xiao Songwen[2]; Li Qihou[2]; Nie Zuoren[1]; Zhang Duomo[2]; Yamamoto Ryoichi[1]; [1]University of Tokyo, Ctr. for Collaborative Rsrch., Komaba 4-6-1, Meguro-Ku, Tokyo 153-8505 Japan; [2]Central South University, Col. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

Imperial Smelting Process is one of important method to produce Zinc & Lead. Shaoguan Smelter in South China is a representive metallurgical plant to adopt ISP for the zinc and lead production with the annual production capacity of about 200 thousand tons. In this study, by analyzing the resource use, energy consumption and waste emission and disposal, the environmental burden of the ISP practice in this smelter was addressed, the impact assessment was conducted quantatively by applying some environmental indicators, and further the approaches for improving the resource & energy use efficiency as well as releasing the environmental burden were proposed.

5:30 PM

Use of Solid Hematite to Fix Arsenic Contained in a Gas Phase: *A. Gonzalez*[1]; E. Balladares[1]; R. Parra[1]; M. S·nchez[1]; [1]Universidad de ConcepciÛn, Edmundo Larenas 270, Casilla, ConcepciÛn 53-C Chile

Arsenic is one of the main contaminants presents in copper mining resources today and its abatement represents a challenge for metal extraction in the future, particularly under the strong environmental requierements in a global community. The possibility to form ferric arsenate directly from a gas phase by means of a heterogeneous reaction between an arsenic gas bearing and iron oxide was studied at laboratory scale. A thermodynamical analysis for the system Fe-As-O-S has been initially made, in order to obtain the stability conditions for the ferric arsenate or other $Fe_xAs_yO_z$ type compounds. Experiments were made using an hematite sample suspended from a thermogravimetric device, inside a vertical furnace. Gas As4O6 generated from solid As2O3 were passed through the iron oxide sample and its instantaneous weight changes was recorded directly in a computer. A kinetic study for the As4O6 volatilization was made, which shows chemical control between 300 and 600∞C, and with an apparent activation energy of around 14 Kcal/mol As2O3. Samples after reactions were chemically and by XRD analysed. Arsenates detected corresponds to complex compounds. Results shows that formation of compounds depend on temperature and oxygen content of the gas phase. Kinetic for iron arsenates formation is slow, reaching an arsenic capture of 6% after 80 minutes of reaction when an oxygen content of 50% is fed continously at 800∞C. Experiences made at the same temperature but under higher oxygen content in a closed reactor, allowed arsenic capture close to 60%. For higher temperatures capture is notoriously lower.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: General III

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee: See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Tuesday PM — Room: Point Loma
March 4, 2003 — Location: San Diego Marriott Hotel

Session Chairs: Chikabumi Yamauchi, Chubu University, Grad. Sch. of Eng. & Sch. of Eng., Aichi-ken 487-8501 Japan; David G.C. Robertson, University of Missouri-Rolla, Dept. of Metallurgl. Eng., Rolla, MO 65401 USA

2:30 PM Keynote

The Chemistry of Fuming Zinc from Oxide Slags Using Coke: *David G.C. Robertson*[1]; Dhiren K. Panda[2]; Adrian C. Deneys[3]; [1]University of Missouri-Rolla, Dept. of Metallurgl. Eng., Rolla, MO 65401 USA; [2]Nucor-Yamato Steel, Blytheville, AR 72315 USA; [3]Praxair, Inc., Indianapolis, IN 46222 USA

The key reaction in zinc fuming is ZnO(in slag) + CO = Zn(g) + CO2. This paper includes the results of experiments carried out by the authors. In the first series of experiments it was shown that the reaction 2FeO + ZnO = Fe2O3 + Zn(g) does not occur at an appreciable rate. In the second series it was shown that zinc oxice is not redcued unless a reductant has been added. A third series of experiments was carried out with excess coke. The kinetics of zinc oxide reduction were found to be independent of the zinc oxide concentration, but increased with increasing basicity, and temperature. The presence of high concentrations of FeO in the slag is also known to increase the rate. We propose that, in our work, the kinetics were controlled by a chemical step, namely the rate of desorption of an adsorbed CO2 species.

3:00 PM

Effect of Ultrasonic Wave Irradiation on the Electrical Conductivity of Pure and Sodium Doped Sulfur Melts: *Nobuaki Sato*[1]; Rajmund Michalski[2]; Takeo Fujino[3]; Yoshio Waseda[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan; [2]Polish Academy of Science, Inst. of Environml. Eng., Skrodowska-Curie 34 St., Zabrze 41-819 Poland; [3]Tohoku University, Inst. for Adv. Matls. Procg., 19-14 Higashioshima, Hitachinaka 312-0042 Japan

Sulfur melt is expected to have a potential use in the materials synthesis field as a molten process, such as a low temperature synthesis of mixed sulfides. If the adiabatic property of the sulfur is improved to have a relatively high conductivity by the irradiation of ultrasonic waves, it would extend to the electrochemical process. In this paper, the electrical conductivity, σ, of pure and sodium doped sulfur melts was measured in the presence of ultrasonic waves from 433 to 573 K by the two probe method using ultra high resister meter. The σ value of the pure sulfur melt with irradiation was jumped at around 443 K by the order of 10^2 and then monotonically increased from 10^{-5} to 10^{-4} Scm^{-1} with increasing temperature. The addition of sodium metal to the sulfur melt was proved to be more effective for jumping the σ value of the melt to an acceptable level when ultrasonic wave was irradiated.

3:25 PM Invited

Mathematical Modeling of Phase Interaction Taking Place During Fusion Welding Processes: *Michael Zinigrad*[1]; Vladimir Mazurovsky[1]; Alexander Zinigrad[1]; [1]College of Judea and Samaria, Science Park, Ariel 44837 Israel

The quality of metallic materials depends on their composition and structure and these are determined by various physico-chemical and technological factors. To effectively prepare materials with required composition, structure and properties is necessary to carry out research in two parallel directions: 1. Comprehensive analysis of thermodynamics, kinetics and mechanisms of the processes taking place at the solid-liquid-gaseous phase interface during welding processes; 2. Development of mathematical models of the specific welding technologies. We have developed unique method of mathematical modeling of phase interaction at high temperatures. This method allows us to build models taking into account: thermodynamic characteristics of the processes, influence of the initial composition and temperature on the equilibrium state of the reactions, kinetics of heterogeneous processes, influence of the temperature, composition, hydrodynamic and thermal factors on the velocity of the chemical and diffusion processes. The model can be implemented in optimization of various technological processes in welding, surfacing, casting as well as in manufacturing of steels and non-ferrous alloys, materials refining, alloying with special additives, removing of non-metallic inclusions.

3:50 PM

Green Technology to Recovery Value Metal Compounds from Molten Slags: *Zhitong Sui*[1]; Li Zhang[1]; Taiping Lou[1]; Zhida Sui[1]; [1]Northeastern University, Sch. of Matls. & Metall., Shenyang 110006 China

Based on several case studies in precipitating behavior of value metal compounds (VMC) in molten slags a green technology to recovery VMC from molten slags is proposed, in which three steps are involved: (1) The selective concentrating of dispersed VMC into the designed mineral phase in molten slag; (2) The selective coarsening of the designed mineral phase to critical grain size in molten slag; (3) The selective separating of the grown mineral phase in solidified slag from tailing by dressing or hydrometallurgy processes. The features of the technology are economic, clean, intensive and comprehensive. The utilization of recovered VMC such as Titanium, Boron, Vanadium, chromium and Iron compounds was summarized as examples of technology application. It was confirmed by experiments that the precipitating of the designed mineral phases like Perovskite ($CaTiO_3$), Suanite ($2MgO \cdot B_2O_3$), Spinel ($MgO \cdot Cr_2O_3$) and Magnetite (Fe_3O_4) in molten slags are obviously affected by operation factors such temperature, chemical composition, heat-treatment, additives and so on. The precipitating kinetics and mechanism of VMC from molten slags during solidification processes were also investigated.

4:15 PM Break

4:35 PM Keynote

Progress and Problems in the Simulation of Non-Ferrous Extraction Processes: *Arthur E. Morris*[1]; [1]Thermart Software, 12102 Calle de Maria, San Diego, CA 92128-2720 USA

Fifty years ago when Professor Yazawa began his career, the field of process simulation and modeling was in its infancy. Since then, tremendous progress has been made in our ability to model metal production flowsheets from mine to refined metal. This progress has come about by parallel developments in basic understanding of process chemistry and in computational techniques. This paper will review the progress made in process understanding and modeling in lead, zinc, and copper metallurgy from its inception to the present, and the role of Professor Yazawaís work in furthering this progress. Specific examples will be given for the sintering of lead concentrates, slag fuming, zinc concentrate roasting, flash smelting of copper, and pyro-extraction of zinc. Factors limiting further progress will be described.

5:05 PM

The Experiment on Reducing-Matte Smelting of Jamesonite Concentration: Tang Chaobo[1]; Tang Motang[1]; Yao Weiyi[1]; [1]Central South University, Dept. of Metall., Yuelunanlu, Changsha, Hunan 410083 China

The reducing-matte smelting is a new pollution-free metallurgy technique. In the process pyrite cinder or ferric oxide ore reacts with non-ferrous metal sulphide and produce metal. In this paper, the reducing-matte smelting of jamesonite concentrate has been conducted, and the influences of temperature, amount of pyrite cinder and amount of additive on the smelting process have been studied. Under the optimum technical conditions, the direct recovery ratio of antimony and lead are 83.26% and 68.5%. The ratio of fixed sulfur is 98.97%. It shows that this method is feasible and pollution-free of sulfur dioxide. But the distribution ratio of lead and silver in ferrous matte is 15% and 30%, therefore how to recover lead and silver economically and effectively from ferrous matte is still a problem to be solved.

5:30 PM

Melt AtomisationñThe Bridge from Pyro- to Hydro-Metallurgy: *J. J. Dunkley*[1]; D. Norval[2]; [1]Atomising Systems, Ltd., Sheffield S9 1EW England; [2]Bateman Pty, Johannesburg S. Africa

Recent years have seen the increased use of hydrometallurgy in the smelting and refining of metals. However this is often used as a final refining step, following an initial pyrometallurgical smelting process. Huge amounts of material are produced as melts which have to be cast, comminuted, and taken up into solution or leached. A common method is to granulate the melt and then grind it. In the case of metallic melts this can be difficult or unfeasible due to the ductility of the granulated particles. Water atomisation is capable of replacing the granulation and grinding stages with a single process step that can process in excess of 30t/hour of melt into particles as fine as 50 microns. Applications in operation in Co, Cu, Ni, Ag, Au, Pt and Rh refining are discussed together with potential applications in copper smelting.

Notes

15th International Symposium on Experimental Methods for Microgravity Materials Science - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee

Program Organizers: Robert Schiffman, R. S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica & Instituto Nazionale di Fisica della Materia, Bologna 40127 Italy

Wednesday AM — Room: 10
March 5, 2003 — Location: San Diego Convention Center

Session Chair: C. Patuelli, Universita di Bologna, Dipto. di Fisica & Inst. Nazionale di Fisica della Materia, Bologna 40127 Italy

8:30 AM Introduction

8:45 AM
Precursor Phenomena in Melts Preceding the Solidification of In-50Sn and In-10Sn Alloys: *G. Costanza*[1]; F. Gauzzi[1]; R. Montanari[1]; [1]Universit‡ di Roma-Tor Vergata, Dipartimento di Ingegneria Meccanica, Via di Tor Vergata 110, Roma 00133 Italy

The solidification of two Indium-Tin alloys (in-50Sn and In-10Sn) has been investigated by X-ray diffractometry (XRD) in 1-g conditions. During cooling one of the alloys (In-10Sn) exhibits the eutectic transformation. The Sn content of other alloy (In-50Sn) is just lower than the range of peritectic transformation. The XRD spectra have been collected at decreasing temperatures for monitoring step by step the structural changes occurring in the liquid phase before the formation of the first solid and during the liquid-solid transformation till its completion. From the evolution of radial distribution function (RDF) it was possible to draw information about atom clustering in the melt preceding and accompanying the solidification of both the alloys and to evidence correlations between the structures of liquid and solid phases. To avoid convective motions in the liquid and to achieve the best experimental conditions, it is discussed the possibility to perform the same experiments under conditions of reduced gravity.

9:05 AM
Benard-Marangoni Interaction in Liquid Layers: *G. S.R. Sarma*[1]; [1]German Aerospace Center, Gottingen 37073 Germany

Convective instabilities induced by the presence of temperature and/or concentration gradients across liquid layers under the action of body forces due to Boussinesq, Coriolis, and Lorentz fields have been studied for a long time now but only a few of their results have been applied in a practical engineering sense. In the context of microgravity research the main focus has been on the Marangoni flows driven by surface tension gradients in view of their potential applications in crystal growth and other materials processing configurations where fluid-fluid interfaces occur. On the other hand, very few experimental studies have been even conceived to deal with the onset of convective instability in liquid layers under the simultaneous action of density Benard and surface tension (Marangoni) gradients across the layers. The former received by itself a lot of theoretical and experimental interest but the corresponding Marangoni problems definitely lagged behind due to inherent difficulties both in terrestrial and space environments. The pioneering Apollo-era experiments of Grodzka and Bannister from the 1970ís, despite their very preliminary data and inadequate characterization, are still the only ones that could be referred to during theoretical discussions of 1980ís. Basically, the problem here is one of interaction between two different instability mechanisms taken each by itself but which can act to oppose each other under certain circumstances and thereby stabilize the configuration. Although the underlying configuration is rather simple and can match fairly theoretical idealizations, there is, to date, no satisfactory ground based experimental data base to test even linear stability theories, leave alone non-linear aspects of pattern formation in such configurations. This situation offers potentially a plethora of possibilities for fluid physics studies within the microgravity program. The present contribution will attempt to describe such opportunities which seem to have been by-passed during the intervening years of active microgravity work. The starting point of the discussion will be motivated, for reasons of expediency, by the authorís own typical publications of the late 1980ís after which the topical area does not seem to have germinated any suitable experimental ideas. A review of the basic theoretical results available on the Benard-Marangoni interaction problem and a projection of possibilities for their eventual validation will be the main focus of the presentation.

9:25 AM
In Situ and Real Time Characterization of Solid-Liquid Interface Morphology: Directional Solidification in DECLIC Facility: *Nathalie Bergeon*[1]; HaÔk Jamgotchian[1]; Bernard Billia[1]; [1]L2MP, FacultÈ de St. JÈrÙme, Case 151, Av. Escadrille Normandie Niemen, Marseille, Cedex 20 13397 France

A directional solidification device dedicated to in situ and real time characterization of solid-liquid interface morphology on bulk samples of transparent materials is presented. This device, which will be implemented on ISS, is developed in the frame of the DECLIC project of the French Space Agency (CNES). The alloy is contained in a cylindrical crucible and observation is performed in two perpendicular directions: the growth one and the transverse one. In addition to direct observation by light transmission in those directions, an interferometer is set up in growth direction to provide the shape and the motion of the interface through an analysis of the interference fringes. Ground results on interface dynamics are shown: front recoil, triggering of instability, microstructure formation, that are all influenced by fluid flow. Microgravity experiments are planned to obtain critical benchmark data in diffusive environment and a better knowledge of convection influence.

9:45 AM
The Solidification Velocity of Undercooled Nickel and Titanium Alloys with Dilute Solute: *Paul R. Algoso*[1]; Alex S. Altgilbers[2]; William H. Hofmeister[1]; Robert J. Bayuzick[1]; [1]Vanderbilt University, Cheml. Eng., Box 1604, Sta. B, Nashville, TN 37235 USA; [2]Carrier Corporation, 284 Carrier Dr., Morrison, TN 37357 USA

The Ivantsov solution with marginal stability arguments (IMS model) is one of the most widely used models for relating solidification velocity to the undercooling of dilute metal alloys but fails to fit experimental results, particularly for alloy systems with a high equilibrium partition coefficient, k_E. The solidification velocity of undercooled binary alloys in electromagnetic levitation was measured utilizing high-speed thermal imaging techniques in order to reconcile differences between experimental results and theoretical predictions. Plateaus in the solidification velocity versus undercooling curve are observed. The first plateau at intermediate undercoolings is a result of solute addition, while the second plateau at high undercoolings is attributed to oxygen. IMS modeling is applied to the experimental data, and agreement is found when a value for k_E is used that is lower than the actual k_E of the alloy.

10:05 AM Break

10:25 AM
The Devitrification Process in ZBLAN Glasses: *Reginald William Smith*[1]; Ian Robert Dunkley[1]; [1]Queenís University, Matls. Sci. of Microgravity Applic., Nicol Hall, Rm. 228, Kingston, Ontario K7L 3N6 Canada

It is known that a reduced gravity environment will help reduce crystallisation when processing ZBLAN glasses, but the reason for this is still a matter of speculation. This talk will describe recent work at Queenís University concerned with the containerless processing and characterisation of ZBLAN glasses in preparation for reduced gravity studies of the crystallisation process in these glasses.

10:45 AM
Faceted Growth and Kinetics of Doped Germanium Single Crystals: Andrew David Deal[1]; Ercan Balikci[1]; *Reza Abbaschian*[1]; [1]University of Florida, Matls. Sci. & Eng., 100 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA

Semiconductor materials exhibit faceted growth from a melt, a phenomenon characterized by the lateral motion of elementary steps along an atomically smooth solid/liquid (s/l) interface. Understanding the fundamentals of this growth mechanism and the incorporation of the dopant into the solid is necessary to improve the quality and reproducibility of semiconductor devices fabricated within the electronic industry. A novel growth technique called Axial Heat Processing (AHP), which enables close control of geometrical and thermal boundary conditions near the s/l interface, was used to study the effects of several kinetic parameters on the morphological stability of a faceted s/l interface in doped Ge. AHP utilizes a submerged heater to impose a planar thermal isotherms near the interface. The effects of the processing variable such as growth rate and melt height below the submerged heater on the radial and axial distribution of the solute will be presented.

11:05 AM
Wetting and Nucleation Behavior of Binary Immiscible Alloys: *L. J. Little*[1]; J. B. Andrews[1]; [1]University of Alabama-Birmingham, Matls. Sci. & Eng., 1150 10th Ave. S., BEC 254, Birmingham, AL 35294 USA

There are many potential applications for binary immiscible alloys if they can be produced to form composite-like structures (i.e. an aligned, fibrous structure or a solid dispersion with one phase uniformly distributed within the other). During processing of these alloys on Earth, the density difference between the two phases leads to massive segregation of the phases due to gravity-driven sedimentation. Processing the alloys in a microgravity environment should alleviate this problem. However, early experiments that were performed in microgravity conditions still led to massive segregation of the liquid phases. It is the purpose of this investigation to determine the cause of microgravity segregation in binary immiscible alloys. The Wetting Characteristics of Immiscibles (WCI) project was performed in November of 1997 as part of the USMP-4 shuttle mission. It was the objective of this project to determine if droplet migration and perfect wetting of the minor liquid phase were the major factors leading to segregation in these alloys under microgravity conditions. The immiscible transparent metal analog system succinonitrile-glycerol (SCN-GLY) was utilized in the investigation, and an estimated perfect wetting range of the system was identified. Currently, ground-based work is underway based on the results of the WCI shuttle mission. The investigation continues in determining factors that lead to massive segregation in binary immiscible alloys by focusing on the influence of perfect wetting on preferential nucleation of the minor immiscible liquid phase.

Actinide Materials: Processing, Characterization, and Behavior: Advanced Fuels and Materials I

Sponsored by: Light Metals Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS), LMD-Reactive Metals Committee
Program Organizers: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA; Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Wednesday AM — Room: 4
March 5, 2003 — Location: San Diego Convention Center

Session Chair: Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

8:30 AM
Prospects for Nuclear PowerñA System View: *Victor H. Reis*[1]; [1]SAIC

The end of the Cold War, increased international concern for climate change, electric restructuring in the United States have all raised hopes that there might be a resurgence of nuclear power during the first half of the 21st Century. For this to occcur it will require a combination of political, economic and technological factors, both domestically and internationally. Some of these factors will be discussed, and the results of some collaborative models will be described, in particular the need for closing the nuclear fuel cycle. The results indicate that significant, sustained government leadership will be required for nuclear power to be a major contributor to the global and domestic energy supply of the future.

9:15 AM
Cermet Nuclear Fuel Fabrication by Powder Metallurgy Methods: *Sean M. McDeavitt*[1]; M. C. Hash[1]; A. S. Hebden[1]; J. M. Runge[1]; C. T. Snyder[1]; A. A. Solomon[2]; [1]Argonne National Laboratory, Chem. Tech. Div., 9700 S. Cass Ave., Bldg. 205, Argonne, IL 60439-4837 USA; [2]Purdue University, Sch. of Nucl. Eng., W. Lafayette, IN 47907 USA

Cermet nuclear fuels enhance performance and safety through their high matrix thermal conductivity and low internal temperatures. The powder-in-tube drawing method is a simple low-temperature process for cermet fabrication that has advantages over hot methods, especially for fuel systems that require remote hot cell fabrication. Lab-scale equipment has been installed to develop this process for large-scale (~1.2 m length) and small-scale (~0.05 m length) samples. This method is being applied to zirconium matrix cermet fuels containing spray-dried (Th,U)O2 and resin-loaded (Pu,Am,Np)O2 microspheres. These actinide-bearing cermet fuels are being developed for application in advanced nuclear energy systems and actinide transmuter systems, respectively. Oxide microspheres (50 to 1000 mm diameter) and zirconium metal powders (~44 mm nominal diameter) are dry-mixed and loaded into alloy drawing tubes and vibratory-packed to obtain a green packing fraction of ~50%. The powder-containing tube is drawn to reduce the pinís outer diameter and compact the powders within. The internal microstructure is stabilized through a post-drawing anneal between 500∞C and 1000∞C to remove strain hardening and facilitate sintering and interfacial bonding. Multiple cycles are used with sequentially decreasing die sizes to achieve the desired level of densification.

9:40 AM
Investigations of Mononitride Actinide Ceramics for Transmutation Fuels: *Robert W. Margevicius*[1]; *Kenneth J. McClellan*[1]; [1]Los Alamos National Laboratory, Nucl. Matls. Tech., MS E505, Los Alamos, NM 87545 USA

Transmutation fuels work at Los Alamos National Laboratory is currently focused on mononitride ceramic fuel forms that have the advantages of high melting points, good thermal conductivity, and high chemical and thermal stability. The actinide fuels effort at Los Alamos emphasizes the synthesis and fabrication of actinide-bearing nitride fuel pellets. These pellets will be inserted into the Advanced Test Reactor early in 2003 and continue for the next couple of years. The nitride pellets are designed to contain varying amounts of Pu, Am, and Np. The composition ranges of the pellets go from having low minor actinide content (i.e., just PuN) to ones that have significant amounts of AmN (80%). Results presented will outline fabrication techniques which are designed to reduce the volatility of Am. Results from characterization of the powders and pellets will also be given.

10:05 AM
Atomistic Modeling of the Phase Stability in the Am System: *S. G. Srinivasan*[1]; M. I. Baskes[1]; M. Stan[1]; A. M. Niklasson[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

Actinide based ceramics have evoked renewed interest as potential next generation fuels for nuclear reactors. A reliable, many-body atomistic model of Am is necessary for its eventual application to the Am-N and Am-C systems. We describe the development of a semi-empirical model for Am based on the modified embedded atom method. Properties derived from both experimental and full-potential Linear Muffin-Tin Orbital (FPLMTO) method calculations, based on the local spin density and generalized gradient approximations, are used to develop our potential. Using this potential we investigate the phase stability, defect energetics, thermodynamic, and mechanical properties of Am using a combination of molecular dynamics and thermochemical calculations. Our results will be compared and contrasted with the existing experimental data. Lastly, we will also attempt to predict properties that have not been experimentally measured.

10:30 AM Break

10:40 AM
Phase Development in Pu-Zr Alloys with Np and Am Additions: *Dennis D. Keiser*[1]; J. Rory Kennedy[1]; Mitchell K. Meyer[1]; [1]Argonne National Laboratory, Eng. Tech. Div., PO Box 2528, Idaho Falls, ID 83403-2528 USA

Metallic alloys are being investigated as possible nuclear reactor fuels for transmuting long-lived fission products, like Np and Am, into short-lived fission products. The alloys that have been looked at are based on the Pu-Zr system with additions of up to 10 wt% Np and 12 wt% Am. Alloys have been cast using arc-melting techniques and then have been characterized using a scanning electron microscope equipped with energy-dispersive and wavelength-dispersive spectrometers (SEM/EDS/WDS). Additionally, X-ray diffraction analysis was performed to determine developed phases. Observed microstructures for the various as-cast alloys will be discussed along with the identified phases. Comparisons will be made with available phase diagrams.

11:05 AM
Thermal Characteristics and Behavior of Pu-Np-Am-Zr Bearing Alloys: *J. Rory Kennedy*[1]; [1]Argonne National Laboratory, Eng. Tech., PO Box 2528, Idaho Falls, ID 83403 USA

Recent efforts at the Argonne National Laboratory-West have included the fabrication of non-fertile inert matrix metallic fuels for study as part of the DOE) Advanced Accelerator Applications (AAA) actinide transmutation program. Specifically, the binary Pu-40Zr and Pu-60Zr alloys, the ternary Pu-10Np-40Zr and Pu-12Am-40Zr alloys, and the quaternary Pu-10Np-10Am-40Zr alloy have been prepared via an arc-casting method. Of particular interest in the application of these fuels are the volatility of Am and the possible low melting behavior of Np containing phases. These have been addressed with thermal analysis studies including thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Additional studies on fuel-cladding-chemical-interactions (FCCI) and microstructure and phase determination on annealed samples have been performed. The thermal behavior and characteristics of the alloyed materials will be presented and discussed in terms of fuel performance requirements.

11:30 AM
Wet/Dry Processing of Urania/Thoria Powders to Produce Dense Pellets: *Alvin Solomon*[1]; V. Chandramouli[1]; S. Anthonysamy[1]; S. Kuchibhotla[1]; [1]Purdue University, Sch. of Nucl. Eng., W. Lafayette, IN 47907 USA

Urania/thoria powder mixtures were produced by an optimized reverse-strike co-precipitation process to yield very fine powders of high surface area. Dry compaction with organic binders and sintering under controlled atmospheres at >1700C for 10 hours yielded a relative density of only 90%, due to agglomeration of the powder. XRD analysis showed that complete solid solutions were obtained. However, wet milling of a high zeta potential slurry with organic binders effectively broke up the agglomerates and dispersed the organic so as to produce high green and sintered densities above 97% under the same sintering conditions. The resintering behavior and thermal properties of these pellets are also reported.

11:55 AM
A Dynamic Model Describing the Morphological Changes of Powder in a Rotary Kiln: StÈphane HÈbrard[1]; Carine Ablitzer[1]; *Fabrice Patisson*[2]; Denis Ablitzer[2]; [1]CEA Cadarache, DEC/SPUA/LCU, Saint-Paul-lez-Durance 13108 France; [2]Ecole des Mines, LSG2M, UMR 7584 CNRS-INPL, Parc de Saurupt, Nancy 54042 France

A stage of the dry conversion process for producing nuclear fuel consists in defluorinating and reducing UO_2F_2 powder into UO_2 in a rotary kiln. We have undertaken mathematical modeling work that ultimately aims at predicting the morphological characteristics of the powder, which are crucial for the product quality, as a function of the kiln operating parameters. First, we have developed a new dynamic model of the solid flow in a rotary tube equipped with internal lifters. It calculates the flow-rate and hold-up of the powder in both the dense and the airborne phases. Then, a morphological change model, based on population balances of fractal agglomerates and using the results of the dynamic model, has been written to describe the agglomeration (by Brownian motion, by differential sedimentation, and shear-induced) and sintering phenomena. Results of the dynamic model, as well as the first findings concerning the morphological changes, are given and discussed.

Aluminum Reduction Technology: Fundamentals

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Martin Segatz, VAW Aluminum Technology, Bonn D-53117 Germany; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday AM	Room: 5B
March 5, 2003	Location: San Diego Convention Center

Session Chair: Michel Reverdy, Aluminium Pechiney, Paris, Cedex 16 75218 France

8:30 AM
In-Situ Observations of Frozen Electrolyte in Laboratory Reduction Cells: *Craig W. Brown*[1]; [1]Northwest Aluminum Technologies, 1080 W. Ewing Pl., Ste. 202, Seattle, WA 98119 USA

Processes in molten salts, such as those associated with aluminum electrolysis, are difficult to study in-situ. This is because the environment is too harsh for most materials to withstand, so applicable techniques are limited. This paper presents a technique that extends the experimental options. Freezing the salts during electrolysis produces samples that can be studied by various means, including scanning electron microscopy. Examples presented were obtained in studies of low-temperature electrolysis with slurry electrolytes. Elemental maps generated from electron microprobe data obtained with such samples display evidence of mass transport phenomena near the electrodes.

8:55 AM
Error Analysis in the Measurement of Current Efficiency in Hall-Heroult Cells: *Guy Lawrence Fredrickson*[1]; [1]Hazen Research, Inc., 4601 Indiana St., Golden, CO 80401 USA

Upon cursory consideration, the determination of current efficiency (CE) for a Hall-HÈroult cell appears to be a deceptively simple task. After all, what else is required but the measurement of metal production as a function of the cumulative ampere-hours of current flow. However, when attempts are made to determine the CE of a single Hall-HÈroult cell, over a relatively short elapsed time (i.e., less than 48 hours), the data, more often than not, shows little accuracy, precision, or reproducibility. This paper discusses the statistical error, which is associated with the determination of CE, as a function of the various field and analytical measurements that are required for its calculation. A general statistical model is developed that can be applied to a number of different measurement techniques and strategies, and to any type and size Hall-HÈroult cell.

9:20 AM
Characterization of the Fluctuation in Anode Current Density and ìBubble Eventsî in Industrial Reduction Cells: Henrik Gudbrandsen[1]; *Nolan Richards*[2]; Sverre Rolseth[1]; Jomar Thonstad[3]; [1]SINTEF, Trondheim N-7491 Norway; [2]Consultant, 117 Kingswood Dr., Florence, AL 35630 USA; [3]Norwegian University of Science and Technology, Matls. Tech. & Electrochem., Sem Selandsv. 6, Trondheim N-7491 Norway

Whether the studies of CO_2 bubbles evolved from a carbon anode in an alumina reduction cell represents a less well understood aspect of the electrolytic process or a compelling aspect of the anode effect, the subject has attracted increased attention in recent years. In this work the character and impact of ìbubble eventsî were quantified for anodes of industrial cells. The data about the impact and frequency of the bubbles was derived from in situ measurements of the anode overpotential using a fast data logger. Oscillations in overvoltage were correlated with the dynamics of anodic current density and hence, variations in the screening and bubble coverage of the horizontal surfaces of the anodes.

9:45 AM
Measurements of the Anode Overvoltage in Industrial Alumina Reduction Cells: Henrik Gudbrandsen[1]; *Nolan Richards*[2]; Sverre Rolseth[1]; Jomar Thonstad[3]; [1]SINTEF, Trondheim N-7491 Norway; [2]Consultant, 117 Kingswood Dr., Florence, AL 35630 USA; [3]Norwegian University of Science and Technology, Matls. Tech & Electrochem., Trondheim N-7491 Norway

The anode overvoltage, η, in any industrial alumina reduction cell represents 10-15% of the total cell voltage. These laboratory results have been the basis for calculating the η in the mathematical modeling of total voltage for designing and setting the parameters for operating cells. An empirical allowance has often been added for the ìbubble resistanceî. In this presentation the anodic overvoltages were measured on both prebaked anodes with and without slots, as well as Soderberg anode in industrial cells using a robust, stable reference electrode that eliminated the ohmic drop across any bath layer. The ηís were determined over a range of current densities and alumina concentrations on anodes of different geometries and ranged 0.6 to 1.0 volt depending upon alumina concentration and extent of bubble coverage. Overvoltages were followed to anode effect. The actual values reported are higher than would be calculated from currently accepted mathematical models. Some attributions for these differences are presented.

10:10 AM Break

10:20 AM
Addition of Spent Potlining to Aluminum Reduction Cells to Produce High Si Alloys: *Bj rn Moxnes*[1]; HÂvard Gikling[1]; Halvard Kvande[2]; Sverre Rolseth[3]; [1]Hydro Aluminium, Tech. Ctr. ≈rdal, ÿvre ≈rdal NO-6885 Norway; [2]Hydro Aluminium, Metal Products, Oslo NO-0246 Norway; [3]SINTEF Materials Technology, Electrochem. & Ceram., Trondheim NO-7465 Norway

Tests with crushed potlining as feed material have been performed on laboratory cells and in industrial scale in S derberg cells. The purpose was twofold: To produce Si-rich alloys directly in the cells and to utilize spent potlining that otherwise has to be deposited as hazardous waste. The spent potlining also contains bath material and alumina that could be recovered. Carbon was separated from the collected spent potlining materiel before it was crushed to a powder that could be handled like normal alumina. Results from the laboratory tests showed

WEDNESDAY AM

that it took twice as long to dissolve the spent potlining in the bath compared to primary and secondary alumina. The S□derberg test cell was operated continuously for five month with a feed consisting of a mixture of alumina and spent potlining. The operational results were acceptable, the level of Si content in the aluminum exceeded 10%.

10:45 AM

An Improved Method for Removing Light Metals from Molten Aluminum in a Reduction Plant: *Nolan Richards*[1]; Helge O. Forberg[2]; [1]Consultant, 117 Kingswood Dr., Florence, AL 35630 USA; [2]Consultant, 5118 New Bedord Place, Marietta, GA 30068 USA

Both modern magnetically compensated and modernized cells using low ratio bath with quiet metal pads can produce aluminum with sodium content higher than preferred. Older potlines using LiF-modified bath as a means to enhance profitability, produce virgin Al with 10-20 PPM Li in addition to some Na. To improve both quality and efficiency in processing Al to obtain premium grades or meet specifications for certain products, the light metals should be removed before the tapping crucibles are loaded into holding furnaces. Technologies ranging from a bed of [charcoal + AlF_3] to treatment-in-crucible (TAC) with powdered AlF_3 added to a vortex for dispersion are being used to lower the concentration of light metals. Here, an improved TAC method is described based upon the efficient dispersion of powdered $NaAlF_4$ introduced on a carrier gas stream down a hollow impeller, which further disperses the fluoride, which, because of its higher vapor pressure, vaporizes and improves the contact with molten Al. The system includes other advantageous features. This process has the expectation for removing light metals more efficiently and will reduce concentrations of Na and Li to 1 PPM within 10-12 minutes.

11:10 AM

Anode Reaction in Aluminium Electrolysis Prior to and During Anode Effect: *Hongmin Zhu*[1]; Jomar Thonstad[2]; [1]University of Science & Technology Beijing, Dept. of Physl. Chem., 30 Xueyuan Rd., Beijing 100083 China; [2]Norwegian University of Science and Technology, Matls. Tech. & Electrochem., Sem Selandsv. 6, Trondheim N-7491 Norway

Anode reactions in aluminium electrolysis prior to and during anode effect were studied by cyclic voltammetry and gas chromatography. Three voltammetric peaks at around 2.2 V, 2.6 V and 4.3 V were observed in low-alumina melts. The currents of the first two peaks increased sharply by the addition of alumina. In a highly acidic melt, a sharp rise in current was observed above 5.8V. CO was the dominant oxygen-containing gas in low-alumina melts. CO2 was only found at low potentials (< 4V). CF4 generation was detected at potentials above 2.8 V. The rate of CF4 generation was similar in the various melts, and it was in all cases low. It has been shown elsewhere that alumina depletion precedes a potential shift in the positive direction, to reach the potential region for generation of CF4. This is accompanied by a sudden drop in current, marking the onset of the anode effect.

11:35 AM

Technique and Mechanism of Aluminum Floating Electrolysis in Molten Heavy Na3AlF6-AlF3-BaF2-CaF2 Bath System: *Lu Huimin*[1]; *Yu Lanlan*[1]; [1]University of Science and Technology Beijing, Metallurgl. Eng. Sch., 30 Xueyuanlu Haidian, Beijing 100083 China

In this paper, the authors studied a new low temperature aluminum electrolysis process in 3000A experimental cell with Na3AlF6-AlF3-BaF2-CaF2 bath system. The influences of cathodic current density, electrolytic temperature and density differences between bath and liquid aluminum on current efficiency (CE) were studied; when electrolytic cryolite ratio was 1, w(BaF2) and w(CaF2) were 20mass% and 15mass%, respectively, CE reached 90.57% and specific energy consumption was 12.27kWh/kg Al. Because aluminum obtained floated on the surface of molten electrolyte, named this electrolysis method as low temperature aluminum floating electrolysis. The mechanism of aluminum floating electrolysis was also studied at 1023K and Al2O3 concentration of 5.8◊10-4mol/cm3 by use of potential sweep voltammetry at tungsten electrode. It was found that the reduction of aluminum was a simple and rapid process involving the exchange of three electrons: Al3+ + 3e = Al and the diffusion coefficient of aluminum ions was 1.893◊10-5cm2/s.

Aluminum Reduction Technology: Modernization

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Martin Segatz, VAW Aluminum Technology, Bonn D-53117 Germany; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday AM Room: 6B
March 5, 2003 Location: San Diego Convention Center

Session Chair: Jean Paul Aussel, Pechiney Aluminium Smelter, Voreppe Cedex 38341 France

8:30 AM

Research on the Instabilities in the Aluminum Electrolysis Cell: *Panaitescu Aureliu*[1]; Moraru Augustin[1]; *Panaitescu Ileana*[1]; [1]University ìPolitehnicaî of Bucharest, Electl. Eng., Spl. Independentei 313, Bucharest 77206 Romania

In order to reduce the specific electrical energy consumption of the aluminum electrolysis cells their instabilities were experimentally studied. The study was focused on the instabilities produced during the cellsí operation by reducing the anode-to-cathode distance below a certain value and on the instabilities due to the anode effects. The researches were performed in the electrolysis halls of the ALRO Slatina plant in Romania, on standard cells which operate without a vertical magnetic filed compensation and also on new designed cells with a global compensation of Lorentz forces. The method of study consist in the real time visualization of the molten metal surface shape. The experimental research didnít evidence the existence of traveling waves on the aluminum surface but only vertical local oscillations (Pinch effects). The MHD phenomena are due to the asymmetric distribution of Lorentz forces in the molten media. For the electrolysis cells designated in order to obtain the global compensation of the Lorentz forces the vertical oscillations of the molten metal surface are strongly reduced. These cells can operate with small anode to cathode distance and therefore average specific consumption of 12800 kWh/t were obtained. The anode effects are considered in this paper as instabilities in the cell operation. Anode effects appear in the area where the bath circulation velocity is small. In these places alumina concentration is low, and are small also the electrical conductivity of the bath and the anode current density. The results of our researches have also shown that the device for the visualization in real time of the current distribution in the electrolysis cells allows the identification of the most proper area for the alumina feeding.

8:55 AM

Process Modelling as a Key Factor in AP14 Retrofit at Aluminij d.d. Mostar: *Christian Droste*[1]; Ingo Eick[1]; [1]Hydro Aluminium Primary Metals, Process Modlg., Georg-von-Boeselager-Str. 25, 53117 Bonn Germany

In October 2001, Aluminij d. d. Mostar began a major upgrade of its 92,000-tpy smelter in Bosnia-Herzegovina. By introducing the latest potroom technology, the smelter became internationally competitive with respect to production costs, working conditions and environmental protection. The main part of the modernisation project consisted of converting 256 side-worked, prebake AP14 cells, originally built in 1981, to centre-worked, point-fed cells and installing a modern pot control system and a pneumatic alumina transport system. The hot change of the cells was completed in May 2002. The financial success of the project is closely related to a substantial increase in metal production after modernisation. Before embarking on the modernisation project, the expected production gains from converting the cells, increasing the amperage and improving current efficiency had to be forecast reliably. The limitations and bottlenecks of the existing technology were therefore evaluated carefully with the help of well-established process modelling tools. The results showed that, in addition to the conversion from side to centre operation, some modifications to the busbar system and pot lining were also necessary to meet the ambitious target of a 25% increase in metal yield.

9:20 AM

The 3D Modeling of MHD-Stability of Aluminum Reduction Cells: *S. A. Shcherbinin*[1]; A. V. Rozin[2]; S. Yu Lukashchuk[3]; [1]PSC Bratsk Aluminum Plant, Bratsk 665716 Russia; [2]Moscow State University, Inst. of Mech., 1 Michurinskii Pr., Moscow 119899 Russia; [3]Ufa Air Technological University, 12 K. Marx St. 450025 Russia

The wide range and complicated character of technical problems, facing ìRussian Aluminumî Company from the date of its foundation in 2000, formed the background of intensive development of the important sector of mathematical simulation. In particular, the simulation of magneto-hydrodynamic processes followed several directions. One of researches in the area of MHD-stability was initiated by ìRussian Aluminumî Bratsk Aluminum Plant. The aforementioned MHD-model allowed to estimate 3D non-stationary hydrodynamic processes in metal and bath, including the cell wall/anode region. Two-parameter turbulent model was used. The electromagnetic fields, gravity and surface tension force were taken into account. The model response to external impacts (such as current jumps during anode effects) were used for estimation of MHD-stability of aluminum reduction cell. Different variations of the cell configuration, bath properties, busbar arrangements were analyzed in order to study the dynamical behavior of MHD-parameters.

9:45 AM

Twenty Years of Progress at HINDALCOís Aluminium Smelter: *S. C. Tandon*[1]; R. N. Prasad[1]; [1]Hindalco Industries, Ltd., Renukoot-231217, Sonebhadra 231217 India

Hindalcoís aluminium smelter was initially started in 1962 with 20,000 tpy capacity and technology was based on 1950ís generation pots incrementally from 20,000 tpy to 120,000 tpy by early 80ís by adding number of potlines but the basic design of pots remained unchanged. In view of the energy price hike and to keep pace with developments in aluminium smelting, Hindalco undertook serious efforts to modernise its smelting facilities in mid 80ís by retrofitting new technologies and since then continuing improvement has been the normal mode of operation in the smelter. Now Hindalco smelting capacity stands at 275,000 tpy and two potlines are under construction to complete brownfield expansion to achieve 350,000 tpy by end of 2003. Today plant is competitive in the world market being one of the lowest cost producer of primary metal, operating at 94.5% current efficiency and 14.2 DC KWH/Kg specific energy consumption with pot life of 2800 days. This paper traces the evolution of the retrofit technology that has been applied to smelter which includes revised cathode and insulation design, mechanised aluminium feeding and conveying, computerised potline control, bath chemistry, anode improvements, environment protection and waste utilisation.

10:10 AM Break

10:20 AM

Conversion of the AP 14 Potline at Aluminij d.d. Mostar from Side to Point Feeding: *Volker von der Ohe*[1]; Bruno Cale[2]; Zdravko Cuturic[3]; [1]Hydro Aluminium Primary Metals, Eng., Koblenzer Str. 122, Neuss 41468 Germany; [2]Aluminij d.d. Mostar, Dir. Technique, ul 25 Studeni bb, Mostar Bosnia Hercegowina; [3]Aluminij d.d. Mostar, Mgr. Potroom, ul 25 Studeni bb, Mostar Bosnia Hercegowina

Without the loss of production, the 256 side-worked AP 14 cells at Aluminij d.d. Mostarís aluminium smelter were converted to centre-worked point-feeder cells in a hot-change operation lasting only 9 months. 2 cells were converted every day between September 2001 and May 2002. Each cell was equipped with three point-feeders, two crust breakers, an ELAS process controller and network to a central control room for observation and adjustment of the pot parameters. An alumina transport system was installed and the gas cleaning system upgraded to meet international environmental protection standards and working conditions. Auxiliary plants, such as the anode rodding shop and compressor station and operating procedures at the smelter, e.g. cavity cleaning and anode covering, were also adapted to fit the new technology. To ensure optimum performance, it was essential to fine-tune pot operation and pot tending to the upgraded technology and give the Aluminij workers intensive training. The main technological challenge of the project was to replace each pot superstructure in the potroom within a few hours. This involved widening the anode centre channel and separating the anodes in the hot bath to allow the installation of point-feeders while keeping the power cut-off time of the cell as short as possible. Subsequent production results show that such a pot conversion can be controlled technically and technologically and that a project of this kind can be carried out cost effectively.

10:45 AM

The Development of GAMIís Large Aluminum Reduction Cell: *Yang Yi*[1]; Yao Shihuan[1]; [1]Guiyang Aluminum and Magnesium Design and Research Institute (GAMI), Guiyang 550004 China

The stability of an Aluminum reduction cell is the main factor affect the cellís current efficiency. We introduce the method of increase the stability of cells which used in GAMIís 230KA, 280KA, 320KA and 350KA pre-bake side-by-side cellís design, for example, optimizing the bus-bar configuration, modifying the cellís length-width ratio, increasing the size of anode and cathode, i.e., as well as the model and real value of velocity and magnetic fields of cell, are presented in the paper.

11:10 AM

Development of 230KA Prebake Pots: *Leng Zhengxu*[1]; Li Hongpeng[1]; Li Yun[1]; Yang Caohong[2]; Yang Yi[2]; Yao Shihuan[2]; [1]Guizhou Aluminum Smelter, Guiyang 550014 China; [2]Guiyang Aluminum and Magnesium Design and Research Institute, Guiyang 55004 China

In this paper, we introduce 230KA experimental pre-bake cell in Guizhou Aluminium Plant. We describe the structure and magnetic of the cell and the new lining material. Via the two years practiced, it is proved the stability of the cell. The cellís current efficiency reached 94%, DC cost less 13400Kw/t-Al, AE<0.1. The new type cell will be used in the new project of Guizhou Aluminum Smelter.

Carbon Technology - III

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Amir A. Mirchi, Alcan Inc., Arvida Research and Development Centre, Jonquiere, QC G7S 4K8 Canada; Don T. Walton, Alcoa Inc., Wenatchee Works, Malaga, WA 98828-9784 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday AM	Room: 6D
March 5, 2003	Location: San Diego Convention Center

Session Chair: Christian Dreyer, Aluminium Pechiney, St. Jean De Maurienne 73303 France

8:30 AM

Exhaustion, Pneumatic Conveyor and Storage of Carbonaceous Waste Materials: *Paulo Douglas Santos Vasconcelos*[1]; [1]Albras AlumÌnio Brasileiro SA, Carbon Plant, Rod. PA 483, km 2, Vl de Murucupi., Barcarena, Par· 68447000 Brazil

In smelters that use prebaked anodes, operations such as butt cleaning, butt and anode reject crushing and grinding, handling of coke packing material in the bake furnace, floor sweeping and discharge of dust from bake furnace cranes causes significant problems with the high generation of carbon dust and, consequent environmental pollution. To control the dust pollution is a very difficult task. Another problem, meanwhile, is how to convey and store the dust collected. This paper presents the problem existing in the carbon plant of Albr·s, describing the pneumatic conveying system developed by the Carbon Plant Engineering department, and the storage of the dust collected in the carbon plant. Examples of application of the system to use the carbon dust to sale to the cement industry, or to generate water vapor for the carbon plant.

8:55 AM

The Impact of the Firing and Control System on the Efficiency of the Baking Process: Jaffar Ameeri[1]; *Khalil M. Khaji*[1]; *Wolfgang K. Leisenberg*[2]; [1]Aluminium Bahrain, Carbon & Metal Services, Manama Bahrain; [2]Innovatherm, Carbon Div., Leisenberg GmbH + Co., Roter Lohweg 22, Butzbach, Hessen D-35510 Germany

Based on the recent conversion of two existing anode baking furnaces at ALBA, Bahrain, the paper will demonstrate the improvements that can be achieved by using new features of firing and control system for baking furnaces. The evaluation covers the most relevant parameters of the baking process such as quality, consistency, fuel efficiency and the combustion of volatiles. In addition to the advanced built-in features of the new system, which already offered a distinct improvement over the existing one, fine tuning of the system by the carbon plant management in close co-operation with the system designer enabled the team to adapt the advanced features of the new system to the actual furnace performance, in order to unleash the full potential of the system. New features, such as prediction of anode temperatures and the modification of the firing strategy have been tested.

9:20 AM

Performance Enhancement of Carbon Anode Baking Plant at Hindalco, India: *Subhash Chandra Tandon*[1]; [1]Hindalco Industries Ltd., Reduction Plant, PO Renukoot, UP 231217 India

Hindalco is Indiaís largest integrated aluminium producer and amongst the worldís lowest cost producers. It is a flagship company of Aditya Birla Group, Indiaís second largest business house, committed to Global benchmarking. The company is known for its culture of innovation, experimentation and creativity. Under the same spirit, performance enhancement plan of Anode Bake Furnaces was undertaken to optimise fuel oil consumption, improved fire cycle time, enhanced baked anode quality and longer refractory life. The objectives were achieved by regulating the heat distribution in furnaces through appropriate process control systems with customised software. The control strategy was developed from Hindalcoís long experience in plant operation. Hamilton Research and Technology Pvt., Ltd. (HART), India, Hindalcoís development Partner and Hindalco had jointly conceptualized and implemented the system. This paper describes the technology and strategy used. The results of the technology and improvement on plant performance have been presented with actual plant data.

9:45 AM

Creep and Sodium Expansion for Cathode Carbon Materials: Experimental Results, Constitutive and Numerical Modelling: Alexander Zolochevsky[1]; J□rund G. Hop[1]; Guillaume Servant[1]; Trygve FoosnÊs[1]; *Harald A. ÿye*[1]; [1]Norwegian University of Science and Technology, Dept. of Chem., Sem SÊlands veg 12, Trondheim N-7491 Norway

The creep strain and sodium expansion of semigraphitic cathode material have been measured on solid and hollow cylinder samples under various values of applied external pressure. Experimental results have been obtained for various current density values using the Rapoport-Samoilenko-type apparatus. A constitutive model for cathode carbon materials which is able to reproduce the relationship between the sodium expansion and time as well as the relationship between creep strain, external pressure and time during the Rapoport-Samoilenko-type test has been proposed. Parameters required in the proposed model have been calculated from experimental data by a least-square minimisation process. The constitutive model has been extended to a three-dimensional case and has been implemented into the commercial finite element code ANSYS. The distributions of sodium concentration, coordinate components of stress and von Mises equivalent stress in the semigraphitic cathode block with time have been obtained.

10:10 AM Break

10:20 AM

Mathematical Modeling of Sodium Expansion in Prebaked Aluminium Reduction Cells: *Yang Sun*[1]; Qianpu Wang[2]; Morten S□rlie[3]; Harald A. ÿye[1]; [1]Norwegian University of Science and Technology, Dept. of Chem., Trondheim N-7491 Norway; [2]National Research Council Innovation Centre Canada, 3250 E. Mall, Vancouver V6T 1W5 Canada; [3]Elkem Aluminium ANS, Rsrch., PO Box 8040 Vaagsbygd, Kristiansand S N-4675 Norway

A two dimensional transient mathematical modeling of an aluminium reduction cell was developed to study thermomechanical movements and stresses during heat up and early operation using the ANSYS program. In this nonlinear finite element model, material non-linearity and influence of temperature on the mechanical properties were taken into account. Then sodium concentration distribution with time in the cathode carbon was calculated, and the stress distribution and deformation were calculated and analyzed.

10:45 AM

Reactivity of Carbon Materials Against Metallic Sodium: *Noboru Akuzawa*[1]; Ryu Nakajima[1]; Masashi Yamashita[1]; Masahide Tokuda[2]; Chihiro Ozaki[2]; Kenji Ohkura[2]; [1]Tokyo National College of Technology, 1220-2 Kunugida, Hachioji, Tokyo 193-0997 Japan; [2]SEC Corporation, 3-26 Osadano, Fukuchiyama, Kyoto 620-0853 Japan

Carbon blocks being used as the cathode of aluminum-reduction industry are subject to severe degradation under practical operation conditions. One of possible reasons of the problem may be the formation of intercalation compounds induced by the attack of sodium on the cathode blocks. The present investigation aims at clarifying the reactivity of carbon blocks, prepared from petroleum needle cokes, with different heat-treatment temperatures (HTT) against metallic sodium. HTT-2800 carbon gave intercalation compound (NaCx) with the stage 8 structure of identity period (Ic) of 2.8 nm. The electrical resistivity of NaCx showed metallic temperature dependence with a unique hysteresis between 200 and 350 K. HTT-1000 carbon, on the contrary, fixed a large amount of sodium leading to the stage 2 structure (Ic=0.79 nm). Resistivity measurements suggested that the reaction with sodium brought about a fatal damage on the carbon matrix.

11:10 AM

Comparative Characterisation of Graphitised and Graphitic Cathode Blocks: Amir A. Mirchi[1]; *Weixia Chen*[1]; Michel Tremblay[1]; [1]Alcan Inc., Arvida R&D Ctr., 1955 Mellon Blvd., PO Box 1250, JonquiÈre, QuÈbec G7S 4K8 Canada

Graphitised and graphitic blocks have been used in many plants over the last decade in order to reduce the cathode voltage drop and energy consumption as well as to adapt to increased amperage. Graphitised and graphitic blocks have higher thermal and electrical conductivities and lower resistance to erosion as compared to the amorphous and semi-graphitic blocks. A better understanding of the properties of these blocks and their equilibrium between cell productivity and life would allow their use to be considered for certain cell technologies. The present study, aimed at building a portrait of each type of block, compares the mechanical, thermal and electrical properties of the commercial graphitised and graphitic cathode blocks.

Cast Shop Technology: Grain Refining

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; David H. DeYoung, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday AM Room: 6C
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Clark Weaver, Alcan Inc., Jonquiere, QC G7S 4L2 Canada; Paul Cooper, London & Scandinavian Metallurgical Company, Rotherham, S. Yorkshire S60 1DL Great Britian

8:30 AM

Grain Refinement in Secondary Al-Si Casting Alloys: M. N. Binney[1]; *D. H. StJohn*[1]; A. K. Dahle[1]; J. A. Taylor[1]; E. C. Burhop[2]; P. S. Cooper[3]; [1]The University of Queensland, Div. of Matls. Eng., CRC for Cast Metals Mfg., Brisbane, Qld 4072 Australia; [2]Metallurg Aluminium, Newfield, NJ 08344 USA; [3]London & Scandinavian Metallurgical Company, Ltd., Metall. Aluminium, Rotherham S60 1DL England

Secondary aluminium alloy castings generally have inferior ductility compared to primary alloy castings because the higher levels of impurity elements tend to cause formation of brittle intermetallic phases. It is possible to reduce the detrimental effect of these impurities by grain refinement since it assists in reducing the size of particles. However, these impurities may also complicate the efficiency of the grain refinement treatment. This work investigates the effects of Cu and some typical impurities in secondary alloys on the grain size of the primary aluminium-silicon alloy A356. The addition of 3wt% Cu to the A356 alloy had little or no effect on grain size. However, a combined addition of Cu plus Fe, Cr, Zn and Mn to A356 to make a pseudo-secondary 319 alloy significantly increased the grain size. When the impurity elements Fe, Cr, Mn and Zn were added individually, Fe and Cr were identified as the elements primarily responsible for causing the increase in grain size.

8:55 AM

Characterisation of a New Generation of Grain Refiners for the Foundry Industry: *Paul Stephen Cooper*[1]; Angela Hardman[1]; Dave Boot[1]; Ed Burhop[2]; [1]London & Scandinavian Metallurgical Company, Ltd., Fullerton Rd., Rotherham, S. Yorkshire S60 1DL England; [2]Metallurg Aluminium, West Blvd., PO Box 768, Newfield, NJ 08344 USA

There have been a number of studies in recent years relating to the mechanisms of grain refinement, poisoning and fade of Al-Ti-B refiners. In addition there have been several new grain refining products introduced for the foundry sector, such as TiBAlloy and Strobloy, for which studies have also been performed, albeit in a more limited way. The new generation of grain refiners have a number of specific beneficial attributes, which distinguish them from the traditional Al-Ti-B refiners. These benefits are explored by characterising the refiner particle types in terms of composition, size, poisoning tendency and fade characteristics.

9:20 AM

Modeling of Microporosity Formation During Solidification of A356 Aluminum Castings: *Rong Ding*[1]; Daan M. Maijer[1]; Steve L. Cockcroft[1]; Jane Howard[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada

The control of microporosity in aluminum alloy castings produced for the automotive industry posses a substantial challenge because of high cast surface and mechanical performance quality standards. Typically, trial and error methodologies have been used to optimize casting design and casting process parameters, such as cycle time and cooling programs. Microporosity formation during the solification of A356 aluminum castings has been modeled using finite element analysis. Microporosity has been calculated using two fundamentally based approaches. The first is a semi-empirical criterion which takes into account the effect of hydrogen and inclusion content. The second approach employs a critical nucleation pressure and microporosity growth kinetics. The model was developed and implemented in the commercial finite element software, ABAQUS. Calculated results are compared with the experimental data from lab scale test castings and industrial scale wheel castings. The various parameters influencing the formation of microporosity, e.g. the initial hydrogen content, the ingot temperature and the volume shrinkage, are discussed.

9:45 AM
Microstructure Evaluation and Microporosity Formation in AlSi7MgO3 Alloys: *Petru Moldovan*[1]; Gabriela Popescu[1]; Gheorghe Dobra[2]; Carmen Stanica[2]; [1]Polytechnic University Bucharest, Dean of Matls. Sci. & Eng. Fac., Splaiul Independentei 313, Sect. 6, Bucharest 77206 Romania; [2]S. C. ALRO S.A. Slatina, 116 Pitesti St., Slatina 0500 Romania

This article combines the experience of foundry men with research of the theory and prediction of porosity formation. The influence of flux treatment was determined and also the influence of Sr and Sb refining of AlSiMgO.3 alloy over the porosity formation was studied. The densities and densities index (D.I.) were determined of the air and vacuum solidified samples using for this purpose excellent equipment for taking liquid samples and solidifying in vacuum and in air (VAC TEST SYSTEM) and also a device called DENSITY TERMINAL from testing of materials quality laboratory on Materials Science and Engineering Faculty. To establish the mechanism of porosity formation and to evaluate the gaseous porosity, respectively the shrinkage porosity, was used the quantitative microstructure analysis: metallographic microscope BX6M, video camera KPM1 and BUEHLER OMNIMET EXPRESS with a program in 4.0 versions from S.C. ALRO S.A. Slatina. From experimental data the density index (D.I.) and from microstructure analyses it was successfully realize the establishing of the porosity formation mechanism in AlSi7MgO.3 alloy and the influence of different factors over the gaseous porosity and shrinkage porosity.

10:10 AM Break

10:20 AM
Growth-Restriction Effects in Grain Refinement of Aluminium: *Tom Quested*[1]; Lindsay Greer[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

The solute content in aluminium alloys affects grain refinement predominantly through the growth rates of solid grains, or by poisoning the nucleation on refiner particles. Decreases of as-cast grain size with solute content can be described in terms of a universal growth-restriction parameter Q. The fundamental definition of Q is examined. Solute effects, rather than thermal, predominate in growth restriction, even in commercial-purity alloys. The linearity of Q with solute content and its additivity for combined solutes are tested, making use of the thermodynamics software MTDATA. This facilitates separation of growth restriction and poisoning in interpreting measured grain-size trends. Solute addition can accelerate growth, but not in a regime relevant for refinement of conventional alloys; increases in grain size reported to accompany some increases in solute content are attributable entirely to poisoning. Finally, effects of solute diffusivity in, and flow of, the liquid are considered.

10:45 AM
A Comparison of the Family of AlTiB Refiners and their Ability to Achieve a Fully Equiaxed Grain Structure in DC Casting: *Wolfgang A. Schneider*[1]; T. E. Quested[2]; Lindsay Greer[2]; Paul S. Cooper[3]; [1]Hydro Aluminium Germany, R&D, Georg von Boeselager Str. 25, Bonn 53117 Germany; [2]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK; [3]London & Scandinavian Metallurgical Company, Ltd., Fullerton Rd., Rotherham S60 1DL UK

There are a number of grain refiners, based on the AlTiB system, used in the DC casting process. These vary in both concentration and Ti:B ratio. An in depth experimental programme has been performed to compare some of these refiners in the DC casting process. One of the key success criteria used, was to compare the quantity of TiB2 particles from the different refiners, required to achieve a fully equiaxed grain structure. The key role of TiB2 particles is highlighted. The results are discussed in relation to recent theories and modelling on the key characteristics of the TiB2 particles. In particular the particle size distributions of the different refiners are reviewed in relation to the theoretical ideal.

11:10 AM
Effect of the Grain Refinament Process on the Kind and Morphology Intermetallics Phases in the Unalloying Aluminium: *Tomasz Stuczynski*[1]; [1]Institute of Non-Ferrous Metals, Light Metals Div., Pilsudskiego 19, Skawina 32-050 Poland

Effect of the grain refinement process on the kind and morphology of intermetallic phases existing in the two varieties of the unalloying aluminium (Fe+Si=0,5 and Fe+Si=1,0) solidifying at difference cooling rate have been presented. The five kinds of intermetallic phases type AlxFeySiz and AlTFe(Si) have been classified taken as criterion their chemical composition. The main conclusion is, that the grain refinement process conducted by AlTi5B1 (100 ppm Ti) addition promotes to growth the intermetallic phases type AlTFe(Si) (Si/Fe=0,13-0,04) as eutectics grains.

11:35 AM
Production of Al-B Master Alloys from Boron-Bearing Salts Using Different Techniques: *Ibrahim Hamed Aly*[1]; M. A. Shaheen[2]; Abdel-Nasser M. Omran[3]; [1]Minia University, Cheml. Eng. Dept., El-Minia Egypt; [2]Suez Canal University, Metallurgl. Dept., Suez Egypt; [3]R&D Aluminum Company of Egypt, Nag-Hammady Egypt

An Al-B alloy containing about 5% boron has been obtained by reacting of potassium fluoroborate (KBF4) with aluminum. The parameters affecting the reaction process such as: temperature, time, mixing speed, potassium fluoroborate to aluminum weight ratio and particle size were studied. The experiments were carried out in three techniques: first by using KBF4 only, second adding mixture of KBF4 and granular aluminum and third using mechanical alloying. The results indicated that the second technique is considered to be the best one. Microstructure examinations and X-ray diffraction tests were carried out on the produced alloys.

Computational Phase Transformations: Effect of Internal Defects and External Fields

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Wednesday AM — Room: 11B
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: John W. Morris, University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA; Yunzhi Wang, The Ohio State University, Dept. Matls. Sci. & Eng., Columbus, OH 43210 USA

8:30 AM Invited
Phase Field Microelasticity in Modeling of Multi-Phase, Multi-Dislocation and Multi-Crack Systems: Yu U. Wang[1]; Yongmei M. Jin[1]; *Armen G. Khachaturyan*[1]; [1]Rutgers University, Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA

Phase Field Microelasticity as a particular case of the Phase Field theory has been developed to describe evolution of stress-generating structural inhomogeneities in solids. These inhomogeneities can be coherent new phase precipitates or structural domains formed as a result of diffusional transformations (decomposition, ordering, polymorphic transformation) or diffusionless martensitic transformations. The greatest advantage of the phase field approach is that it does not impose any a priori constraints on possible options in morphological changes during microstructure evolution - the system itself ëíchoosesíí the optimal way of microstructure rearrangement. Recently, this theory has been extended to the cases of arbitrary multi-void, multi-crack, and multi-dislocation systems spontaneously developing under applied stress. This extension also allows one to characterize an elastically inhomogeneous system regardless of a value of the elastic modulus misfit. In particular, it extends the theory and method to the case of polycrystalline systems. Being combined, these theories and corresponding models can realistically describe simultaneous evolution of three-dimensional nano- and mesoscale microstructures in phase transformation, plastic deformation, and fracture as well as their response to the applied stress. The latter permits to find a relation between structural and mechanical properties of the system. The development of this theory and availability of high-speed computers open the way to a realistic 3D simulation of technologically important materials, such as intermetallic alloys, shape memory alloys, and structural ceramics. Examples of 3D simulations of decomposition in decomposing intermetallics, martensitic transformation in polycrystals of Shape Memory Alloys as well as evolution of self-multiplied cracks and dislocations will be presented.

9:00 AM Invited

Microstructure Evolution Under the Influence of Internal Defects and External Constraints: Yulan Li[1]; Shenyang Hu[1]; Zikui Liu[1]; *Long-Qing Chen*[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

Phase transformations and the accompanying microstructure evolution can be significantly influenced by the presence of internal defects and external constraints. This talk will be focused on the effect of structural defects such as dislocations and mechanical constraints. A three-dimensional phase-field model will be presented. It may be applied to predicting the domain structure evolution in an elastically anisotropic single crystal film, taking into account of the presence of both arbitrary spatial distribution of dislocations and mechanical constraints imposed by a substrate. A number of examples involving structural transformations in thin films with coherent or semi-coherent interfaces will be discussed. It is shown that the mechanical constraint by a substrate can drastically change the relative volume fractions of differently orientated domains and the domain-wall orientations while dislocations can affect the nucleation, domain wall motion, and the final spatial distribution of domains.

9:30 AM Invited

Modeling Topological Defects in Non-Equilibrium Elastic Media: *Ken Elder*[1]; [1]Oakland University, Physics, Rochester, MI 48309-4487 USA

Topological defects created in non-equilibrium processing often play a key role in determining the physical and mechanical properties of materials. Understanding the creation and kinetics of such defects is a challenging theoretical problem since these defects correspond to a limit in which classical elasticity theory becomes singular. In this talk I would like to discuss the use of continuum or phase fields to model the role of topological defects in non-equilibrium elastic media. In particular I would like to focus on a new model that treats elastic and plastic deformations exactly in media with multiple grains and free surfaces.

10:00 AM

Phase Field Modeling of Metal Hydride Growth in a Plastically Deforming Matrix: *Christopher R. Krenn*[1]; Adam J. Schwartz[1]; Wilhelm G. Wolfer[1]; Yongmei M. Jin[2]; Armen G. Khachaturyan[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci. Direct., PO Box 808, L-353, Livermore, CA 94551 USA; [2]Rutgers University, Dept. of Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA

Charging many metal films or powders with hydrogen results in the growth of second phase metal-hydride particles. A large volume difference between hydride and metal can induce plastic deformation and result in a significant hysteresis upon removal of the hydrogen. For a palladium hydride system (with an 11% volume change), we model this hysteresis using a three-dimensional phase field model that incorporates a simple model of plasticity. We examine the effects of yield strength and relative nucleation rate on the hysteresis and on the microstructural evolution. Finally, we discuss the applicability of our models for other phase transformations involving large plastic accommodations. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

10:20 AM Break

10:35 AM Invited

Pattern-Directed Phase Separation in Stressed Thin Films: *William C. Johnson*[1]; Steven M. Wise[1]; Jerome Favergeon[1]; [1]University of Virginia, Dept. of Matls. Sci. & Eng., PO Box 400745, Charlottesville, VA 22904-4745 USA

Simulations of phase decomposition in binary and ternary thin films on patterned substrates, are performed using coupled Cahn-Hilliard and phase field equations. Phase formation, growth, coarsening, and the resulting two-phase microstructures are strongly influenced by the substrate geometry and composition, film thickness, the composition dependence of the surface energy density, composition and epitaxial strains, film and substrate elastic constants, and alloy composition. Phase decomposition on a patterned substrate can yield self-organized structures of small particles of various sizes and spacings. Particle sizes are often much smaller than the pattern dimensions and the technique may provide a method for developing monolayers of periodic nanostructures from an initially homogeneous alloy. This work is supported by the US National Science Foundation through the Center for the Nanoscopic Design of Materials and Grant DMR-9902110.

11:05 AM Invited

Elastic Domains in Epitaxial Layers: *Alexander L. Roytburd*[1]; Julia Slutsker[2]; [1]University of Maryland, Matls. & Nucl. Eng., College Park, MD 20742 USA; [2]National Institute of Science and Technology, Matls. Sci. & Eng. Lab., 100 Bureau Dr., Gaithersburg, MD 20899 USA

Theory and phase-field modeling of formation of polydomain structures in epitaxial layers due to relaxation of misfit stress are presented. These structures consisting of the periodic arrangement of domains of the product phase (twins) are results of phase transformations (1st or 2nd orders) of epitaxial layers. Engineering of constraint (changing misfit and the crystallographic orientation of epitaxial layers) is used to design different structures of elastic domains. Results of modeling are compared with homogeneous theory and analytical solutions for simplest polytwin structures. Available experimental data are discussed.

11:35 AM Invited

Effects of Elastic Inhomogeneity on the Diffusional Microstructure Evolution in Two Phase Solids: *Ingo Schmidt*[1]; [1]Institut fuer Mechanik, TU-Darmstadt, Hochschulstr. 1, Darmstadt 64289 Germany

Many of the properties of metallic materials are caused by their microstructure. Specifically, the properties of phase separated alloys are closely related to the morphology of the precipitate particles. A particular microstructure is typically the result of a phase transformation which is interrupted at a certain stage to produce desired material properties, a well known example being single crystalline super alloys. There, the presence of a lattice misfit between the phases results in a morphological evolution driven by the competition between the elastic strain energy and the interfacial energy. The paper deals with the simulation of the diffusional evolution of particle morphologies in orthotropic media, with special attention directed to the influence of the elastic inhomogeneity in the system. Two different approaches are followed in studying the problem, namely the search for equilibrium morphologies of a given number of precipitates in an infinite matrix and the simulation of the temporal evolution of such systems. In particular, shape bifurcation- and rafting phenomena as well as the stability of particle arrangements are addressed. Continuum descriptions assuming a sharp interface between the phases are evaluated with boundary and finite element methods in two and three dimensions. The results are compared with a ëdiscrete atomí model in which the phases are represented by a large number of elementary particles of two different species which are interconnected with linear springs. Here, the evolution of the morphology is simulated using a Monte-Carlo method. This work is joint with Dr. R. Mueller and Prof. D. Gross.

Defects and Deformation of Crystalline Solids - in Honor of Dr. Man H. Yoo - V

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
Program Organizers: Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Department, Houghton, MI 49931-1200 USA; Sean R. Agnew, University of Virginia, Materials Science and Engineering Department, Charlottesville, VA 22904-4745 USA; K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA

Wednesday AM March 5, 2003 — Room: 17A — Location: San Diego Convention Center

Session Chairs: Richard Wagner, GKSS, Geesthacht 21502 Germany; Kwai Chan, Southwest Research Institute, Dept. of Matls. Sci., San Antonio, TX 78228-0510 USA

8:30 AM Invited
Association Between Twinning and Fracture in Two-Phase Titanium Aluminides: *Fritz Appel*[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str., Geesthacht D-21502 Germany

Mechanical twinning has long been recognized as an important deformation mode in γ -based titanium aluminides. The mechanism apparently compensates for the lack of independent slip systems that can operate at comparable stresses and, thus, supports the plasticity of polycrystalline materials. On the other hand, the octahedral planes of γ -TiAl serve as slip planes, twin habit planes and cleavage planes. Thus, blocked slip may easily lead to fracture. This complex association of twinning and fracture is investigated in the present paper. The major areas of the study are: twin nucleation and growth, effects of solid solution and precipitation hardening and stability of twin structures. The study is based on conventional and high resolution transmission electron microscope observations of defect structures in deformed TiAl alloys.

8:55 AM
Twinning as a Crack Tip Deformation Mechanism During Fracture: *Diana Farkas*[1]; [1]Virginia Tech, Matls. Sci. & Eng., Blacksburg, VA 24061 USA

We report atomistic studies of the deformation mechanisms in the crack tip region during low temperature fracture of fcc and bcc metals. The large scale simulations use empirical potentials to study the crack tip response. Twinning is observed in both fcc metals such as Al and bcc metals such as Fe, in the favorable crystallographic orientations. In the case of Al the twins are formed by the emission of Shockley partial dislocations in adjacent slip planes of the (111) type. In the case of bcc Fe, the twins are also formed by the emission of partial dislocations in adjacent slip planes of the {112} type. The importance of this mechanism is discussed.

9:15 AM Invited
Grain Boundary Character Distributions of Zr and Ti Grain-Refined Through Equal Channel Angular Pressing: S. Yu[1]; H. S. Ryoo[1]; *S. K. Hwang*[1]; [1]Inha University, Sch. of Matls. Sci. & Eng., 253 Yonghyun-Dong, Nam-Gu, Incheon 402-751 Korea

Commercially pure Zr and Ti were grain-refined through equal channel angular pressing (ECAP). With variation of the die design and the processing parameters such as the temperature and subsequent annealing, varieties of microstructures resulted in. Up to a strain of 1.8 was obtained by using a 90?a/20?a die, which resulted in a grain refinement to about 200nm with repeated ECA-pressing. The two metals showed a considerable difference in the initial texture. However, ECA-pressing converted the texture into a basically same preferred orientation, a strong (0001) texture inclined to the specimen axis. The grain boundary character distribution of both metals was such that the high angle boundaries were predominant while the fraction of the low angle boundaries was slightly higher than that in a random microstructure. Both the texture and the misorientation distribution of the severely deformed Zr and Ti persisted during the subsequent exposure to an elevated temperature.

9:40 AM
Ab Initio Calculations of the Structure and Energy of the (11-21) Twin Boundary in HCP Metals: *James R. Morris*[1]; Yiying Ye[1]; Man H. Yoo[2]; [1]Iowa State University, Metal & Ceram. Scis., Ames Lab., USDOE, Ames, IA 50011 USA; [2]Oak Ridge National Laboratory, Metall. & Ceram. Div., Oak Ridge, TN 37831-6115 USA

The (11-21) twin boundary is important in controlling the deformation behavior of HCP metals under tensile strain along the c-axis, and can affect the polycrystalline ductility of a material. We have examined two possible structures of this boundary using accurate ab initio calculations. The two structures have different symmetries: one is a pure mirror twin, the other has a glide plane symmetry. Both structures have been seen experimentally. While empirical potentials predict different energies for these structures, we find the surprising result that the two structures are nearly identical in energy, for all materials examined. We compare the results with calculations from various empirical potentials. The close energy is consistent with the experimental observations that both may occur. The results suggest that the structure found from deformation experiments may be due to the dynamics of twinning, rather than from energetic considerations of the boundary.

10:00 AM
Monazite Deformation Twins: *Randall S. Hay*[1]; David B. Marshall[1]; [1]AFRL/MLLN, Bldg. 655, Area B, WPAFB, OH 45433-6533 USA

Polycrystalline monazite (monoclinic $LaPO_4$) was deformed at room temperature by a spherical indenter. Deformation twins were identified by TEM in 70 grains. Twinning on (100) was by far the most common. Twinning on (001) and (120) was less common, and twinning on (122) was rare. (120) twins were kinked. The twinning modes on these planes were inferred from the surface expression of twinning shear, predictions of classical deformation twinning theory, and other crystallographic considerations. Twinning modes appear to be strongly influenced by the required atomic shuffling. Twin abundance may be related to twin interface energy. The three most common twins have low strain to low Sigma CSLs. Abundance of (100) twins may be related to a low-energy interface structure of a layer of xenotime of Ω unit cell thickness. The (001) twin forms a xenotime interface structure similar to that for (100). Minimal shuffling requires the twin interface to be a glide plane with displacement vector R=Ω[010] for (001) twins, and R=Ω[011] for (122) twins.

10:20 AM Break

10:40 AM Invited
Nucleation of Twins in Face Centered Cubic Metals: Y. Q. Sun[1]; *P. M. Hazzledine*[2]; [1]University of Illinois, Dept. of Matls. Sci. & Eng., Urbana, IL 61801 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

The nucleus for a twin, even one as small as a single loop of Shockley partial dislocation, may be modeled as an Eshelby inclusion embedded in a matrix of untransformed material. Shear nuclei of this type, consisting of one or many dislocation loops, interact with each other attractively through their elastic fields. Because of this interaction, the nucleation process becomes collective and therefore more probable than it would be in the absence of the interaction. In this paper, a model is presented of collective nucleation of twins. The model shows that, at a given temperature, the onset of twinning is a sudden process which occurs at a critical stress. The value of the critical stress falls gradually, and almost linearly, with increasing temperature.

11:05 AM
Linking Electronic and Dislocation Parameters to Fracture Resistance in Metals and Intermetallics: *Kwai S. Chan*[1]; [1]Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA

The possible linkage of electronic and bonding variables to material parameters that describe dislocation nucleation, dislocation mobility, and the fracture resistance of metals and intermetallics are investigated in this paper. Using appropriate analytical methods, the d-level energy, bond order, unstable stacking energy (USE) and the Peierls-Nabarro (P-N) barrier energy are computed for a variety of intermetallics either with or without alloy additions. Similar calculations are also performed for Nb-Ti-Al solid solution with either the bcc or B2 structure. The computed values of d-level energy, bond order, USE and P-N barrier energies are compared and correlated against fracture toughness data to establish (1) relations between electronic variables, dislocation parameters, and fracture resistance, and (2) means for improving the fracture resistance through alloying additions. Work supported by AFOSR through Contract No. F49620-01-C-0016, Dr. Craig S. Hartley, Program Manager.

11:25 AM Invited
Investigation on the Superdislocation Dissociation Widths in $L1_0$ TiAl: Xiping Song[1]; *Guoliang Chen*[1]; Y. R. Ren[1]; [1]University of Science and Technology Beijing, State Key Lab. for Adv. Metals & Matls., Beijing 100083 China

The paper systematically investigates the effects of anisotropic elasticity, superdislocation dissociation configurations, deformation temperatures as well as lamellar interfaces on the superdislocation dissociation widths at the condition of the same stacking fault energy (SFE). The calculation results showed: 1. In the anisotropic elasticity condition both the superdislocation dissociation widths and the difference in the dissociation widths between edge and screw superdislocations become smaller than that in the condition of isotropic elasticity. 2. The superdislocation dissociation widths of the superlattice intrinsic stacking fault (SISF) in the 3-fold or 4-fold superdislocation dissociations configurations were calculated to be smaller than that in 2-fold dissociation. 3. The superdislocation dissociation widths were also affected by the non-coplanar dissociation configuration. The influence of non-coplanar dissociation configuration is related to the Burgers vectors of dissociated superdislocations. 4. The experimental results showed that deformation temperatures had little effect on the superdislocation dissociation widths. 5. The dissociation width of superdislocations is affected by the dislocation location. The dissociation width became wider from the twin interfaces of γ-γ_T type than inside of the γ lamellar. On the basis of the investigation the accuracy of the measurement of SFE by using weak beam TEM technology was improved significantly, and the dislocation mobility is also discussed.

11:50 AM Invited
Encounters with Twinning: *Terence E. Mitchell*[1]; [1]Los Alamos National Laboratory, Struct./Prop. Relations, MST-8, MS G755, Los Alamos, NM 87545 USA

Man Yoo has made numerous contributions to the science of twinning. The present author has also had occasional but regular encounters with the phenomenon of twinning starting with his doctorate studies. These have included both deformation twinning in simple fcc, bcc and hcp metals (copper, tantalum and hafnium respectively), in ceramics such as sapphire and in intermetallics such as the C15 Laves phase HfV_2. Transformation twinning has also been encountered in a variety of ceramics such as zirconia, silicon carbide and perovskite-based high temperature superconductors and in a variety of intermetallics such as CuAu and $MoSi_2$. Twinning has also been found in incommensurate structures of $ReSi_{2-x}$. More recently twinning has been encountered in plutonium and uranium, two materials of vital importance to Los Alamos. Stories of these serendipitous encounters will be woven together to address the question of why twinning is so widespread but why it is not universal.

Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong: Grain Size Effects

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Marc Andre Meyers, University of California-San Diego, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; George T. Gray III, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA; Kenneth S. Vecchio, UC San Diego, Dept of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA

Wednesday AM   Room: 16B
March 5, 2003   Location: San Diego Convention Center

Session Chair: Lawrence E. Murr, University of Texas at El Paso, Metallurgl. & Matls. Eng., El Paso, TX 79968-0520 USA

8:30 AM
Grain Size Dependence of the Flow Stress of Metals from Millimeters to Nanometers: *Hans Conrad*[1]; [1]North Carolina State University, Matls. Sci. & Eng., Raleigh, NC 27695-7907 USA

The effect of grain size ranging from mm to nm on the flow stress of metals at low and intermediate homologous temperatures is considered. Three grain size regimes are identified: Regime I (d=10^-6-10^-3m), Regime II (d=10^-8-10-^6m) and Regime III (d= -10^-8m). Grain size hardening occurs in Regimes I and II and grain size softening (inverse Hall-Petch effect) in Regime III. Regimes I and II correspond to intragranular dislocation activity, with Regime II being characterized by the absence of dislocation cells and twins. Regime III is characterized by the absence of intragranular dislocations and results from grain boundary shear produced by the applied stress alone.

9:00 AM
Grain Size Effects on the Strength and Work Hardening in HCP Metals Titanium, Zirconium, and Cadmium: *Placid Rodriguez*[1]; [1]Defence Research & Development Organisation, Recruit. & Assess. Ctr., Lucknow Rd., Timarpur, Delhi 110054 India

Ronald W. Armstrong is a pioneer in combining Hall-Petch analysis with thermal activation strain rate analysis (TASRA). One of his early predictions was that for HCP metals that deform by basal slip, the Hall-Petch slope K would be dependent on temperature and strain rate. (To maintain continuity across grains, more than one slip system has to be activated and temperature dependence of flow stress will be controlled by the more difficult prism or pyramidal plane.) The situation is different for zirconium and titanium; multiple slip is possible on the primary prism plane and K is expected to be independent of temperature and strain rate. This paper presents the results of combining TASRA (strain rate change, stress relaxation and temperature cycling experiments) with Hall-Petch analysis in titanium; K is shown to be independent of temperature and strain rate. The results are compared with earlier results on cadmium and zirconium. The results on zirconium are also analyzed to highlight the influence of dynamic strain ageing on the Hall-Petch parameters.

9:30 AM
Computational Modeling of the Mechanical Response of Polycrystalline and Nanocrystalline Metals: *Hsueh-Hung Fu*[1]; David J. Benson[1]; Marc A. Meyers[1]; [1]University of California-San Diego, Mechl. & Aeros. Eng., La Jolla, CA 92093-0411 USA

The grain size effect on the mechanical response of polycrystalline metals was investigated computationally. A phenomenological constitutive description is adopted to build the computational crystal model. The material is envisaged as a composite; the grain interior is modeled as a monocrystalline core surrounded by a mantle grain boundary with a higher work hardening rate response. Both a quasi-isotropic approach and a crystal plasticity of the Pierce-Asaro-Needleman type are used to simulate the grain interiors. The grain boundary is modeled by a Voce equation. Elastic and plastic anisotropy are incorporated into this simulation. An Implicit Eulerian Finite Element Formulation with von Mises plasticity or rate dependent crystal plasticity is used to study the nonuniform deformation and localized plastic flow. The computation predictions are compared with experimentally determined mechanical response of copper with grain sizes ranging from millimeters to the nanocrystalline domain. Research supported by the National Science Foundation and the ARO MURI.

9:45 AM
On the Strength and Ductility of Nanocrystalline Materials: *Marek Doll·r*[1]; Anna Doll·r[2]; [1]Miami University, SEAS, Oxford, OH 45056 USA; [2]Miami University, Mfg. & Mechl. Eng. Dept., Oxford, OH 45056 USA

In the early nineties, there was a widespread belief that nanocrystalline materials would exhibit the unique combination of room temperature ductility and ultrahigh strength. However, the hopes have not materialized. In the present study, we analyze plastic deformation and strengthening mechanisms in the nanometer range, based on results of our own research studies on nanocrystalline NiAl and the literature review. The analysis indicates that the nanomaterials are stronger than their coarse grain counterparts but not as strong as predicted using the conventional Hall-Petch approach. Also, in the nanometer range, grain size softening with decreasing grain size seems to be prevalent as a result of the predominance of diffusional mechanisms and/or the deterioration of sample quality. In intermetallics, and ceramics, the enhancement of diffusional mechanisms results in measurable, though limited, room temperature ductility. In nanocrystalline metals the increase in ductility provided by diffusional mechanisms is insignificant compared to the loss of dislocation controlled ductility.

10:00 AM
Grain Size Effects in the Fracture of Nanocrystalline Fe: A Simulation Study: *Diana Farkas*[1]; Antoine Latapie[1]; [1]Virginia Tech, Matls. Sci. & Eng., Blacksburg, VA 24061 USA

Crack propagation studies in nanocrystalline bcc iron samples with grain sizes ranging from 6 to 12 nm are reported at temperatures ranging from 100K to 600K using atomistic simulations. For all grain sizes, a combination of intragranular and intergranular fracture is observed. Mechanisms such as grain boundary accommodation, grain boundary triple junction activity, grain nucleation and grain rotation are observed to dictate the plastic deformation energy release. The effects of grain size on the observed deformation mechanisms is discussed.

10:15 AM
Molecular-Dynamics Study of Mechanical Deformation in Nanocrystalline Metals: *Kai Kadau*[1]; Timothy C. Germann[2]; Peter S. Lomdahl[1]; Brad Lee Holian[3]; Dirk Kadau[4]; Peter Entel[5]; Magnus Kreth[5]; Frank Westerhoff[5]; Dietrich E. Wolf[5]; [1]Los Alamos National Laboratory, Theoretl. Div., T-11, MS B262, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Appl. Physics Div., X-7, MS D413, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Theoretl. Div., T-12, MS B268, Los Alamos, NM 87545 USA; [4]Gerhard-Mercator-Universitaet Duisburg, Theoretische Physik, Lotharstrasse 1, Duisburg 47048 Germany; [5]Gerhard-Mercator-Universitaet Duisburg, Theoretische Tieftemperaturphysik, Lotharstrasse 1, Duisburg 47048 Germany

Nano-crystalline metals, in which the size of grains is in the nanometer range, exhibit mechanical properties different from ordinary polycrystalline materials, thus these materials are of technological interest. One important example is the increasing hardness with decreasing grain size due to dislocation immobilisation at the grain boundaries, known as the Hall-Petch effect. However when decreasing the grain size below a critical value, sliding processes between the grains decrease the hardness which is called the reverse Hall-Petch effect. We report on molecular-dynamics simulations of tensile loading of nanophase Cu and Al modelled by an embedded-atom method (EAM) potential. Usage of two different preparation methods of the nano-phase material allow us to compare mechanical properties for different sample qualities: A Voronoi-constructed polycristal exhibit nearly no pores and has different mechanical properties compared to a material that is sintered under pressure and temperature from nano particles.

10:30 AM Break

10:45 AM
Characterization of Nanocrystalline and Microcrystalline Copper Consolidated Using Equal Channel Angular Extrusion: Mohammed Haouaoui[1]; *Ibrahim Karaman*[1]; K. Ted Hartwig[1]; [1]Texas A&M University, Dept. of Mechl. Eng., College Station, TX 77845 USA

Materials with ultrafine grains and nanostructures (<100 nm) have attracted considerable interest because of their unique properties as compared with conventional materials. Although this class of materials seems to offer new opportunities for small scale applications, utilizing them in large scale structural applications is still a challenge due to the difficulty of fabricating nanocrystalline materials in bulk. The present work is focussed on fabrication of full density bulk micro and nanocrystalline materials from powder precursors using equal channel angular extrusion (ECAE). The initial powder sizes that were consolidated were 50 nm, 150 nm and 45 micron. Different processing routes was selected for comparison purposes and to determine the best processing route for specific end microstructures. The density of consolidates is compared to the fully dense theoretical value as a first assessment of consolidation performance. The microstructure is characterized for different ECAE routes and number of passes. Indentation techniques are utilized to measure the hardness. The stress strain responses are determined by tension and compression tests at room temperature. The effect of initial powder size and resulting consolidate grain size on the mechanical properties are discussed in view of the Hall-Petch relationship. SEM is utilized to quantify the residual porosity. Mechanical behavior of the consolidates is compared with. In this talk, some of these experimental observations will be presented in comparison with microcrystalline consolidates and with severely deformed grain refined pure copper. This study helps to clarify the relationship between different ECAE processing parameters, mechanical properties and the microstructure of nanocrystalline copper.

11:00 AM
The Effect of Grain Size on Low Cycle Fatigue of Polycrystalline Al-4.5wt.%Cu Alloy: *Aezeden Omar Mohamed*[1]; Yassir Fouad El-Madhoun[1]; M. N. Bassim[1]; [1]University of Manitoba, Dept. of Mechl. & Industl. Eng., 15 Gillson St., Rm. 356, Eng. Bldg., Winnipeg, Manitoba R3T 5V6 Canada

The effect of grain size on low cycle fatigue of Al-4.5wt.%Cu alloy was studied. Two different grain sizes, 90mm and 150mm, were tested at room temperature. The fatigue response of the alloy was evaluated macroscopically in terms of cyclic stress strain curve and microscopically in terms of appearance of cyclic slip bands. The cyclic stress strain curve exhibited a plateau region where the saturation stress remained constant with plastic strain. Grain size of 90mm exhibited lower cyclic saturation stress and longer plateau than grain size of 150mm. This result contradicts the Hall-Petch relationship of grain size effect. Microscopic observations using optical and scanning electron microscope revealed the presence of persistent slip bands (PSBs) where the strain is highly localized. It was shown that the 90mm grain size contains a higher volume fraction of PSB than the 150mm grain size. Such higher volume fraction of PSBs would result in lower saturation stress since these slip bands act as soft regions within the matrix of cyclically deformed metals.

11:15 AM
Grain Size Effects on Deformation Behavior: A Two-Dimensional Discrete Dislocation Simulation: *James R. Morris*[1]; S. Bulent Biner[1]; [1]Iowa State University, Metal & Ceram. Scis., Ames Lab., USDOE, Ames, IA 50011 USA

The evolution of the flow stress for grain sizes ranging from 2 to 16 microns under shear deformation was simulated using two dimensional discrete dislocation dynamics. The analyses were confined to a single slip system and to the collective behavior of a large number of edge dislocations. Flow stress values increased with decreasing grain size and correlated with grain size with a classical Hall-Petch relationship $d^{-1/2}$, or in the form of d^{-1}. The flow stress values for different grain sizes unified to a single curve when expressed as a function of the dislocation density normalized by the grain size. It was observed that dislocation pileups can activate neighboring dislocation sources and also shutdown the active dislocation sources. This work was performed for the US Dept. of Energy by Iowa State University under contract W-7405-Eng-82. and supported by the Office of Basic Sciences.

11:30 AM
A Critical Assessment of the Grain Size Effects on Fatigue Deformation and Fracture of a Metastable Austenitic Stainless Steel: K. Bhanu Sankara Rao[2]; Sardari Lal Mannan[2]; *Placid Rodriguez*[1]; [1]Defence Research & Development Organisation, Recruit. & Assess. Ctr., Lucknow Rd., Timarpur, Delhi 110 054 India; [2]Indira Gandhi Centre for Atomic Research, Kalpakkam 603 102 India

The effects of grain size (75, 300 and 700 mm) on LCF life, cyclic stress response, and deformation and damage mechanisms of a type 304 stainless steel in the temperature range of 300 to 1023K were investigated through strain-controlled fatigue tests employing strain amplitudes in the range ± 0.25 to 1.25%. The cyclic deformation modes and damage mechanisms were significantly influenced by grain size and the temperature and strain amplitude employed during LCF test. The combined influence of temperature and grain size on cyclic stress response and life is explained on the basis of slip character, martensitic transformation, dynamic strain ageing, creep deformation, deformation induced precipitation and oxidation in their respective temperature domains of predominance. Hall-Petch relationship is valid at 823K and 923K between half-life stress and grain size; it is found not valid at 300K in this metastable steel due to the occurrence of a pronounced phase change from austenite to martensite.

11:45 AM
Grain Size Effects on Fatigue: *Shankar M.L. Sastry*[1]; [1]Washington University, Mechl. Eng., CB 1185, One Brookings Dr., St. Louis, MO 63130 USA

The effect of grain size on fatigue behavior was investigated in polycrystalline Ti-6Al-4V, dispersion strengthened Cu, precipitation strengthened Al, and low carbon steel. Ultra fine grain sized samples were produced by severe plastic deformation processing by equal channel angular extrusion and concurrent or post deformation recrystallization annealing. A reduction in grain size from 20 to 2 mm resulted in an improvement of fatigue life by three orders of magnitude and 30% increase in fatigue strength in Ti-6Al-4V. Similar but less dramatic improvements were observed in other materials.

Friction Stir Welding and Processing II: Friction Stir Joining: Structures & NDE

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Thomas J. Lienert, University of South Carolina, Mechanical Engineering Department, Columbia, SC 29208 USA; Rajiv S. Mishra, University of Missouri-Rolla, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Wednesday AM Room: 7B
March 5, 2003 Location: San Diego Convention Center

Session Chair: Ali A. Merati, National Research Council Canada, Inst. for Aeros. Rsrch., Ottawa, Ontario K1A 0R6 Canada

8:30 AM
Enabling Technologies for Manufaturing Metallic Cryogenic Tanks: *Ray Miryekta*[1]; Carolyn Russell[2]; [1]Boeing, Integrated Defense Sys. USA; [2]Marshall Space Flight Center, Huntsville, AL 35812 USA

Engineers and scientists from NASA and Boeing are exploring the feasibility of the developmental aluminum technologies related to materials and related processes necessary for manufacturing metallic cryogenic tanks. This team currently is in the process of developing manufacturing technologies suitable for processing aluminum-lithium alloys. Feasibility of using these alloys is greatly dependent on their optimized properties required for the above application. Extensive effort is being directed towards identifying a suitable joining process with superior capabilities, quality, and minimum properties loss inherent to the joining process characteristics. Friction Stir Welding (FSW) technology is being seriously pursued. To date, the preliminary weld test results for welds made using conventional FSW equipment have higher mechanical properties over equivalent fusion welds. Similar studies in the areas of friction stir plug-welding (FSPW) technologies were also conducted by different groups of engineers. Results of these activities showed that a local rework can be performed by push or pull plug-welding methodologies. This talk will discuss some of this ongoing work.

8:55 AM
Monitoring of Friction Stir Welding Process Using Acoustic EmissionñA Preliminary Study: *Changming Chen*[1]; *Radovan Kovacevic*[1]; [1]Southern Methodist University, Rsrch. Ctr. for Adv. Mfg., 1500 International Pkwy., Ste. 100, Richardson, TX 75081 USA

Acoustic emission (AE) generated during friction stir welding (FSW) is detected and a preliminary study is being conducted in order to investigate the possibility of applying this sensing technique for an in-process monitoring of FSW. Using two, static transducer sensors well-coupled with the workpieces, the AE signals are acquired for various welding parameters: the tool rotational speed, the traverse speed and down force, the probe, the tool with a probe, and the tool without a probe. In addition to the normal samples, two butted 6061 aluminum alloy plates with three, equal-spaced gaps made of two notches aligned along the butt joint of the parts are also used for the AE investigation. The AE signals are characterized by the hit rate vs. amplitude, and the rise time and energy vs. time. The analysis of the AE signals provides useful indicators of the tool transient movement and weld quality during FSW, and can be used to identify tool penetration, tool pullout and material defects formed during FSW.

9:20 AM
New Developments of the Ultrasound Phased Array and Eddy Current Arrays Technologies for the Evaluation of Friction Stir Welds: *Colin Bird*[1]; AndrÈ Lamarre[2]; [1]TWI Ltd, Granta Park, Great Abington, Cambridge CB1 6AL UK; [2]R/D Tech, Industl., 505 Blvd. du Parc-Technologique, QuÈbec G1P 4S9 Canada

The use of friction stir welding techniques (FSW) to assemble thin aluminum plates in aerospace applications brought about the need for a high-resolution, non-destructive testing technology to find and characterize the small defects, as kissing bonds, that may occur when using FSW. Phased array ultrasonics and eddy current arrays have been applied to FSW with success. This paper will discuss phased array ultrasonics and eddy current arrays and its application to friction stir welding of thin aluminum plates. Emphasis will be on detection of kissing bonds with both techniques.

9:45 AM
A New Method of Obtaining the Part-Through Fracture Toughness (KIe): *Bahram (Bob) Farahmand*[1]; [1]Boeing Technical Fellow, 5301 Bolsa Ave., Huntington Beach, CA 92647 USA

This work reports on a new approach for estimating the part-through fracture toughness, KIe. The KIe is used to calculate the wall thickness in life analysis of the base and weld regions of pressurized tank. It can be estimated through the ASTM-E740 and its value depends on specimen thickness, crack size, and the characteristic of the material. Improper value of KIe can yield unacceptable results. Smaller KIe value results in higher wall thickness, and a larger KIe value can provide smaller thickness, which can cause premature failure of the tank. Therefore, it is recommended to estimate a meaningful KIe for life analysis that is most suitable for direct application to design. That is, a KIe test that simulates the service condition and without violating the linear elastic fracture mechanics assumptions. The KIe versus plate thickness for several surface crack specimens were investigated with this approach. Results were plotted for base and friction stir weld metals.

10:10 AM Break

10:20 AM
Materials Fracture Properties Estimation by Eliminating Physical Testing: *Bahram (Bob) Farahmand*[1]; [1]Boeing Technical Fellow, 5301 Bolsa Ave., Huntington Beach, CA 92647 USA

In material selection study of aircraft or aerospace components, fracture toughness value and fatigue crack growth rate data are important parameters for service life analysis. When calculating the number of cycles to failure, both quantities must be available through ASTM testing standards, which are costly and time consuming. The proposed analytical approach can provide fracture toughness and fatigue crack growth rate curve by using only static parameters. The energy absorption rate at the crack tip is derived from Griffith concept and used to calculate fracture toughness. This quantity is used to establish region III of the da/dn curve. The threshold region was estimated through the Kitagawa concept. Two additional points were estimated in region II and were used to establish the Paris constants. Several alloys were selected and results were compared with this approach. Excellent agreement between experimental data and analytical method were found. The application of this concept will result in significant saving.

10:45 AM
Friction Stir Welding of Steels: *Tracy W. Nelson*[1]; Carl D. Sorensen[1]; Colin Sterling[1]; Scott M. Packer[2]; [1]Brigham Young University, Mechl. Eng., 435 CTB, Provo, UT 84602 USA; [2]Advanced Metal Products, 2320 North 640 W., W. Bountiful, UT 84087 USA

Friction stir welding (FSW) is a well established joining process for welding aluminum and other lower melting temperature metals. The application of this process to steels and stainless steels has primarily been limited by the availability of suitable tool materials. This paper will present the result of FSW in hi-hard armor steel. FSW were successfully made using polycrystalline cubic boron nitride (PCBN) tool material which exhibited negligible wear after 5 meters of weld. Transverse metallographic samples indicate excellent weld quality. Post weld transverse tensile properties were in excess of 70% of the base metal yield and tensile strengths. More detailed characterization of tool life along with weld microstructure and mechanical properties will be presented.

11:10 AM
FSW of Titanium Turbine Engine Components: *Timothy J. Trapp*[1]; [1]Edison Welding Institute, Navy Joining Ctr., 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

This abstract describes a USAF Metals Affordability Initiative Project being perfromed by GE Aircraft Engines and EWI to develop, demonstrate, and deploy friction stir welding for joining titanium engine components. The goal of this project was to qualify FSW procedures for titanium alloy applications. Other goals included improved joint strength, production of welds that were free of tool debris, and reduced manufacturing cost and distortion. To achieve these goals, significant advancement in FSW tool design and materials were required. The project developed new FSW tool designs and materials, developed welding procedures for dissimilar titanium alloy combinations, and provided metallurgical and mechanical property data. This presentation will provide a brief overview of the project and potential applications. The presentation will discuss in detail the FSW tool and procedure development efforts, the FSW microstructure and mechanical properties, the effect of pre and post weld heat treatment on microstructure and properties.

11:35 AM
FSW of the Advanced Amphibious Assault Vehicle: *Timothy J. Trapp*[1]; [1]Edison Welding Institute, Navy Joining Ctr., 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

This abstract describes a Navy MANTECH Project to develop and demonstrate FSW for joining Aluminum 2519-T87 armor on the Advanced Amphibious Assault Vehicle (AAAV). Alloy 2519, which is known for high strength and superior ballistic performance, is being used on the Marine Corpsí AAAV. General Dynamics Land Systems, the AAAV Program Office, and EWI conducted the project. The goals of this project were to produce welds with equivalent or greater strength and ductility compared to conventional arc welds, to successfully pass the ballistic shock test, to develop procedures to reduce weld distortion and manufacturing costs. Efforts included development of robust FSW tools, of a FSW distortion process model, and of procedures for groove, corner, and t-joint configurations. This presentation will provide a brief overview of the project and discuss in detail the weld joint designs, resultant microstructures and mechanical properties, and ballistic test results.

12:00 PM
Metallurgical and Mechanical Properties of a Friction-Stir Welded Aluminium Alloy 6056 T4 and T6 Conditions: L. Roldo[1]; C. Schilling[1]; M. Chludzinski[1]; *J. F. dos Santos*[1]; T. Strohacker[1]; [1]GKSS Forschungszentrum GmbH, Inst. for Matls. Rsrch. Joining Tech., Max-Planck-Str., Geesthacht D-21502 Germany

The present work has been carried out as part of the European project WAFS ñ Welding of Airframes with Friction Stir and addresses the structure property relationships in a friction stir welded Al 6056 alloy produced with different energy inputs in T4 and T6 conditions. The microstructure has been investigated using electron microscopy. The local properties of the weld zone have been determined using microflat tensile testing and an optical deformation measuring system. Additionally, thermal cycles have been measured during welding in order to support the understanding of metallurgical transformations in the weld zone.

High Temperature Alloys: Processing for Properties: Wrought Alloys

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee
Program Organizers: Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA; Jacqui B. Wahl, Cannon-Muskegon Corporation, Muskegon, MI 49443-0506 USA

Wednesday AM	Room: 13
March 5, 2003	Location: San Diego Convention Center

Session Chair: Gerhard E. Fuchs, University of Florida, Dept. of Matls. Sci. & Eng. , Gainesville, FL 32611-6400 USA

8:30 AM
Processing of Cold-Rolled Nickel by Directional Recrystallization: *I. Baker*[1]; J. Li[1]; B. Iliescu[1]; A. Badmos[1]; H. J. Frost[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

The effects of hot zone velocity and temperature gradient ahead of the hot zone during directional annealing at 1273 K have been investigated for 10 mm thick, cold-rolled nickel sheets using both electron back-scattered patterns on a scanning electron microscope and optical microscopy. Some cold-rolled nickel specimens were first statically recrystallized at 643 K prior to directional recrystallization at 1273K in order to examine whether directional recrystallization at 1273 K was by primary or secondary recrystallization. It was shown that directional recrystallization to a columnar grain structure or, under the optimum conditions, a single crystal was always by directional secondary recrystallization. Columnar grains could be produced over a wide range of hot zone velocities (2-100 mm/h) when a large temperature gradient (1000°C/cm) was maintained ahead of the hot zone, the column width decreasing with increasing hot zone velocity. In contrast, for a low temperature gradient ahead of the hot zone, only equiaxed microstructures were produced. The results will be compared with simulations of the processing. Research supported by AFOSR grant F49620-00-1-0076 and NSF grant DMI9976509.

8:50 AM
An Investigation of Processing-Microstructure-Property Relationships of Inconel 718 Through Grain Boundary Engineering: *Carl Boehlert*[1]; Nate Eisinger[2]; Serkan Civelekoglu[1]; Gaylord Smith[2]; James Crum[2]; [1]Alfred University, Sch. of Ceram. Eng. & Matls. Sci., CEMS/McMahon Hall, 2 Pine St., Alfred, NY 14802 USA; [2]Special Metals Corporation, Huntington, WV USA

Since the development of Inconel 718 by H. L. Eiselstein almost 40 years ago, this nickel-based superalloy has gained wide industrial acceptance due to its properties, fabricability, and cost effectiveness. It is the most widely used superalloy, and its applications include ranging from rotating and static components in aircraft jet engines to high-temperature tooling for extrusion and shearing to components of nuclear reactors and space vehicles. Recently an effort to improve the elevated-temperature creep resistance of this alloy through grain boundary engineering has been initiated. Grain boundary engineering involves evaluation of the grain boundary character distribution (GBCD) as a function of processing. Grain boundary engineering of face-centered-cubic metals and alloys, including nickel-based superalloys, has shown that dramatic improvements in creep resistance are possible when the concentration of low-angle boundaries (LABs) and coincident-site lattice boundaries (CSLBs) is increased. This may be accomplished through sequences of cold rolling and annealing inducing strain recrystallization. Using electron backscatter diffraction (EBSD), the GBCD of Inconel 718 was evaluated after cold rolling 0, 10, 20, 30, and 40% followed by annealing at temperatures between 1610-1850∞F. After subsequent aging treatment, the alloy was tensile-creep tested and the influence of GBCD on creep rupture life and elongation was evaluated. In addition the room-temperature tensile strength, elongation, and hardness were evaluated. With increased cold rolling the concentration of LABs+CSLBs increased and this was correlated to increased tensile strength, hardness, and creep rupture life. The potential for optimization of properties for Inconel 718 through grain boundary engineering is discussed.

9:10 AM
Realistic Subscale Evaluations of the Mechanical Properties of Advanced Disk Superalloys After Supersolvus Quenching Heat Treatments: *Tim P. Gabb*[1]; Jack Telesman[1]; John Gayda[1]; Peter T. Kantzos[2]; William A. Konkel[3]; [1]NASA Glenn Research Center, 21000 Brookpark Rd., MS 49-3, Cleveland, OH 44135 USA; [2]Ohio Aerospace Institute, 21000 Brookpark Rd., MS 49-7, Cleveland, OH 44135 USA; [3]Wyman-Gordon Forgings, 10825 Telge Rd., Houston, TX 77095 USA

In order to fully utilize the higher temperature combustor and airfoil concepts under development, there is a need to increase the temperature capabilities of superalloy turbine disks. One approach to meet this goal is to modify the processing and chemistry of advanced alloys, while preserving the ability to use supersolvus heat treatments to achieve coarse grain microstructures. An important step in this effort is to understand the key high temperature mechanical properties of advanced alloys after such supersolvus heat treatments. However, it can be very expensive to screen the effects of alloy and process modifications on mechanical properties using full-scale, production disk shapes having thick bore and narrow rim sections. In this study, the mechanical properties of several advanced disk superalloys were compared using subscale pancakes after supersolvus heat treatments with realistic cooling paths. Selected results will also be compared to properties from large disks.

9:30 AM
Alternate Processing of Two Heat-Resistant Alloys for Improved Properties: *L. M. Pike*[1]; S. K. Srivastava[1]; [1]Haynes International, Eng. & Tech., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

A number of sheet applications exist in the aerospace and LBGT industries in which the operating temperatures are in the intermediate range (1000∞F to 1400∞F). These include pneumatic ducting, tailcones, thrust reverser parts, bellows, recuperators, etc. While stainless steels are often used in that temperature range, many of these applications require greater tensile and low cycle fatigue (LCF) strength. Heat resistant alloys such as 625 and HR-120Æ alloys offer substantially improved mechanical properties, but in many cases their processing is controlled to produce a microstructure which provides optimum properties in the high temperature range (1400 to 2000∞F) where creep strength is of primary concern. In this paper we will describe how alternate processing routes have led to the development of two alloy products (625SQÆ alloy and fine-grained HR-120 alloy) which have improved tensile and LCF properties in the intermediate temperature range.

9:50 AM Break

10:10 AM

Optimization of Oxide Dispersion Strengthened FeCrAl and Fe3Al Alloy Properties: *Bimal Kad*[1]; James Heatherington[1]; Claudette G. McKamey[2]; Ian G. Wright[2]; Vinod K. Sikka[2]; Rod R. Judkins[2]; Gaylord Smith[3]; Mark A. Harper[3]; [1]University of California-San Diego, 409 University Ctr., La Jolla, CA 92093-0085 USA; [2]Oak Ridge National Laboratory, M & C Div., MS-6114, Oak Ridge, TN 37831-6114 USA; [3]Special Metals Corporation, 3200 Riverside Dr., Huntington, WV 25705-1711 USA

Mechanically alloyed oxide dispersion strengthened (ODS) Fe-Cr-Al and Fe_3Al alloy thin walled tubes and sheets, produced via powder consolidation methodologies, are promising materials for eventual use at temperatures up to 1200°C in the power generation industry, far above the temperature capabilities of conventional alloys. Target end-uses range from gas turbine combustor liners to heat exchanger tubes. Grain boundary creep processes at service temperatures are the dominant failure mechanisms for such components. The processed ODS alloy microstructure consists of elongated grains parallel to the tube axis, a result of dominant axial metal flow which aligns the dispersoid particles and other impurities in the longitudinal direction. This dispersion distribution is unaltered by recrystallization treatments and the high aspect ratio grain shape typically obtained limits transverse grain spacing and consequently the hoop creep response. This represents a critical materials design and development challenge that must be overcome in order to fully exploit the potential of ODS alloys. We will describe our attempts to improving hoop creep in ODS-alloy components by manipulating the factors that dictate and control the recrystallization behavior. *Research sponsored by the Office of Fossil Energy, Advanced Research Materials Program, US DOE under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

10:30 AM

High Temperature Microstructural Stability of a MA/ODS Ferritic Alloy: *Michael K. Miller*[1]; David T. Hoelzer[1]; Suresh S. Babu[1]; Edward A. Kenik[1]; Kaye F. Russell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6136 USA

Mechanical-alloying (MA) of fine pre-alloyed metal and Y2O3 powders has been shown to produce oxide dispersion-strengthened (ODS) ferritic alloys with dramatically improved high temperature mechanical properties. Atom probe tomography has revealed that the improved high temperature mechanical properties are correlated with the presence of ultrastable 4-nm-diameter Ti-, Y- and O-enriched particles and significant enrichments of Cr, W, Ti, Y, O, C and B in the vicinity of dislocations. These ultrafine particles were stable during long term creep experiments and annealing at temperatures of up to 1300∞C. The Y and O levels in the ferrite matrix were significantly higher than the equilibrium levels. These results may be related to the O-Ti, O-W, O-Y and O-Cr solute atom interactions influencing solute diffusion. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Laboratory Directed Research and Development Program and the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

10:50 AM

Microstructural Refinement of Pure Nb by Severe Plastic Deformation: *K. Ted Hartwig*[1]; *Don Bryant*[1]; [1]Texas A&M University, Mechl. Eng., 319 Eng. Physics Bldg., College Station, TX 77843-3123 USA

Electron beam remelted and cast pure niobium was deformed at room temperature up to strains of 9.2 by multipass equal channel angular extrusion through a tool containing an abrupt 90° angle. The as-worked microstructure shows a progression from dense dislocation tangles to subgrains with dimensions on the order of several hundred nanometers at a strain of 4.6. Recrystallization temperature, recrystallized grain size and the uniformity of the recrystallized microstructure as a function of the level of prior cold work will be reported.

11:10 AM

Microtexture Analysis in a Forged Alpha/Beta Ti Alloy: Crystallographic and Morphological Aspects of Phase Constituents: *Dhriti Bhattacharyya*[1]; Gopal Viswanathan[1]; Rajarshi Banerjee[1]; David Furrer[2]; Hamish Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Ladish Company, 5481 S. Packard Ave., Cudahy, WI 53110 USA

Texture is an important component in Ti alloy development both in terms of processing and the mechanical properties. While the macrotexture is important for understanding the flow characteristics of the forging process, the microtexture is crucial for the mechanical properties. Especially, the crystallography of the alpha and beta phases and the orientation relationship between them have a prominent role in the microtexture and the mechanical properties of alpha/beta Ti alloys. In this study, the micro texture measurements have been obtained from selected (alpha+beta) and beta forged Ti-6246 alloy by Orientation Imaging Microscopy (OIM). The results indicate that in beta forged alloy the a/b colonies having close crystallographic relationship but morphologically different are present both within and across prior beta grain boundaries. TEM samples have been extracted from these special boundaries using Focussed Ion Beam (FIB) slicing technique and detailed analysis have been conducted. This analysis combined with the micro texture measurements from OIM will be presented and possible explanations for the observed phenomena given.

Hot Deformation of Aluminum Alloys: Processing, Structure and Property - I

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Shaping and Forming Committee

Program Organizers: Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA; Armand J. Beaudoin, University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; Balasubramaniam Radhakrishnan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6359 USA

Wednesday AM   Room: 6E
March 5, 2003   Location: San Diego Convention Center

Session Chairs: Amit K. Ghosh, University of Michigan, Matls. Sci. & Eng., Ann Arbor, MI 48109-2136 USA; Armand J. Beaudoin, Jr, University of Illinois at Urbana-Champaign, Dept. of Mechl. & Industl. Eng., Urbana, IL 61801 USA; Menno van der Winden, Corus Research, IJmuiden Techl. Ctr., IJmuiden 1970 CA The Netherlands

8:30 AM Invited

Friction Stir Processing for High Strain Rate Superplasticity: *R. S. Mishra*[1]; Z. Y. Ma[1]; I. Charit[1]; [1]University of Missouri, Dept. of Metallurgl. Eng., 218 McNutt Hall, Rolla, MO 65409 USA

Friction stir processing is a new solid state technique for microstructural modification in metallic materials. This has been used to develop very fine grained microstructure in various aluminum alloys. Friction stir processing leads to predominantly high angle grain boundaries. Combination of very fine grain size and high grain boundary misorientation leads to high strain rate superplasticity in many commercial aluminum alloys. However, abnormal grain growth limits the upper temperature in some aluminum alloys. Various metallurgical issues related to use of friction stir processing for high strain rate superplasticity will be discussed. The authors gratefully acknowledge the support of (a) the National Science Foundation through grant DMR-0076433 and the Missouri Research Board for the acquisition of a friction stir welding and processing machine, and (b) the National Science Foundation through grant DMI-0085044.

8:55 AM

Modeling, Analysis, and Validation of Friction Stir Welding Processes: *Abe Askari*[1]; Stewart Silling[2]; Blair London[3]; Murray Mahoney[4]; [1]The Boeing Company, Bellevue, WA 98008 USA; [2]Sandia National Laboratory, Albuquerque, NM 92647 USA; [3]California Polytechnic State University, Matls. Eng. Dept., San Luis Obispo, CA 93407 USA; [4]Rockwell Scientific Company, Thousand Oaks, CA 91360 USA

Friction Stir Welding (FSW) is a solid-state joining process that has found wide spread use in aerospace applications. Because FSW is an emerging technology, significant advances in innovation and technology transition are possible. Science-based simulation and modeling toolkits, once validated, enable the rapid expansion of FSW technology by providing long-term facility for process design and optimization. These models facilitate rapid innovations in such key areas as optimal pin and shoulder design, new joint designs, optimal tool and work-piece material, joining of dissimilar materials, and optimization of feed, speed, and power requirements. Boeing, in collaboration with Rockwell Science Center and Sandia National Laboratories, has developed predictive computational model for FSW. The model is based on a three-dimensional Eulerian code with complete thermo-mechanical coupling. The code models all the important physical effects with a minimum of assumptions. It includes frictional heating and energy dissipation due to plastic work. Thermal convection and conduction, including conduction into the tool and base-plate, are included. The Eulerian nature of the code permits very large strains to be modeled. It also allows for prediction of mixing between the work-piece materials. All relevant geometrical details, including pin screw threads and shoulder design are included in the model. The model also predicts detailed thermal and deformation histories that impact the final microstructure of the weld zone and can be exploited to improve fracture, fatigue, and stress corrosion properties of the weld. Detailed welding tests and post weld analysis in high-strength aluminum alloys are used to validate the computational models and improve understanding of basic aspect of FSW process. Placement of both tracers and thermocouples in the weld path, metallographic examination, and computed tomography are used to investigate metal flow dynamics and the mixing of material as well as thermal profile and history in the weld zone.

9:15 AM Invited
High-Temperature Mechanical Behavior of Cryomilled Nanostructured Al Alloys: *E. J. Lavernia*[1]; B. Q. Han[1]; F. A. Mohamed[1]; [1]University of California-Irvine, Dept. of Cheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

Deformation mechanisms of nanostructured materials have attracted intensive investigation recently. We use a processing called consolidation of cryomilled aluminum powders successfully to manufacture large amounts of bulk nanostructured or ultrafine grained aluminum alloys. In the present presentation, the cryomilling processing and the characteristic of microstructure of bulk cryomilled Al-7.5%Mg alloys were reviewed, and mechanical properties at elevated temperatures of cryomilled Al-7.5%Mg alloys, together with microstructure characteristics by transmission electron microscopy and X-ray diffraction patterns, were investigated. The correlation of microstructural characterization, temperature-dependence of strength, plastic deformation mechanisms was discussed in terms of dislocation activity.

9:40 AM Invited
Severe Plastic Deformation of an Al-6061 Metal Matrix Composite: *Yi Huang*[1]; Cheng Xu[1]; Minoru Furukawa[2]; Zenji Horita[3]; Terence G. Langdon[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matls. Sci., 3650 McClintock Ave., OHE430, Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Eng., Fukuoka 812-8581 Japan

Equal channel angular pressing (ECAP) is an ideal procedure for processing metallic alloys. This report describes the application of ECAP to an Al-6061 metal matrix composite. It is shown that the use of a solid solution treatment prior to ECAP provides the opportunity of performing ECAP at room temperature for up to five passes through the die. This paper discusses the mechanical properties after ECAP including the effect of a post-ECAP peak aging treatment.

10:05 AM
Development of a Homogeneous Microstructure in Pure Aluminum Processed by ECAP: *Cheng Xu*[1]; Terence G. Langdon[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-Channel Angular Pressing (ECAP) is a newly-developed process utilizing simple shear to refine the grains of materials without changing the dimensions of the samples. It has been found that aluminum alloys processed by ECAP achieve high tensile ductilities and hardness and there is even a potential for attaining superplasticity at high strain rates in some alloys. This report shows that the processing of pure aluminum by ECAP leads to a significant increase in the microhardness and there is a tendency to reach a greater homogeneity with the imposition of increasing strain. The tendency of developing a homogeneous microstructure is advantageous in industrial applications.

10:25 AM Invited
Large-Strain Softening of Aluminum in Shear: *M. E. Kassner*[1]; M. Z. Wang[1]; M. T. Perez-Prado[2]; S. Alhajeri[3]; [1]Oregon State University, Mechl. Eng., Matls. Prog., Rogers Hall, Corvallis, OR 97331-6001 USA; [2]CENIM, CSIC, Madrid Spain; [3]Kuwait Institute Tech, Kuwait City 7065 Kuwait

Pure aluminum deformed in pure shear at elevated temperature reaches a broad ìpeakî stress and then undergoes about a 17% decrease in flow stress with deformation with, roughly, 1-2 equivalent uniaxial strain. Beyond this strain the flow stress is approximately constant. The sources for this softening are unclear. The suggested basis includes texture softening, microstructural softening, enhanced dynamic recovery, and discontinuous dynamic recrystallization. Experiments were performed where specimens were deformed in torsion to various strains within the softening regime followed by compression tests at ambient and elevated temperature. Analysis of the compressive yield strengths indicate that the softening is most likely substantially explained by a decrease in the average Taylor factor.

10:50 AM
Mechanical and Material Responses of an Al-Zn-Mg-Cu Alloy at Intermediate Strain Rates: *Zhe Jin*[1]; Paul Wang[1]; [1]Alcoa Technical Center, Alcoa Ctr., PA 15069 USA

Hot torsion tests were conducted at strain rates from 10/s to 300/s and temperatures from 300F to 880F to study the mechanical and material responses of aluminum alloys over the friction stir welding conditions. The material used was a partially rolled Al-Zn-Mg-Cu aluminum alloy. All test samples were immediately quenched after testing to freeze the deformation structure. Flow softening was observed at lower temperatures for all strain rates studied. At high temperatures, however, the flow softening was insignificant. The flow stress was observed to drop monotonically with increasing temperature and increase with increasing strain rate. The fast decay of flow stress with temperature was seen to occur at 525F. The flow stress could be 1 GPa at the high strain rate and low temperature in this study. The deformed samples were characterized using optical and electron microscopy to understand the microstructure evolution during deformation. The observed mechanical response of the material was discussed based on the microstructure observations.

11:10 AM
Hot Rolling Textures in Al Alloys: *J. Hirsch*[1]; [1]Hydro Deutschland GmbH, R&D, Bonn 53014 Germany

The microstructure and texture evolution during hot rolling of aluminium alloys is described and analyzed for industrial process parameters like single-stand or multi-stand hot rolling. Systematic variations in texture intensity occur which depend in a complex form on the combination of a number of process parameters like temperature, strain, strain rate, interstand time. Simulation tools have been developed and applied that can help to clarify the correlations and to describe them on the basis of the physical processes involved. The evolution of microstructure and flow stress in several non-heat-treatable Al alloys during high temperature deformation was analysed experimentally in laboratory tests. They are quantitatively described and used in hot rolling simulation models for the prediction of metallurgical effects like the amount of recrystallization, grain size and texture formation. For hot-line gauge recrystallization textures the cube texture is the dominant feature characteristic for most Al alloys and of major technical importance for the control of anisotropy. It is shown how quantitative prediction of texture formation during hot rolling can be applied in practice for on-line process control and off-line material optimization strategies.

11:35 AM
Parameters Affecting Strain Rate Sensitivity Index of Al-Mg Alloy: *Jyoti Mukhopadhyay*[1]; [1]Hindalco Industries, Ltd., R&D, Renukoot, Dt. Sonbhadra, Uttar Pradesh 231217 India

The values of strain rate sensitivity index (m) of Al-Mg alloys for two different grain sizes (16 & 25 μm) were determined under uniaxial tests at ambient as well at high temperatures in the range of 523-723 K and strain rates of 0.69 to 1.4 x 10-2 S-1. The maximum values of strain rate sensitivity for both grain sizes were found to occur at 723 K and at a strain rate of 1.4 x 10-3 S-1. At high temperatures these values were approximated to be around 0.34 and 0.3 respectively. They were strongly positive, whereas negative strain rate sensitivity values at ambient temperature were also observed for both grain sizes. The negative strain rate sensitivity index strongly suggests that dynamic strain ageing mechanism is operative in Al-Mg alloys. Measurements of strain rate sensitivity (m) as a function of strain and temperature were also evaluated. The value of (m) for a grain size of (16 μm) as compared to 26μm was found to be higher for all parameters.

11:55 AM
Grain Refinement in an Al-Li-Mg-Sc Alloy During Intense Plastic Straining at 300°C: *Rustam Kaibyshev*[1]; Ksenya Saytaeva[1]; Fanil Musin[2]; Yoshinobu Motohashi[2]; [1]Institute for Metals Superplasticity Problems, Khalturina, 39, Ufa, Bashkortostan 450001 Russia; [2]Ibaraki University, Rsrch. Ctr. for Superplastisity, Nakanarusawa-cho, 4-12-1, Hitachi, Ibaraki 316-8511 Japan

Microstructural evolution has bee studied in an Al-Mg-Li-Sc alloy with an initial grain size of ~60 μm during equal-channel angular extrusion at a temperature of 300°C and a strain rate of $\sim 10^{-2}$ s^{-1}. It was established that the formation of ultrafine grains with an average size of ~0.9 μm takes place during intense plastic straining by subsequent evolution of microstructure via continuous dynamic recrystallization. At $\varepsilon \sim 1$, the structure has become subdivided into bands of elongated subgrains alternating with areas of equiaxed subgrains. With increasing strain the essentially equiaxed arrays of subgrains form and low angle boundaries gradually convert to true high-angle boundaries. After ε~4, the mixed arrays consisting of boundaries with low and high angle misorientation were observed. Fully recrystallized structure is evolved after a true strain of ~12. Mechanism of grain refinement is discussed.

International Symposium on Gamma Titanium Aluminides: Processing - IM, PM, and Sheet

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee

Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

Wednesday AM — Room: 6F
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Paul A. McQuay, Howmet Corporation, Whitehall, MI 49461-1895 USA; Lee S. Semiatin, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA

8:30 AM Invited

Melting and Casting of Gamma Titanium Aluminide Ingots: *J. R. Wood*[1]; [1]Allvac, R&D, 20/20 Ashcraft Ave., PO Box 5030, Monroe, NC 28111 USA

Allvac operates two plasma arc melt (PAM) hearth furnaces which have been used over the years for the melting and casting of gamma titanium aluminide (TiAl) ingots. The PAM furnaces operate with He as the carrier gas and are capable of casting round ingots from 150 mm (6 in) to 760 mm (30 in) diameter and weighing from 140 kg (300 lb) to 6000 kg (13,600 lb). Input materials consist of titanium sponge, master alloys and other alloying elements which are precisely weighed, blended and pressed into compacts for charging into the furnace by a drum feeder. A variety of alloys have been made ranging from basic gamma TiAl chemistries to more complex chemistries for specific applications. A description of the melting process in each furnace and various alloys produced is presented herein.

9:00 AM Invited

Status and Prospects of Gamma TiAl Ingot Production: *Volker Guether*[1]; Anita Chatterjee[1]; Helmut Kettner[1]; [1]GfE Metalle und Materialien, Nuremberg 90431 Germany

The development of an industrial production process for homogeneous gamma-TiAl ingots has enabled processing industries to introduce final parts with excellent properties based on wrought materials. Since the first use of TiAl components in racing car engines in 2000, the demand for ingots increased remarkably. The paper describes the present status of ingot production with the focus on quality aspects and gives an outlook for the midterm future. Improved ingot manufacturing technologies will be directly adjusted to the specific materials requirements and widen the field of gamma-TiAl applications. Based on this and under the assumption of cost reductions of semi-finished material due to the effects of a mass production and a more efficient manufacturing procedures, wrought gamma-TiAl components are expected to enter service in gas turbines, aircraft engines and premier class vehicles within the next 4-6 years.

9:30 AM

Microsegregation in Major-Alloyed Gamma TiAl Based Alloys: *Ze-Wen Huang*[1]; Wayne Voice[2]; Paul Bowen[1]; [1]The University of Birmingham, Dept. of Metall. & Matls., Elms Rd., Edgbaston, Birmingham B15 2TT UK; [2]Rolls-Royce plc, PO Box 31, Derby DE24 8BJ UK

Major alloying of the Ti-Al binary system, by adding refractory metals, has been applied to raise the liquidus temperatures for improving high-temperature strength, creep resistance and oxidation resistance. However, these additions exacerbate the already severe segregation problems facing cast ?x-TiAl alloys, and may cause pronounced partitioning of heavy elements from light elements in dendrites. As a result, significant microsegregation occurs during solidification. This study assesses the degree and causes of microsegregation. Work focuses on 100-mm diameter, 50-kg ingots produced by both plasma arc cold-hearth remelting and vacuum arc remelting. 20-mm diameter casting bars produced by induction skull melting are also assessed. The effects of subsequent HIPping and heat treatment on microsegregation have also been studied in detail. The influence of such microsegregation on tensile behaviour and fatigue strength is also considered in this paper.

9:50 AM Invited

Powder Metallurgy Processing of Gamma Titanium Aluminide: *C. F. Yolton*[1]; Young-Won Kim[2]; Ulrike Habel[1]; [1]Crucible Research, 6003 Campbells Run Rd., Pittsburgh, PA 15205-1022 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA

As gamma titanium aluminide alloys transition from research and development into production, processing issues become increasingly important. Gas atomization of prealloyed powder followed by hot isostatic pressing (HIP) to full density is a viable approach for the production of forging and rolling performs, and as well as as-HIP components. A distinct advantage of the powder metallurgy (PM) production route is the uniform and fine microstructure in preforms/components of any size. The first part of this paper will describe gamma titanium aluminide powder production and processing with emphasis on powder characterization and powder cleanliness. Microstructures of consolidated material will also be discussed. Boron is a common alloying element in gamma titanium aluminide alloys. Observations on the effect of boron on microstructure evolution as a function of processing will be presented. The effect of heat treatment on boride and carbide distribution will also be discussed. Thermally induced porosity (TIP) is a phenomenon, which can occur in all inert gas atomized powders. The effect of HIP temperature and time combinations and post HIP heat treatment conditions on the microstructure and occurrence of TIP in gamma titanium aluminide powders will also be discussed.

10:20 AM Invited

Powder Production Techniques and PM Processing Routes for Gamma Titanium Aluminides: *Rainer Gerling*[1]; Frank Peter Schimansky[1]; Helmut Clemens[1]; [1]GKSS Forschungszentrum, Inst. for Matls. Rsrch., Max Planck Strasse 1, Geesthacht D 21502 Germany

As a consequence of the reactivity of the melt, only distinct techniques can be used for the production of high quality TiAl alloy powders. Such techniques are presented and properties of the resulting alloy powders with respect to process peculiarities are discussed. The use of TiAl alloy powders within the following processing routes is described. (i) Forging of HIP-compacted alloy powder at 850∞C and characterization of the submicron microstructure with respect to superplastic behaviour. (ii) Sheet rolling of HIP-compacted high Niobium containing TiAl alloy powder and tensile testing of these high strength sheets between RT and 1000∞C, (iii) Metal injection moulding of alloy powder <45 micrometer and discussion of the present achievable strength and impurity levels. With respect to the feasibility to produce large TiAl preforms of high chemical and microstructural homogeneity, the results of laboratory scale spray forming experiments are presented and the process related characteristics are discussed.

10:50 AM Invited

The Development of Sheet Gamma TiAl Technology Under the Enabling Propulsion Materials/High Speed Civil Transport (EPM/HSCT) Program: *Gopal Das*[1]; P. A. Bartolotta[2]; H. Kestler[3]; H. Clemens[4]; [1]Pratt & Whitney, 400 Main St., E. Hartford, CT 06108 USA; [2]NASA GRC, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [3]Plansee Aktiengesellschaft, Reutte/Tirol A-6600 Austria; [4]GKSS Research Center, Max-Planck Strasse 1, Geesthacht D-21502 Germany

The sheet gamma TiAl technology is one of several new and challenging technologies successfully developed under the NASA sponsored EPM/HSCT program. The task was to design and fabricate the divergent flap of the nozzle that would meet the overall weight goal of the HSCT engine. The designers selected gamma TiAl for its lightweight, high modulus, and elevated temperature properties to design the divergent flap of an extremely large exhaust nozzle required to reduce exhaust and noise pollution. This presentation will review diverse technologies that were developed to fabricate the nozzle subelement using sheet gamma TiAl. These include: production of large sheets, hot-die forming of sheets into corrugations, joining of sheets by brazing and diffusion bonding, EB welding, as well as by rivets, evaluation of microstructure and mechanical properties of sheets and joints, fabrication of a nozzle subelement involving brazing of large corrugations to face sheets, non-destructive evaluation of bond quality, and successful bend test of the subelement. The sheet gamma TiAl technology was successfully transferred to BFGoodrich for manufacturing of industrial scale components. Since the termination of the EPM/HSCT program, use of sheet gamma TiAl is being explored for aerospace applications and critical technologies are being developed to support their needs. These are: nozzles for gas turbine engines and helicopters, and thermal protection systems for the reusable launch vehicles (RLV), to name a few. Additional technologies such as superplastic forming/diffusion bonding, laser joining and drilling, waterjet machining, and fabrication of honeycomb structures are being developed in order to support these activities.

11:20 AM

An Innovative Method for Manufacturing Gamma-TiAl Foil: *Stephen John Hales*[1]; Mohammad Saqib[2]; Joel Alexis Alexa[2]; [1]NASA Langley Research Center, Struct. & Matls., MS 188A, 2 W. Reid St., Hampton, VA 23681 USA; [2]Lockheed Martin Engineering & Science Company, c/o NASA-LaRC, MS 188A, 2 W. Reid St., Hampton, VA 23681 USA

The manufacture and entrance into service of thin gage (~ 0.010 in.) gamma-TiAl product has been hampered by the inherent low room temperature ductility of the material. At NASA-LaRC a new approach is being explored for the efficient manufacture of gamma-TiAl foil with improved ductility. The objective is to produce a very clean material (low interstitial content) with a highly refined, homogeneous microstructure placed in a fully lamellar condition. The processing route involves the use of RF plasma spray deposition of pre-alloyed powders, followed by consolidation via vacuum hot pressing and heat treatment. The approach takes advantage of a deposition process which includes no electrodes, no binders and high cooling rates. Results and discussion of the work performed to-date will be presented.

11:40 AM Invited

Low-Temperature Sheet Rolling of Gamma-TiAl+Alpha2-Ti3Al Alloys: Approach and Implementation: *Renat Imayev*[1]; Valery Imayev[1]; Andrey Kuznetsov[1]; Marat Shagiev[1]; Gennady Salishchev[1]; [1]Institute for Metals Superplasticity Problems, Khalturin str. 39, Ufa 450001 Russia

The present work proposes a new approach for producing sheet material from gamma-TiAl+alpha2-Ti3Al alloys. This approach includes correct choice of alloy composition, effective thermomechanical processing for producing the most workable sheet prematerial and subsequent sheet rolling below the eutectoid temperature under near-superplastic conditions. The proposed low-temperature sheet rolling process was successfully demonstrated for wrought ingot-metallurgy Ti-45.2Al-3.5(Nb,Cr,B) and Ti-44.2Al-3.5(Nb,Cr,B) alloys. As was shown for the Ti-45.2Al-3.5(Nb,Cr,B) alloy, the proposed process provided sheet with excellent and isotropic superplastic properties in the temperature range of 1000-1100∞C. Using a strain rate 10-3 s-1 elongation of up to 330-550% were measured with flow stress at 50% true strain ranging between 60-140 MPa. It is thought that the developed processing route is more cost-effective when compared with current used processing routes, which include sheet rolling above the eutectoid temperature.

12:10 PM

Equal Channel Angular Extrusion (ECAE) Processing of Titanium Aluminides for Microstructural Refinement and Mechanical Property Improvements: *Shankar M.L. Sastry*[1]; [1]Washington University, Mechl. Eng., CB 1185, One Brookings Dr., St. Louis, MO 63130 USA

ECAE process consists of extruding a well-lubricated billet through two intersecting channels of identical cross section. Deformation is achieved at the plane formed by the crossing of the channels through simple shear. A large and uniform strain intensity per pass can be obtained through this process without a reduction in the cross-sectional area of the billet. If the process is carried out at high temperatures at certain extrusion rates, grain refinement by dynamic recrystallization and/or spheroidization of lamellar microstructure can be produced. ECAE experiments were carried out on several gamma-based titanium aluminides in a vacuum of 10í5 Torr at 1000 and 1100°C at extrusion rates of 0.01 to 0.05 in/min. Grain refinement down to 1 mm was observed in all the ECAE processed alloys. ECAE processing results in a 25-50% increase in yield strength and 50-90% increase in ductility, 20-40% increase in fracture toughness. At temperatures and stresses where creep is predominantly controlled by dislocation glide and climb, the creep rates are not adversely affected by the fine grained microstructure produced by ECAE processing.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C. T. Liu: Intermetallics V–Iron Aluminide

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Seetharama C. Deevi, Philip Morris USA, Research Center, Richmond, VA 23234 USA; Fritz Appel, GKSS Research Centre, Geesthacht D-21502 Germany; Robert W. Cahn, University of Cambridge, Materials Science and Metallurgy, Cambridge CB2 3QZ UK; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Guo Liang Chen, University of Science and Technology-Beijing, State Key Laboratory for Advanced Metals and Materials, Beijing 100083 China; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208-3108 USA; Shuji Hanada, Tohoku University, Sendai 980-8577 Japan; Linda Horton, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6132 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Dong-Liang Lin, Shangai Jiao-Tong University, Shangai 200030 China; T. G. Nieh, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; Masaharu Yamaguchi, Kyoto University, Department of Materials Science & Engineering, Kyoto 606-8501 Japan

Wednesday AM Room: 8
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Dongliang Lin, Shanghai Jiao Tong University, Sch. of Matls. Sci. & Eng. & Open Lab. of Edu. Ministry of China for High Temp. Matls. & Tests, Shanghai 200030 China; Fritz Appel, GKSS Research Centre, Inst. for Matls. Rsrch., Geesthacht D-21502 Germany

8:30 AM Invited

Development, Processing and Applications of High Strength, High Ductility Iron Aluminides: *S. C. Deevi*[1]; R. S. Sundar[1]; S. Gedervanishvili[1]; [1]Philip Morris USA, Rsrch. Ctr., Richmond, VA 23234 USA

Iron Aluminides based on Fe-40Al have been investigated as possible replacements for a wide variety of structural and functional applications due to their low density, high strength, and excellent oxidation and corrosion resistance. Several approaches were considered to enhance the strength, creep resistance and rupture life of Fe-40Al alloys while maintaining or enhancing the ductility. In this paper, we present an overview of our approaches on enhancing the creep strength by mechanical alloying, solid solution and precipitation strengthening mechanisms. In addition, we will discuss the cold rolling and powder processing characteristics of iron aluminides, and the advantages and applications offered by powder processing approaches towards commercialization of iron aluminides.

8:50 AM Invited

Strain-Induced Ferromagnetism in Plastically-Strained FeAl Single Crystals: *I. Baker*[1]; D. Wu[1]; P. R. Munroe[2]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA; [2]University of New South Wales, Electron Microscope Unit, Sydney, NSW 2052 Australia

An overview of plastic strain-induced ferromagnetism in FeAl will be presented. Recent research has indicated that the effect arises mostly from the generation of anti-phase boundary (APB) tubes, where Fe atoms can have ≥3 like nearest neighbors (NNs). The resulting saturation magnetization, Ms, depends on both the Fe:Al ratio and the degree of deformation. A quantitative mode will be presented that describes the effects of both of these parameters in terms of the local environment theory applied to the atoms in APB tubes. The behavior of ternary alloys will be explained by considering the site preferences of ternary atoms on the FeAl sublattices, which change the probabilities of a Fe atom having like NNs in the APB tubes. Transmission electron microscopy observations of APB tubes in lightly-strained FeAl single crystals will be presented, and the annealing out of APB tubes and the associated activation energy will be discussed. Research sponsored by National Science Foundation through grant DMR 9973977.

9:10 AM

Effects of Lattice Vacancies on the Phonon DOS of FeAl: *Tabitha Liana Swan-Wood*[1]; [1]California Institute of Technology, Matls. Sci., MS 138-78, Pasadena, CA 91125 USA

FeAl alloys can have vacancy concentrations as high as 3-4%. The equilibrium thermal vacancy concentration is given by c(T)=a*exp(-E/kT) E being the energy of formation and a=exp[S(vib)/k] with S(vib) the vibrational entropy of formation. Prior experiments show S(vib) is between 5 and 6 k_B/vacancy. We expected vibrational entropy to be a significant component of the thermodynamics of vacancy formation in FeAl. A sample with 2% vacancies would have 16% of atoms with first-nearest neighbor vacancies, so we expected measurable changes in its phonon spectrum. Inelastic spectra were measured on LRMECS at IPNS from three Fe-50 at.% Al samples with vacancy concentrations approximately 0 to 2%. The phonon modes shifted slightly with vacancy concentration. Nevertheless, the data do not show any significant change in vibrational entropy between concentrations.

9:25 AM Invited

Interpretation of the Electrical Properties of Fe-Al Alloys from Electronic Structure Calculations: *P. Jena*[1]; S. C. Deevi[2]; G. P. Das[1]; B. K. Rao[1]; A. C. Lilly[2]; [1]Virginia Commonwealth University, Dept. of Physics, Richmond, VA 23284 USA; [2]Philip Morris USA, RD&E Ctr., Richmond, VA 23234 USA

Lilly et al (Mat. Sci. Engg. A258, p.42, 1998) have shown that the electrical resistivity of Fe-Al alloys increases from 0 at.% to about 33 at.% after which there is a steep decrease with further increase of Al concentration. A qualitative explanation was provided based on the phenomenological s-s and s-d scattering theory of Mott-Jones for AB alloys. In this paper, we carried out a systematic theoretical investigation of the electronic, magnetic, and cohesive properties of Fe1-XAlX alloys by successively replacing Fe atoms by Al atoms and studying the electronic structure within the density functional theory and generalized gradient approximation for exchange and correlation. We have shown that the stoichiometric FeAl exists in two nearly degenerate statesña non-magnetic and a ferromagnetic state, while Fe3Al is clearly ferromagnetic. The bonding between Fe and Al atoms is dominated by nearest neighbor interaction including hybridization of Fe 3d with Al 3p states. With increase of Al concentration, the total density of states at the Fermi energy increases, reaches a peak around X0.33, and then decreases. This behavior is very similar to the concentration dependence of electrical resistivity in Fe1-XAlX suggesting that the resistivity anomaly has an electronic origin. In addition, these results are consistent with model calculations where atomic clusters are used as a model of the bulk. In this paper, we review the electrical resistivities of Fe1-XAlX alloys and present our electronic structure calculations carried out during the last four years.

9:45 AM

Periodicities of Chemical Environments in Fe3Al Measured by Mossbauer Diffraction: *Jiao Y.Y. Lin*[1]; Ryan Douglas Monson[1]; Brent Fultz[1]; [1]California Institute of Technology, Matls. Sci., 1200 E California Blvd., MC 138-78, Pasadena, CA 91125 USA

Mossbauer diffractometry combines the capability of Mossbauer spectrometry to distinguish local chemical environments with the capability of diffractometry to measure long-range order (LRO) in materials. The energy spectra of intensities of fundamental and superlattice Bragg diffractions in partially-ordered Fe3Al were measured and compared to theoretical calculations. The energy spectra gave the expected results that Fe atoms with 4 Al first-nearest neighbor (1nn) are arranged as a simple cubic lattice, while Fe atoms with 0 Al 1nn atoms have an fcc periodicity. More interestingly, Fe atoms with 3 Al 1nn atoms have a simple cubic LRO, similar to that of Fe atoms with 4 Al 1nn atoms. This unexpected LRO was related to distributions of antisite defects.

10:00 AM Invited

Composite Based on Iron Aluminide Intermetallic Alloy and CrMo Steel: *Shuji Hanada*[1]; *Naoya Masahashi*[1]; [1]Tohoku University, Inst. for Matls. Rsrch., Katahira 2-1-1, Aoba-ku, Sendai, Miyagi 980-8577 Japan

Composite of iron aluminide intermetallic alloy and CrMo steel was prepared by solid state bonding to improve corrosion resistance of the steel. The microstructure observation reveals that a sound joint is achieved without producing any defects and columnar grains are evolved towards the steel matrix from the joint interface bonded at high temperatures above A3. Composition changes continuously near the joint interface, and the interdiffusion coefficient of Al depends on the content of Cr. The formation mechanism of the columnar microstructure is explained by a nucleation at the joint interface caused by Al diffusion to stabilize α, followed by grain growth to steel side. The composite demonstrates high bonding strength due to a sufficient interdiffusion between the constituents. A sound composite is also fabricated by clad-rolling. These results suggest that the composite has a potential for corrosion-resistant applications.

10:20 AM Break

10:35 AM Invited

Development of High Strength High Ductility and High Temperature Iron Aluminide: *David G. Morris*[1]; Maria A. MuÒoz-Morris[1]; J. Chao[1]; Carmen Garcia Oca[1]; [1]CENIM, CSIC, Dept. of Physl. Metall., Avenida Gregorio del Amo 8, Madrid E-28040 Spain

Significant research activities over the last ten years have led to the development of a high strength and high ductility Fe-40Al alloy produced by mechanical alloying of the intermetallic with dispersed oxides. Strength and ductility and high temperature behaviour of this material are analysed to deduce the important microstructural features that contribute to each aspect of behaviour. High strength is seen to be a consequence of solution hardening, particle strengthening, and grain size hardening, in that order of importance. High ductility is seen to depend mostly on a fine grain size which, together with dispersed particles, ensures slip homogenisation and delays failure crack nucleation to high strains. Strength at high temperature falls because of rapid diffusion in the open bcc base lattice. Ways for improving high temperature strength are based on including large volume fractions of rather coarse and stable second phase particles.

10:55 AM Invited

Thermal-Cycling Deformation of Superplastic Coarse-Grained Fe-24.5%Al-1.4%Ti alloy: *Jinn P. Chu*[1]; C. L. Chiang[1]; H. Y. Yasuda[2]; Y. Umakoshi[2]; K. Inoue[3]; T. Mahalingam[4]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2, Pei-Ning Rd., Keelung 20224 Taiwan; [2]Osaka University, Dept. of Matls. Sci. & Eng., Osaka 565-0871 Japan; [3]University of Washington, Dept. of Matls. Sci. & Eng., Seattle, WA 98195 USA; [4]Alagappa University, Dept. of Physics, Karaikudi 630 003 India

Many studies have demonstrated that coarse-grained Fe-Al based alloys show all the deformation characteristics that conventional fine-grained superplastic materials possess. Using an electron backscattered diffraction technique, we have identified several important crystallographic features of a superplastic coarse-grained Fe-27at.%Al alloy. As a result of dynamic recovery and recrystallization during deformation, major microstructural evolutions are summarized as a sequence of (1) subgrain-boundary formation at 600°C, (2) grain-boundary migration at 700°C, (3) formation of new grains resulting in grain refinement and hence greater superplastic elongation at 800°C, and (4) growth of recrystallized grains at 850°C and above. In the present study, effects of thermal-cycling deformation on superplastic properties of coarse-grained Fe-24.5%Al-1.4%Ti alloy have been examined in air under an initial strain rate of $1x10^{-4}$ s^{-1}. Thermal cycling deformation between 800-850°C results in refined grains as a dominant structure and evidently improves the superplastic elongation to 391%.

11:15 AM Invited

Microscopic and Macroscopic Deformation Observations in FeAl and Fe3Al Alloys: *Bimal Kad*[1]; Joe Horton[2]; Chain T. Liu[2]; [1]University of California-San Diego, Structl. Eng., 409 University Ctr., MC-0085, La Jolla, CA 92093-0085 USA; [2]Oak Ridge National Laboratory, M&C Div., MS-6115, Bldg. 4500S, PO Box 2008, Oak Ridge, TN 37831-6115 USA

Macroscopic texture measurements of FeAl, Fe_3Al-based alloys deformed at 925<T<1325K in the B2 structure regime by rolling, forging and extrusion, at deformation rates of 10^{-4}-10^{-1} sec^{-1}, agree well with the numerically predicted textures for the <111>{110}+<110>{112} slip system activation for each of the deformation histories. These results appear to be in disagreement with microscopic TEM observations, that overwhelmingly support the activation of <100>{011} and <100>{001} slip systems at high temperature. We revisit the issue of TEM observations within the framework of a deformation experiment in the Gleeble apparatus, where the high temperature deformation sub-structure is quenched-in by imposing cooling rates of about 10^3 $K.sec^{-1}$, thereby minimizing post deformation dislocation reorganization. TEM studies reveal that such quenched-in substructure is indeed dominated by <111> and not <100> dislocations. Furthermore, the <111> to <100> thermal reorganization is quite rapid, as observed via short annealing treatments, and this propensity increases with aluminum content.

11:30 AM Invited

Microstructures and Mechanical Properties of Fe-Al-C and Fe-Al-M-C (M = Ti, V, Nb, Ta) Alloys: *AndrÈ Schneider*[1]; Ladislav Falat[1]; Gerhard Sauthoff[1]; Georg Frommeyer[1]; [1]Max-Planck-Institut f¸r Eisenforschung GmbH, Matls. Tech., Max-Planck-Str. 1, D¸sseldorf 40237 Germany

This paper presents results on constitution, microstructure and mechanical properties of a variety of Fe_3Al-based alloys. Alloys based on the systems Fe-Al-C and Fe-Al-M-C (M = Ti, V, Nb, Ta) with strengthening carbides and Laves phase were investigated. The Fe-Al-C materials contain 23 to 29 at% Al and 1 to 3 at% C. The Fe-Al-M-C alloys contain 26 at% Al, 2 at% Ti, V, Nb, or Ta, and 1 at% C, respectively. The alloys were processed by vacuum induction melting and cast into Cu-moulds. The alloys were investigated in the as-cast state and after various heat treatments. For evaluating the mechanical properties as a function of the temperature, compression tests were performed. Microstructural analysis was performed by means of light optical microscopy (LOM), scanning electron microscopy (SEM), electron probe micro-analysis (EPMA), and X-ray diffraction analysis (XRD). The experimentally determined phase equilibria are compared with thermodynamic calculations using Thermo-Calc.

11:50 AM
Improvement of the Creep Strength of FeAl-Based Alloys: *Wei-Jun Zhang*[1]; R. S. Sundar[1]; S. C. Deevi[1]; [1]Philip Morris USA, PD&T, PO Box 26603, Richmond, VA 23261 USA

Great attention has been paid to the environmental embrittleness, vacancy hardening and corrosion behavior in FeAl-based alloys during the last decade. However, the creep behavior of FeAl has not been well understood as yet. The relatively poor creep strength of FeAl limits the wide spread applications of FeAl alloys. In this paper, we discuss the major factors leading to the poor creep resistance of FeAl in terms of high diffusional coefficient, low activation energy, high dislocation mobility, high vacancy concentration and microstructural instability. We will demonstrate that creep resistance can be improved through precipitation hardening and solid solution strengthening. However, more studies are needed to enhance the microstructural stability of FeAl alloys during long-term service.

12:05 PM
Reactive Thermomechanical Processing of Ni3Al and FeAl: *K. Morsi*[1]; S. O. Moussa[1]; J. Wall[1]; J. Rodriguez[1]; [1]University of Missouri, Mechl. & Aeros. Eng., E3411 Eng. Bldg. E., Columbia, MO 65211 USA

Nickel aluminides (Ni3Al) and Iron aluminides (FeAl) are intermetallics of significant technological importance. Major drawbacks, which have so far restricted the use of these materials, are the high-energy usage in synthesis and processing and difficulty in fabrication into the final component shape. The proposed work explores a new near net shape, low-energy approach which can be used to process a range of important aluminide intermetallics and their composites (e.g. titanium aluminides, nickel aluminides, iron aluminides and niobium aluminides), with wide ranging applications. The work discusses the application of thermomechanical processing (extrusion & forging) during the high temperatures achieved in reaction synthesis to form and simultaneously shape Ni3Al and FeAl aluminide intermetallics. The effect of varying processing parameters on the developed microstructure and properties is presented. Results confirm the feasibility of this new approach to process these materials at operating temperatures ~400 C lower than conventionally used in the extrusion and forging of these materials with potentially considerable energy and cost savings.

International Symposium on Structures and Properties of Nanocrystalline Materials: Microstructure and Properties

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Wednesday AM	Room: 14B
March 5, 2003	Location: San Diego Convention Center

Session Chairs: Ian Baker, Dartmouth College, Thayer Sch. of Eng., Hanover, NH 03755 USA; Chandra S. Pande, Naval Research Laboratory, Div. of Matls. Sci. & Tech., Washington, DC 20375 USA

8:30 AM
An Electron Microscopy Study of the Structure and Deformation Behavior of Electrodeposited Nanocrystalline Nickel Alloys: *Sharvan Kumar*[1]; Subra Suresh[2]; Matthew F. Chisholm[3]; Joe A. Horton[4]; Ping Wang[1]; [1]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA; [2]Massachusetts Institute of Technology, DMSE, Cambridge, MA 02139 USA; [3]Oak Ridge National Laboratory, Solid State Div., Oak Ridge, TN 37831 USA; [4]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

The deformation of electrodeposited nanocrystalline nickel with a mean grain size in the range of 30-40 nm and a columnar grain morphology with column lengths being anywhere from 3 times to 8 times the in-plane grain diameter was studied by ex-situ and in-situ deformation experiments. Results of the in-situ experiments confirmed the presence of extensive dislocation activity at the crack tip in these specimens. Voids form in the region ahead of the crack tip at grain boundaries and triple junctions and partially relieve the constraints on the grain, permitting dislocation processes to occur more readily. Examination of the fracture surfaces of such specimens after the test in an SEM confirms failure by dimpled rupture. The scale of these dimples is significantly larger than the average grain size and fracture in the bulk does not appear to propagate along grain boundaries in this material.

9:00 AM
Analysis of Misfit Dislocations in Epitaxial Ni/Cu Bilayers: *David Mitlin*[1]; Amit Misra[1]; Richard G. Hoagland[1]; Mike Nastasi[1]; Harriet Kung[1]; John P. Hirth[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-8, Los Alamos, NM 87545 USA

Understanding bilayer behavior is a critical step in the successful development of ultra-high hardness nano-layer composites. We have analyzed misfit dislocations at the interface of Ni/Cu bilayers using plan-view and cross-sectional transmission electron microscopy (TEM). The bilayers consisted of varying thickness of Ni (5-1000 angstroms) deposited on 1000 angstroms of Cu. Conventional g-b analysis was used to determine the types of dislocations present at the interface, while high-resolution TEM performed on cross-sectional samples was used to analyze the interface in more detail. It is demonstrated that there is minimum Ni thickness below which interface dislocations do not nucleate. As the Ni layer thickness is increased, the dislocations spacing decreases. Agreements and discrepancies of the experimental results with the existing interface dislocation theory are discussed.

9:20 AM
Microstructures and Mechanical Properties of Nanoscale Copper-304 Stainless Steel Multilayers Synthesized by Magnetron Sputtering: *Xinghang Zhang*[1]; Amit Misra[1]; Harriet Kung[1]; John D. Embury[1]; Michael A. Nastasi[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-8, MS G755, Los Alamos, NM 87545 USA

Copper_304 stainless steel (SS) multilayers synthesized by magnetron sputtering were studied systematically in an attempt to understand the role of metastable crystal structures on the strength of nanolayered composite. The composites were composed of Cu and SS layers of equal thickness, with the layer thickness varying from 1 nm to 500 nm. Transmission electron microscopy analysis shows that the SS layers with thickness of greater than 10 nm have a mixture of metastable bcc and equilibrium fcc phases. The co-existence of the two phases offers a special opportunity to study stabilization mechanisms of unusual phases at the nanometer scale. Nanoindentation measurements show that the hardness of these nanolayered materials increases with the decreasing layer thickness, reaching a maximum value of about 5.5 GPa at the layer thickness of 5 nm. A decrease in hardness was noted with decreasing layer thickness below 5 nm. The effects of layer thickness, interfaces and the formation of metastable bcc phase in the SS layer on strength of Cu-SS multilayer are discussed.

9:40 AM
Structure-Property Studies of Bias Sputter Deposited Cu1-xNbx Alloys: *Guoyi Tang*[1]; [1]North Carolina State University, Matls. Sci. & Eng., CB 7907, Raleigh, NC 27695-7907 USA

Nanocrystalline, non-equilibrium Cu1-xNbx (x = 2 to 75) alloys have been deposited onto glass and Cu substrates using dual magnetron sputter deposition. In addition to the usual sputter deposition parameters of cathode power and Ar working pressure, these films were deposited under a range of temperature and substrate bias (low energy Ar+ ion bombardment) conditions. X-ray diffraction, scanning electron microscopy, transmission electron microscopy, and atomic force microscopy (AFM) were used to characterize film microstructure, texture, and surface morphology. The film microstructures consist of nanoscale, discrete Nb particles in a Cu matrix. Films with Nb concentration >5 atomic percent have strong [111] type textures. Increasing Nb concentrations altered the surface morphologies from irregular (<5% Nb) to spherical (>10% Nb).

10:00 AM Break

10:20 AM
Formation and Characterization of CuOx Nanowire Arrays: Chen Jin-Ming[1]; [1]Industrial Technology Research Institute, Matl. Rsrch. Labs., 240r, Bldg. 77, Chutung 310 Taiwan

We reported a novel method for preparing one-dimensional configuration and well-ordered nanowires of copper oxide by using electrodeposition followed by oxidation treatment. The morphology of CuOx nanowires were examined by scanning electron microscopy and transmission electron microscopy and showed that the diameters of nanowires were in range of 10-50 nm. The result of X-ray photoelectron spectroscopy indicated the presence of two stages of copper oxides, CuO and Cu2O in the nanowire array. The analysis results suggest that the CuOx nanowires are promising candidates for field-emission devices and Li-ion anode materials.

10:40 AM
The Interaction Between Nanocrystalline Grains in Cryomilled Nanocrystalline Al-Mg Alloys: *Zonghoon Lee*[1]; Bing Q. Han[2]; Farghalli A. Mohamed[2]; Enrique J. Lavernia[2]; Steven R. Nutt[1]; [1]University of Southern California, Matls. Sci., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA; [2]University of California-Irvine, Dept. of Cheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

The deformation of bulk nanocrystalline Al-Mg alloys was investigated using transmission electron microscopy and high-resolution electron microscopy. Grain refinement was achieved by cryomilling of elemental powders, and powders were consolidated by hot isostatic pressing (HIP) and extrusion to produce bulk nanocrystalline Al-Mg alloys. The microstructure of cryomilled Al-Mg alloys consisted of equiaxed and elongated grains, which were elongated along the extrusion direction. The nanocrystalline Al-Mg alloys exhibited unusual deformation characteristics involving complex interactions between nanocrystalline grains in contrast to a conventional Al-Mg alloy. Elongated grains were comprised of dislocation cells, sub-grains, and slip bands. Investigation of bulk tensile and compression fracture specimens revealed unusual failure mechanisms and interactions between nanocrystalline grains. Tensile behavior was characterized by high yield strength, high ductility, and low strain hardening. After the yield point, the alloy exhibited nearly perfectly plastic behavior and low strain hardening.

11:00 AM
Structure, Mechanical and Transport Properties of Nanocrystalline Nb and Ti Thin Films: *Rajarshi Banerjee*[1]; Evan Sperling[1]; Gregory B. Thompson[1]; Parinda Vasa[2]; Pushan Ayyub[2]; Hamish Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Tata Institute of Fundamental Research, Condensed Matter Physics & Matls. Sci., Homi Bhabha Rd., Mumbai, Maharashtra 400005 India

Nanocrystalline thin films of Nb and Ti with varying grain sizes have been deposited using high pressure magnetron sputtering. The structure of these films as a function of grain size is being characterized using x-ray diffraction and TEM. Initial results suggest that in case of Nb, there is a significant lattice expansion with reduction in grain size while in case of Ti, the c/a ratio changes with the grain size. Nanoindentation experiments are being conducted to evaluate the influence of these structural changes on the modulus and hardness of these nanocrystalline films. In addition to the mechanical properties, the electrical transport properties of these films are also being investigated. Thus, a systematic investigation of the influence of grain size and structure on the superconducting properties of the Nb and Ti films is being carried out through the measurement of the ac-Meissner effect using a SQUID magnetometer and the results will be presented in this paper.

11:20 AM
DC Magnetron Sputtered Fe Films for Growth of C Nanotubes: *Guoyi Tang*[1]; Yunyu Wang[2]; Robert J. Nemanich[2]; J. M. Rigsbee[1]; [1]North Carolina State University, Matls. Sci. & Eng., CB 7907, Raleigh, NC 27695-7907 USA; [2]North Carolina State University, Dept. of Physics, Raleigh, NC 27695 USA

Thin films of Fe have been DC magnetron sputter deposited onto Si (100) wafers using cathode powers from 30 to 150Watts and times up to 60 minutes. These processes led to four iron thin films with thicknesses between 40 and 90 nm. X-ray diffraction, transmission electron microscopy, scanning electron microscopy and atomic force microscopy were used to characterize film structure and surface morphology. It is shown that the surface morphology and roughness of the iron films varied with sputter cathode power density. Above 70 Watts cathode power, the film surfaces consisted of nearly spherical grains. The ability of these films to nucleate and grow carbon nanotubes were found to be strongly influenced by surface morphology and nanotube growth was optimum at 70 Watts. The results for film and carbon nanotube growth will be presented.

Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging: Mechanical Properties and Fatigue Behavior

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Srini Chada, Motorola, Department APTC, Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Jud Ready, MicroCoating Technologies, Atlanta, GA 30341 USA; Jin Yu, KAIST, Center for Electronic Packaging Materials 305-701 Korea

Wednesday AM Room: 15B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Kwang-Lung Lin, National Cheng Kung University, Dept. of Matls. Sci. & Eng., Tainan 70101 Taiwan; K. N. Subramanian, Michigan State University, Cheml. Eng. & Matl. Sci., E. Lansing, MI 48824 USA

8:30 AM Invited
Compression Stress Strain Behavior of Sn-Ag-Cu (Cu = 0.2, 0.6, and 0.7): *Paul T. Vianco*[1]; Jerome A. Rejent[1]; Joseph Martin[1]; [1]Sandia National Laboratories, MS0889, PO Box 5800, Albuquerque, NM 87185-0889 USA

Several Sn-Ag-Cu Pb-free solders have been identified to replace Sn-Pb eutectic solder in reflow process applications. The compositions differ by a few tenths of a percent of Cu. The sensitivity of the mechanical properties was investigated for Sn-Ag-Cu solders having nominal Cu contents of 0.2, 0.6 and 0.7 wt.% Cu. Compression stress strain tests were performed to measure the yield stress of the solders as a function of test temperature and strain rate. The test temperatures were 25C, 25C, 75C, 125C, and 160C. Two strain rates were used: 0.00043 1/s and 0.000082 1/s. Samples were tested in the as-cast condition and following an initial annealing treatment at 125C for 24 hours. The microstructure of the solders was surveilled after testing. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Dept. of Energy under contract DE-AC04-94AL85000.

8:55 AM
A Potential Drop-in-Replacement for Eutectic Sn-Pb Solderñ The Sn-Zn-Ag-Al-Ga Solder: *Kwang-Lung Lin*[1]; Kang-I Chen[1]; Po-Cheng Shi[1]; [1]National Cheng Kung University, Matls. Sci. & Eng., 1 Ta-Hsuey Rd., Tainan 701 Taiwan

Sn-Zn alloy exhibits the closest eutectic temperature to the eutectic Sn-Pb solder, although it suffers easy oxidation characteristic. A novel alloy basing on the Sn-Zn eutectic alloy was developed and found promising as a drop-in-replacement for the eutectic Sn-Pb solder. This alloy, consisting of Sn-Zn-Ag-Al-Ga combination, was developed showing promising properties. DSC (Differential Scanning Calorimetry) investigation reveals that the investigated solder exhibits eutectic temperature of around 199°C. The TGA (thermal gravimetric analysis) study shows it attains much better oxidation resistance than the eutectic Sn-Pb solder at 250°C. Stress-strain curve indicates that this solder also possesses a greater UTS (ultimate tensile strength) and a better ductility than the eutectic Sn-Pb solder. A wetting time of less than one second was found for this solder with a suitable flux. Ga was seen to enhance wetting behavior. Cost of this solder was also estimated and showing comparable with that of other solders.

9:15 AM
Effect of Sb Addition on Microstructure and Shear Strength of Sn-Ag Solder Joints: *Hwa-Teng Lee*[1]; *Chuan-Lien Yang*[1]; Ming-Hung Chen[1]; Cheng-Shyan Li[1]; [1]National Cheng Kung University, Mechl. Eng. Dept., No. 1, Dashiue Rd., Tainan 701 Taiwan

The effect of Sb addition on microstructure, intermetallic compound (IMC) and mechanical properties of Sn-Ag solder joints is investigated. The compositions of selected solders are Sn2.58Ag, Sn2.82Ag1.75Sb, Sn2.87Ag4.75Sb and Sn2.7Ag8.78Sb. Experimental results show that most of the added Sb are solved in beta-Sn matrix, and the rest react with the Ag3Sn to form Ag3(Sn,Sb) phase, which contributes to suppress the coarsening of Ag3Sn phase. SbSn phase can be observed in beta-Sn matrix as the Sb addition exceeds 4.75% and remains stable during the thermal storage test. The solder microhardness increases with increasing Sb. And the growth rate of interfacial IMC layer decreases as Sb addition increases. EPMA analysis indicates there are some Sb diffusing into the interfacial IMC layer. Shear strength of solder joints are raised by adding Sb. The shear strength by as-soldered condition are 27.8MPa(0%Sb), 29MPa(1.75%Sb), 30.4MPa(4.75% Sb) and 43.4MPa(8.78%Sb) respectively.

9:35 AM
Assessment of Microstructural Evolution and Change in Mechanical Properties of Sn-Pb and Pb-Free Chip ResistorsñSolder Joints in Response to Aging and Accelerated Thermal Cycling: Adam R. Zbrzezny[1]; Polina Snugovsky[2]; *Doug D. Perovic*[1]; [1]University of Toronto, Matls. Sci. & Eng., Wallberg Bldg., 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]Celestica, Matls. Lab., 844 Don Mills Rd., Toronto, Ontario M3C 1V7 Canada

Aging and Accelerated Thermal Cycling (ATC) are currently being performed on 2512 chip resistors assembled with the Sn-Pb eutectic and Sn95.5Ag3.8Cu0.7Cu solders. The test vehicles were divided into 12 cells comprised of 5 boards per cell, 16 components per board. The boards were finished with immersion Ag, ENIG, and HASL, and components terminations were 100% Sn and SnPb. The microstructure is being investigated by cross-sectioning with subsequent optical microscopy and SEM/EDX. The mechanical properties are being assessed by the shear tests. The cross-sections and shear tests are being performed every 500 hrs of aging and 250 cycles of ATC. The results from this study, which will be presented, will allow for a direct comparison between the solder joints of various metallurgies. In addition, the microstructure evolution and its influence on the mechanical properties will be studied in detail.

9:55 AM
Isothermal Aging of Near-Eutectic Sn-Ag-Cu Solder Alloys and its Effect on Electrical Resistivity: *Bruce Allan Cook*[1]; Iver E. Anderson[1]; Joel H. Harringa[1]; Robert L. Terpstra[1]; Sung K. Kang[2]; [1]Iowa State University, Ames Lab., Metal & Ceram. Scis. Prog., 47 Wilhelm, Ames, IA 50011-3020 USA; [2]IBM T. J. Watson Research Center, Yorktown Heights, NY 10598 USA

Solder joints were prepared from seven near-eutectic Sn-based alloys and characterized for electrical resistivity after 100 and 1000 hours of isothermal aging at 423K. The solder joint samples were prepared by hand soldering to copper substrates and the post-heat treatment resistivity was measured at room temperature in a specially-designed 4-point fixture. Compositions tested included Sn-3.5Ag, Sn-3.7Ag-0.9Cu, Sn-3.0Ag-0.5Cu, Sn-3.6Ag-1.0Cu, and Sn-3.9Ag-0.6Cu; moreover, the effect of minor addition of a fourth element, designed to improve high temperature shear strength, was also evaluated in the compositions Sn-3.7Ag-0.6Cu-0.3Co and Sn-3.7Ag-0.7Cu-0.2Fe. The observed changes in electrical transport are discussed in terms of microstructural coarsening, diffusional transport from the substrate, and nucleation of precipitate phases. Results are compared with previous studies conducted on cross-shaped specimens formed by joining two ìL-shapedî copper coupons.

10:15 AM
Influence of Adding High Sb Content into Sn-Ag Solder Joints on Microstructure and Shear Strength: *Hwa-Teng Lee*[1]; Chuan-Lien Yang[1]; [1]National Cheng Kung University, Mechl. Eng. Dept., Tainan 70101 Taiwan

The study investigates the effect of adding Sb into Sn-Ag solder joints on microstructure, intermetallic compound (IMC) and mechanical properties. The compositions of selected solders were Sn2.58Ag, Sn2.82Ag1.75Sb, Sn2.87Ag4.75Sb and Sn2.7Ag8.78Sb. FR-4 PCB is used as the substrate material and the single-lap specimen form is selected for shear test. And thermal storage test is also performed after soldering to estimate the varity of microstructure and shear strength. Experimental results show that most of added Sb are solved in ,-Sn matrix, and the rest react with the Ag3Sn to form Â-Ag3(Sn,Sb) phase, which contributes to suppress the coarseness of Ag3Sn phase. SbSn phase can be observed in ,-Sn matrix as the Sb addition exceeds 4.75%, and the coarseness of SbSn phase is not obvious during thermal storage. The solder microhardness increases with increasing Sb addition. And the growth rate of interfacial IMC layer decreases as Sb addition increases. EPMA analysis indicates there are some Sb atoms diffusing in to the interfacial IMC layer. Shear strength of solder joints are improved as adding Sb. The shear strength of as-soldered condition are 27.8 (0%Sb), 29 (1.75%Sb), 30.4 MPa(4.75% Sb) and 43.4(8.78%Sb) MPa respectively.

10:35 AM Break

10:50 AM Invited
Temperature Effects on Low Cycle Fatigue Behavior of Sn/3.5Ag/0.75Cu and 63Sn/37Pb Solder Joints: Tae-Sang Park[1]; *Soon-Bok Lee*[1]; [1]CARE Electronic Packing Laboratory, Dept. of Mechl. Eng., KAIST, 373-Kusong-dong, Yusong-gu, Daejeon 305-701 Korea

The demand of lead-free solder and high-density interconnection technology in modern microelectronic packaging has been increased. Due to this high degree of integration and component density, the modern advanced SMC assembly has imposed more stringent reliability requirements on packaging design. Service failure of solder interconnections generally arises due to thermomechanical fatigue (TMF) and usually occurs within the solder itself since it is more soft parts of the joint. The combination of temperature fluctuations, either due to power switching or the external environment, and materials in the joint which possesses different coefficients of thermal expansion, produce substantial cyclic strains within the solder. Therefore in most applications, solder joints are under thermo-mechanical loading condition. To understand this thermo-mechanical material behavior of solder joints, isothermal cyclic fatigue tests at several temperature levels are essential. In the present work, low cycle isothermal mechanical fatigue tests of lead-free solder (Sn/3.5Ag/0.75Cu) and lead-containing solder (63Sn/37Pb) were carried out in several temperature levels. The mechanical fatigue tests were performed under conditions of shear loading at the temperature of 25°C, 70°C, and 100°C. Constant displacement tests are performed using a computer controlled micro-mechanical test apparatus. The low cycle fatigue behavior of these solder alloys was found to be strongly dependents on test temperature. Failure patterns of the fatigue tests are observed and discussed. As a fatigue model, the Morrow energy model and Coffin-Manson model were examined. The material parameters in these models are found to be a function of temperature rather than constants.

11:15 AM
Effects of Mechanical Deformation and Annealing on the Microstructure and Hardness of Pb-Free Solders: *Sung K. Kang*[1]; Paul Lauro[1]; Won Kyoung Choi[1]; Da-Yuan Shih[1]; [1]IBM T. J. Watson Research Center, PO Box 218, Yorktown Heights, NY 10598 USA

The microstructure-property relations of several Pb-free solders have been investigated in order to understand the microstructure changes during thermal and mechanical processes of Pb-free solder joints. Pb-free solder alloys investigated include pure Sn, Sn-0.7%Cu, Sn-3.5%Ag and Sn-3.8%Ag-0.7%Cu (in weight). To reproduce a typical microstructure observed in solder joints, the cooling rate, ingot size and reflow conditions of cast alloys are carefully controlled. The cast alloy pellets are subjected to compressive deformation up to 50% and annealing at 150∞C, 48 h. The microstructure of Pb-free solders is evaluated as a function of alloy composition, plastic deformation and annealing. The changes in mechanical property are measured by microhardness test. The work hardening in Sn-based alloys is found to increase as the amount of alloying elements increases. The change in microhardness upon deformation and annealing has been correlated with the microstructural changes, such as recrystallization or grain growth in Pb-free solder alloys.

11:35 AM
Lead (Pb)-Free Ceramic Ball Grid Array (CBGA): Thermo-Mechanical Fatigue Reliability: *Mukta Farooq*[1]; Charles Goldsmith[1]; Ray Jackson[1]; Gregory Martin[1]; [1]IBM Microelectronics, 2070 Rte. 52, MS 87P, Hopewell Junction, NY 12533 USA

Flip-chip carriers have become the preferred solution for high-performance ASIC and microprocessor devices. Typically these are packaged in organic or ceramic Ball Grid Array (BGA) packages which cover a wide range of package I/O capabilities required for high-performance devices, typically between 300 to more than 1600 I/O. Recently, there has been a move towards Pb-free solders, as replacement alloys for standard eutectic Sn/Pb and other Pb-based BGAís. The leading solder that has emerged is an Sn/Ag/Cu (SAC) alloy. One of the primary issues with changing solders is the reliability of the joints when subjected to Thermo-Mechanical Fatigue (TMF). With the need to shrink the I/O pitch to accommodate higher wiring density, it has become important to conduct reliability assessments in a 1.00mm pitch format. This paper describes such an evaluation conducted using SAC BGA assemblies. The results show that for a 1.00mm pitch, the Pb-free SAC CBGA solution provides superior reliability as compared to the standard Sn/Pb CBGA solutions. This finding is an added incentive for a new CBGA offering employing the new Pb-free SAC single alloy self-aligning system.

11:55 AM
Characteristics of Ni Bearing Sn-Ag Composite Solders: *Joo-Won Lee*[1]; Zin-Hyoung Lee[1]; [1]Korea Advanced Institute of Science and Technology, Matl. Sci. & Eng., 371-1 Guseong Dong, Taejeon 305-701 S. Korea

Ni bearing Lead Free Sn-Cu-Ag solders have been investigated by employing various experimental techniques such as conventional cross-sectional metallography, scanning electron microscopy (SEM), electron microprobe analysis, X-ray diffraction, differential scanning calorimetry (DSC) and others. Based on the thermodynamic calculation, compositions of eutectic and composite solders were determined. Ni bearing solder had lower melting temperature and better mechanical properties, compared with Sn-Ag, Sn-Ag-Cu eutectic solders. Structure and composition of reinforcing particle were varied with the change of Cu/Ni ratio. This variation affected the sedimentation of reinforcing particles. Small addition of Ni was found to retard IMC layer thickening and to enhance ball shear strength after reflow and aging. IMC growth model was also discussed.

12:15 PM
Isothermal Fatigue Properties of Lead-Free BGA Joints: Yoshiharu Kariya[1]; *Takuya Hosoi*[2]; Yasunori Tanaka[3]; Masahisa Ostuka[2]; [1]National Institute for Materials Science, Eco-Matls. Ctr., Namiki 1-1, Tsukuba, Ibaraki 3050044 Japan; [2]Shibaura Institute of Technology, Matls. Sci. & Eng. Dept., Shibaura 3-9-14, Minato-Ku, Tokyo 10888548 Japan; [3]NEC Corporation, Mobile Terminal Div., Ikebe-cho, Tsuzuki-Ku, Yokomaha, Kanagawa 224 Japan

The split board fatigue test has been performed at 298K and 358K to look for strain life relationship of the Sn-3.5Ag-0.75Cu and Sn-37Pb solder joints made between a 0.5mm pitch or a 0.8mm pitch CSP and a PCB. The fatigue life of Sn-3.5Ag-0.75Cu joint is not significantly different to that of Sn-37Pb joint at as soldered condition, although the Sn-37Pb joint exhibits poor fatigue when the joint is subjected to the aging at 393K for 20 days. The degradation of Sn-37Pb joint due to the aging is attributed to the formation of Au-Ni-Sn and Ni-Sn layers. As the fatigue life of the joints was correlated to the strain energy density regardless of the pitch, the energy based life prediction model well describes the fatigue life of CSP/PCB interconnects. A set of strain energy density versus fatigue life curves is provided which can be used to predict the joint life.

Magnesium Technology 2003: Magnesium Alloy Development-Mechanical Properties - I

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, LMD-Magnesium Committee, International Magnesium Association, MPMD-Solidification Committee
Program Organizers: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

Wednesday AM — Room: 2
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Mihriban Pekguleryuz, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Robert R. Powell, General Motor Corporation, NAO R&D Ctr., Warren, MI 48090-9055 USA

8:30 AM
Enhanced Ductility and Strength Through RE Addition to Magnesium Die Casting Alloys: *Per Bakke*[1]; Ketil Pettersen[1]; HÂkon Westengen[1]; [1]Norsk Hydro ASA, Magnesium Matls. Tech., PO Box 2560, Porsgrunn N-3907 Norway

Development of new die casting alloys is a multifacetted task, where success depends upon the ability to control a chain of properties, where the weakest link determines the outcome. It is commonly experienced that optimizing one property by alloying comes at the expense of one or more other properties. A typical example is yield strength vs. ductility. In developing alloys for high pressure die casting, the peculiar aspects of the process must be considered. High injection speeds, high metal pressures and the lack of efficient thermal barriers lead to extremely high cooling rates. This is making high pressure diecasting unique, since the resulting refined microstructure provides excellent mechanical properties. In the current paper, the influence of alloy composition on mechanical properties is investigated, with special emphasis on strength and ductility.

8:50 AM
Dead Sea Magnesium Alloys Newly Developed for High Temperature Applications: *Eli Aghion*[1]; Boris Bronfin[1]; Frank von Buch[1]; Soehnke Schumann[1]; Horst Friedrich[2]; [1]Dead Sea Magnesium, Rsrch. Div., PO Box 1195, Beer-Sheva 84111 Israel; [2]Volkswagen AG, Vehicle Rsrch., Wolfsburg Germany

Recently several new magnesium alloys for high temperature applications have been developed with intention to obtain an adequate combination of different properties such as castability, creep resistance, mechanical properties, corrosion performance and affordable cost. Unfortunately, it is very difficult to achieve an optimal combination of properties and in fact, most of the new alloys can only address part of the required properties and performances. This paper aims at evaluating the current status of the newly developed alloys for powertrain applications. The paper also explains the complexity of magnesium alloy development and illustrates the effect of alloying elements on properties and cost. In addition, the paper presents an attempt to set the position of each alloy in the space of combined properties and cost.

9:10 AM
Effects of Ca Additions on Microstructures, Age Hardening Response and Creep Behaviour of Mg-8Zn-4Al Casting Alloy: *Chamini Mendis*[1]; Laure Bourgeois[1]; Barry Muddle[1]; Jian-Feng Nie[1]; [1]Monash University, Sch. of Physics & Matls. Eng., Melbourne, Victoria 3800 Australia

Increased applications of magnesium alloys for elevated temperature service in the automotive industry require the development of low-cost, high-strength and creep-resistant alloys. It has been demonstrated in recent years that Mg-Zn-Al casting alloys exhibit creep resistance superior to that of binary Mg-Al and Mg-Zn alloys, and that quaternary additions of small concentrations of Ca to these Mg-Zn-Al alloys further improve the creep resistance of the alloys. However, the microstructure and creep behaviour of the Mg-Zn-Al(-Ca) alloys have not been characterized in detail, and the role of Ca in improving the creep resistance remains to be elucidated. In this work, the age-hardening response of Mg-8Zn-4Al (wt%) alloys, with and without a quaternary addition of 0.5wt% Ca, in the temperature range 150-200∞C has been measured using Vickers hardness testing, and precipitate microstructures have been characterised using transmission electron microscopy. The peak-aged samples have been creep tested at selected stress levels in the temperature range 125-175∞C. It is found that additions of Ca result in significant change in the commence of over-aging, even though little change was observed in the maximum hardness achievable. Results from creep tests indicate that the Ca-containing alloy has higher creep strengths and lower minimum-creep rates than the Ca-free alloy. Examinations of microstructures of peak-aged samples reveal that the majority of retained intermetallic particles in both alloys are not the cubic T phase, Mg32(Al,Zn)49. The microstructure of peak-aged samples of the Ca-free alloy contains predominantly a distribution of diamond-shaped precipitates, while an additional distribution of rod-shaped precipitates was observed in the Ca-containing alloy.

9:30 AM
The Effect of Exposure to Elevated Temperature on the Microstructure and Hardness of Mg-Ca-Zn and Mg-Ca-Zn-Si Alloys: Amir Finkel[1]; Ludmila Shepeleva[1]; *Menachem Bamberger*[1]; Eugin Rabkin[1]; [1]Technion, Dept. of Matls. Eng., Technion City, Haifa 32000 Israel

Two alloys of Mg-5wt%Ca-6wt%Zn (MCZ) and Mg-5wt%Ca-6wt%Zn-2wt%Si (MCZS) were cast into steel mold and than exposed to 160°C for up to 40 days. The dependencies of microstructure, thermal behavior, microhardness and hardness on the exposure time were determined. This enabled us to monitor the thermal stability of the cast alloys. The as structure of MCZ alloy is composed mainly of α-Mg solid solution, and in the grain boundaries 2μm large precipitation of $CaMg_2$ and eutectic structure of Mg and $Ca_2Mg_6Zn_3$ were found. The $CaMg_2$ precipitates do not appear in as-cast MCZS, whereas coarse CaMgSi grains are scattered between the matrix grains. The α-Mg grain size in both alloys was approximately 10μm. The structure of both alloys did not change during the exposure to the elevated temperature, although a small increase of the amount of inter-granular phases was observed. As-cast MCZ alloy contain less inter-granular precipitates, but the relative change in their amount during exposure to elevated temperature is higher compared to MCZS. Exposure to 160°C resulted in a decrease of micro-hardness of α-Mg grains in both alloys, but no change in the overall hardness of MCZ alloy was observed during this time, in contrast with the age hardening behavior of MCZS alloy. MCZ alloy is harder than MCZS alloy for all exposure times. Based on thermodynamic calculations utilizing the Thermo-Calc software package the composition-microstructure-hardness variations were studied and the effect of Si content in MCZS alloys on the microstructure was evaluated.

9:50 AM
Phase Identification and Microanalysis of Phases in Mg-Al-Ca Alloy System: *Koray Ozturk*[1]; Zi-Kui Liu[2]; Alan A. Luo[3]; [1]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., 107 Steidle Bldg., University Park, PA 16802 USA; [2]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., 209 Steidle Bldg., University Park, PA 16802 USA; [3]General Motors Research and Development Center, Matls. & Processes Lab., 30500 Mound Rd., Warren, MI 48090-9055 USA

A fast and efficient characterization route was employed to the Mg-Al-Ca system for the construction of the ternary phase diagram. Two alloys (Alloy A: Mg-4.5%Al-1.9%Ca and Ally B: Mg-4.5%Al-3.0%Ca) and one Mg-Al-Ca diffusion triple were used in the present investigation. The alloys were sealed under an inert atmosphere and heat treated at 290C and 370C for one week. The diffusion triple was assembled carefully using precisely machined pure elements of Mg, Al and Ca and heat treated at 370C under an inert atmosphere. Crystal structure identification of the phases was made by electron backscatter diffraction (EBD) technique. And, the chemical composition of the individual phases was determined using electron probe microanalysis (EPMA).

10:10 AM Break

10:20 AM
Magnesium Diecasting Alloy Aj62x with Superior Creep Resistance, Ductility and Diecastability: *Mihriban Ozden Pekguleryuz*[1]; Pierre Labelle[1]; Donald L. Argo[1]; Eric Baril[1]; [1]Noranda, Noranda Tech. Ctr., 240 Hymus, Montreal, Quebec H9R1G5 Canada

Magnesium diecasting alloys for elevated temperature applications are coming of age. Several research centers and companies have been working on alloy systems based on alkaline earth and rare earth alloying additions to push the limits for the creep performance of Mg-based diecasting alloys. Norandaís Mg-Al-Sr based alloys have shown superior creep performance and high-temperature performance at temperatures as high as 150-175C and stress levels as high as 50 MPa. The most recent alloy formulation AJ62x (Mg-6Al-2Sr) has in addition shown excellent castability, high ductility and superior hot-tear resistance. Based on these attributes AJ62x is positioned well for applications such as transmission cases and oil pans. In this paper, mechanical properties (creep and tensile) and microstructure of AJ62x are presented. High ductility is an added advantage for this alloy. Industrial trials indicating that the alloy is highly castable and lab-scale evaluation shows that the alloy is more resistance to hot-tearing and cracking than all other magnesium alloys and the A380 aluminum alloy.

10:40 AM
Die Cast Magnesium Alloys Ae42 and Aj52x for High Temperature Applications: *Yemi Fasoyinu*[2]; Terri Castles[2]; RÈal Bouchard[2]; Mahi Sahoo[2]; Mihriban Pekguleryuz[1]; Pierre Labelle[1]; [1]Noranda, Noranda Tech. Ctr., 240, Hymus, Montreal, Quebec H9R1G5 Canada; [2]CANMET, Matls. Tech. Lab., 568 Booth St., Ottawa, Ontario K1A 0G1 Canada

Several R&D laboratories and companies are working to develop new die cast magnesium alloys with high-temperature properties better than the AZ and AM series. These R&D initiatives were driven by the need for alloys that could be used in such applications as transmission cases and covers, and other structural parts where improved performance at higher operating temperatures (150-175C) is desirable. This paper provides a comparitive evaluation of diecast microstructures and mechanical properties of AE42 (Mg-4Al-2 Rare Earth) and AJ52x (Mg-5Al-2Sr) alloys. Second phases in the two alloys are discussed and their stability are discussed. Tensile, impact, creep and fatigue property data generated in an ongoing research program at Materials Technology Laboratory (MTL)-Canada Centre for Minerals and Energy Technology (CANMET) are compiled. The effects of test bar thickness and test temperature on mechanical properties are investigated. Alloy AJ52x exhibits significantly better creep properties than alloy AE42, especially at higher applied stress (50 MPa) and 200 hrs rupture time. The use of a multi-cavity die to produce test samples of different section thickness or geometry requires optimization of the die design and/or pressurization parameters to reduce turbulence and porosity formation during die casting operation. It is shown that less than optimum properties could occur in die cast test samples due to the presence of surface defects and centerline shrinkage and/or gas porosity.

11:00 AM
Zirconium Alloying and Grain Refinement of Magnesium Alloys: *Ma Qian*[1]; *David H. StJohn*[1]; M. T. Frost[2]; [1]The University of Queensland, CRC for Cast Metals Mfg. (CAST), Div. of Matls. Eng., Sch. of Eng., Brisbane, Queensland 4073 Australia; [2]Australian Magnesium Corporation, Ltd., PO Box 1364, QLD, Milton, BC 4064 Australia

Factors that influence alloying zirconium to magnesium with a Mg-33.3Zr master alloy and the subsequent grain refinement were discussed based on a large number of experiments conducted at the laboratory scale. Particular attention was given to the influence of alloying temperature, stirring pattern, Fe pick-up from steel crucibles and residence time prior to pouring on the soluble and total zirconium in the final alloy. It was shown that excellent grain refinement was dictated by both soluble and total zirconium in the melt immediately prior to pouring. An ideal zirconium alloying process should end up with both high soluble and high total zirconium in order to achieve the best grain refinement in the final alloy. The mechanism of grain refinement of magnesium by zirconium was discussed based on the grain structures observed in the final alloy and the role of soluble and total zirconium in the grain refinement of magnesium.

11:20 AM
Grain Refinement of Magnesium Alloys Using Rolled ZirmaxÆ Master Alloy (Mg-33.3Zr): *Ma Qian*[1]; *David H. StJohn*[1]; M. T. Frost[2]; R. M. Barnett[1]; [1]The University of Queensland, CRC for Cast Metals Mfg. (CAST), Div. of Matls. Eng., Sch. of Eng., Brisbane, Queensland 4072 Australia; [2]Australian Magnesium Corporation, Ltd., PO Box 1364, Milton, BC QLD 4064 Australia

Owing to the limited solubility of zirconium in magnesium, almost all of the zirconium contained in the ZirmaxÆ master alloy (Mg-33.3Zr) is present in the form of nearly pure zirconium particles. Of them, individual particles and solid particle clusters greater than 5 microns in dimension account for approximately 70% of the area of all zirconium particles observed in the microstructure. Rolling a ZirmaxÆ master alloy ingot can allow coarse zirconium particles and particle clusters to fragment. Comparisons were made of the grain refining ability between as-cast ZirmaxÆ and deformed ZirmaxÆ by adding both forms of master alloy to pure magnesium under the same conditions. It was found that the use of rolled ZirmaxÆ delivered higher total and soluble zirconium, therefore better grain refinement in the final alloy than the use of as-cast ZirmaxÆ. The improved performance of rolled ZirmaxÆ master alloy was primarily attributed to less intense zirconium particle settling.

Materials Lifetime Science and Engineering - III

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; D. Gary Harlow, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015-3085 USA; Dwaine L. Klarstrom, Haynes International, Inc., Kokomo, IN 46904-9013 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Robert P. Wei, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015 USA

Wednesday AM Room: 18
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Dwaine L. Klarstrom, Haynes International Inc., Kokomo, IN 46904-9013 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA

8:30 AM Invited
On the Development of Life Prediction Methodologies for the Failure of Human Teeth: R. K. Nalla[1]; V. Imbeni[1]; J. H. Kinney[2]; S. J. Marshall[2]; *R. O. Ritchie*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]University of California-San Francisco, Dept. of Preventive & Restorative Dental Scis., San Francisco, CA 94143 USA

Human dentin is known to be susceptible to failure under cyclic loading. However, there are few reports that quantify the effect of such loading, especially when viewed in conjunction with the fact that a typical tooth experiences typically a million loading cycles annually. In the present study, a systematic investigation is described of the effects of prolonged cyclic loading on human dentin in a simulated physiological environment. In vitro stress-life (S/N) data are discussed in the context of possible mechanisms of fatigue damage and failure. Stiffness loss data collected in situ during these tests are used to calculate crack-growth velocities and the fatigue thresholds, and are presented as plots of the crack-propagation rates (da/dN) as a function of the stress-intensity range (deltaK). The S/N and da/dN-deltaK data are discussed in light of a framework for a fracture mechanics-based methodology for the prediction of the fatigue life of human teeth.

9:00 AM Invited
Fatigue of Interfaces in Adhesive Bonds: *J. K. Shang*[1]; T. Du[1]; [1]University of Illinois at Urbana-Champaign, 1304 W. Green St., Urbana, IL 61801 USA

Polymer adhesives are widely used to bond advanced materials in structural applications where durability of the adhesive bond remains a major concern, especially under cyclic loading in an aggressive environment. In this paper, we will focus on durability of the interface in the adhesive bond. Three experimental techniques for characterizing durability of the interface will be introduced. Effects of mechanical and environmental variables on durability of polymer-metal interface will be examined. Efforts for improving durability of polymer-metal interfaces by using self-assembled monolayers will be described. Finite element models will be presented to predict fatigue lives of adhesive joints based on damage tolerant and total-life approaches.

9:30 AM
Predicting Material Consumption by Cyclic Oxidation Spalling Models: *James L. Smialek*[1]; [1]NASA Glenn Research Center, Matls. Div., 21000 Brookpark Rd., MS 106-1, Cleveland, OH 44135 USA

The cyclic oxidation process has been described by an iterative scale growth and spallation model. Inputs include the selection of an oxidation growth law and a spalling geometry, plus oxide phase, growth rate, spall constant, and cycle duration. Output includes weight change, the amounts of retained and spalled oxide, the total amount of oxygen and metal consumed, and the terminal rates of weight loss and metal consumption. A computer program, COSP for Windows, was used to run multiple families of curves demonstrating the functional behavior of the outputs for various input parameters. A simple summation series has also been developed for a special case of interfacial spallation, producing closed form equations and the expression of various descriptive parameters as direct functions of the inputs. By suitably normalizing the weight change and cycle number, all cyclic oxidation model curves can be represented by a dimensionless, universal expression. Comparison to actual data validates the model prediction.

9:50 AM Invited
Corrosion Damage Functions and Life Prediction: *Russell H. Jones*[1]; [1]Pacific Northwest National Laboratory, Matls. Sci., PO Box 999, MSIN P8-15, Richland, WA 99352 USA

Corrosion damage can lead to reduced operational lifetimes. Often this damage is not as obvious as general corrosion but takes the form of pits, intergranular corrosion, crevice corrosion and hydrogen absorption. These types of corrosion damage lead to stress corrosion cracking, hydrogen induced cracking and corrosion fatigue. A critical step in defining a corrosion damage function is determining the relationship between the corrosion damage, the resulting crack propagation mechanism and component lifetimes. The sequence of events is often some localized corrosion event such as pitting, transition of the pit to a planar crack, propagation of this short crack, transition of the short crack to long crack conditions and continued propagation through stage I, II and III of the long crack SCC regimes. A description of critical corrosion damage processes and examples of the transition to long crack SCC conditions will be discussed.

10:20 AM Invited
Deuterium-Induced Fracture of Beryllium Films from Exposure to Experimental Reactor Environments: *Neville R. Moody*[1]; Rion A. Causey[1]; David F. Bahr[2]; Kenneth L. Wilson[1]; [1]Sandia National Laboratories, PO Box 969, MS 9404, Livermore, CA 94551-0969 USA; [2]Washington State University, Pullman, WA 99164 USA

Beryllium is a prime candidate material in the design of the International Thermonuclear Experimental Reactor (ITER) where it is proposed for use in components exposed to the hot tritium plasma. It has excellent thermal conductivity and a lower affinity for tritium than other candidate materials. However, deposition of energetic tritium ions with beryllium atoms can lead to formation of films that delaminate and blister. We therefore studied blister formation in beryllium films by replacing tritium with deuterium and using Atomic Force Microscopy to characterize blister sizes and shapes. Mechanics-based models were then combined with these measurements to determine the effect of deuterium on interfacial fracture energies. The analysis showed that deuterium induced very high compressive residual stresses in beryllium films. More importantly, the results showed that deuterium markedly lowered the interfacial fracture strength of these films. The authors gratefully acknowledge the support of the USDOE through Contract DE-AC04-94AL85000.

10:50 AM
Quantitative Cyclic Oxidation Modelling on Alumina Former: *Dominique Poquillon*[1]; Daniel Monceau[1]; [1]CIRIMAT, INPT/ENSIACET, 118 Rt. de Narbonne, Toulouse 31077 France

Structural materials for high temperatures are often subjected to cyclic conditions of atmosphere, mechanical loading and temperature. Most components used over 900∞C are made of alumina formers alloys. Oxidation is an important source of degradation which may even control the time of life of these systems, especially over 1050∞C. Lifetime prediction then requires the quantitative assessment of the metal rate consumption. Modeling of the oxide scale growth and degradation kinetics, as well as in-situ direct measurements are developed simultaneously in order to provide a quantification of cyclic oxidation experiments, to allow extrapolations for long life prediction, to supply data in diffusion models, but also to test the physical models which describe oxide spallation.

11:10 AM
Effects of Test Sample Thickness, Coating and Environment on the Creep Rate and the Time to Rupture of Single Crystal Nickel Base Superalloy: *SÈbastien Dryepondt*[1]; Eric Andrieu[1]; Daniel Monceau[1]; Fabrice Crabos[2]; Cyril Vernault[2]; Pierre Monge-Cadet[2]; [1]ENSIACET/CIRIMAT, 118 rte. de Narbonne, Toulouse, Cedex 4 31077 France; [2]Turbomeca, DE/MTA/EAP, Bordes 64511 France

Creep tests were carried out on thin plates and cylindrical MC2 samples coated or not with NiCoCrAlYTa, at 1150∞C, 80 and 140 MPa. The creep curves obtained in laboratory air show an increase in the steady-state creep rate leading to a decrease in time to rupture in the case of uncoated thin samples compared with thick ones. The gain in term of life duration increases along with decreasing the creep stress. The coating improves the creep life duration for thin samples whereas it has no significant effect for thick samples. This observation suggests that environment has a deteriorating effect on uncoated MC2 creep properties, which can be erased with the use of a NiCoCrAlYTa coating. Further creep tests have been performed to estimate the influence of environment under different temperatures and loading conditions. Interactions between oxidation and creep properties are studied using experimental setups under controlled atmosphere.

11:30 AM
Influence of Environment on Mechanical Behavior of Alloy 718 at 650∞C: *Veronique Garat*[1]; Bernard Viguier[2]; Jean Marc Cloue[3]; Eric Andrieu[2]; [1]FRAMATOME-ANP, 10, rue Juliette Recamier, Lyon 69456 France; [2]ENSIACET, CIRIMAT, 118, rte de Narbonne, Toulouse 31077 France; [3]Framatome-ANP, 1, rue Baptiste Marcet, Le Creusot 71205 France

The 718 alloy is widely used in aeronautical industry for its good creep and corrosion resistance. To study the influence of oxidation on the flow rules, creep tests were carried out at 650∞C on thin specimens (0.27 mm), under vacuum and under laboratory air testing conditions. The results of these tests showed that the steady-state creep strain rate is higher under laboratory air than under vacuum, whereas no damage, in terms of intergranular cracking, is observed on the surface specimen. In parallel, SIMS analysis were performed on specimens aged from 1 to 100 hours, at 650 or 700∞C under air and vacuum conditions. This characterization allows us to link the type of oxide formed to the modification of creep behavior of 718 alloy. The consequences of those observations on both flow rules and in the field of oxidation assisted cracking are addressed.

11:50 AM
The Behavior of HaynesÆ C-2000Æ Superalloy Subjected to Strain-Controlled Fatigue Loading: *R. L. McDaniels*[1]; L. Chen[1]; R. Steward[1]; P. K. Liaw[1]; R. A. Buchanan[1]; D. L. Klarstrom[2]; [1]The University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The strain-controlled fatigue behavior of the new HaynesÆ Hastelloy C-2000 nickel-chromium-molybdenum superalloy was investigated at total strain ranges from 0.4% to 2.0% and at temperatures from 24∞C to 927∞C. The test specimens were subjected to fully reversed, push-pull, strain-controlled fatigue tests under axial strain range control. The results indicated that both test temperature and total strain range had a significant effect on the strain-controlled fatigue properties of the alloy. The alloy exhibited cyclic hardening, cyclic stability, or cyclic softening, depending on the temperature and strain to which the individual specimens were subjected. Coffin-Manson and Holloman parameters were found and plotted. Microstructural analysis of the as-received and tested specimens was also conducted using optical and scanning electron microscopy to provide a mechanistic understanding of the fatigue behavior. The present work is supported by the NSF Integrative Graduate Education and Research Training (IGERT) program, with Dr. W. Jennings and Dr. L. Goldberg as contract monitors and the Haynes International, Inc., with Dr. D. Klarstrom as the contract monitor.

Materials Processing Fundamentals: Heat and Fluid Flow: Modeling and Property Effects

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee, EPD-Process Fundamentals Committee
Program Organizers: Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA; Princewill N. Anyalebechi, Grand Valley State University, L. V. Eberhard Center, Grand Rapids, MI 49504-6495 USA

Wednesday AM — Room: 1A
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Rodney L. Williamson, Sandia National Laboratories, Liquid Metal Procg. Lab., Albuquerque, NM 87185-1134 USA; Uday Pal, Boston University, Dept. of Mfg. Eng., MA 02446 USA

8:30 AM
Model-Based Control of the Vacuum Arc Remelting Process: *Rodney L. Williamson*[1]; Joseph J. Beaman[2]; David K. Melgaard[1]; [1]Sandia National Laboratories, Dept. 1835, Liquid Metal Procg. Lab., PO Box 5800, MS 1134, Albuquerque, NM 87185-1134 USA; [2]University of Texas, Mechl. Eng., 700 Texas Ave., Austin, TX 78705 USA

A difficult control problem arises when a critical process variable responds in a nonlinear, history dependent fashion to a control input variable. Such a problem arises in vacuum arc remelting (VAR), an industrial metallurgical process used to cast large alloy ingots. Part of the VAR control problem consists of adjusting arc power to control melt rate. However, melt rate also depends on the electrode temperature distribution (ETD) causing the melt rate response to be history dependent and nonlinear. The situation is complicated further because the ETD cannot be measured, and electrode weight measurements are very noisy. To address this problem, a melting model was used to estimate the ETD. The model was incorporated into a control system that has been successfully used in VAR of nickel-base superalloys and aerospace titanium alloys. Data from laboratory and industrial tests show that the goal of accurate, ìinstantaneousî melt rate control has been achieved.

9:00 AM
An Investigation of Particulate Flows by Particle Image Velocimetry and Mathematical Modeling: Daniel Steingart[1]; *James W. Evans*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94610 USA

The flow of large dense particles has been investigated experimentally and by mathematical modeling. The experiments entail the measurements of the flow of beds of particles through orifices of various sizes by particle image velocimetry (PIV). This technique provides both the flow rate through the orifice and the distribution of particle velocities within the bed. Measurements have been made in air and in water. In some cases the water measurements were carried out with simultaneous flow of water through the orifice. In a few cases a boroscope was used to examine the details of movement of particles within the bed. The model of Hong and Caram has been adapted to simulate the dynamics of bed flow and shows a good fit to the experimental data.

9:20 AM
Flow of Steel in the Mold Region During the Continuous Casting of Steel: X. G. Zhang[1]; D. Steingart[1]; C. D. Seybert[2]; *James W. Evans*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA

Particle image velocimetry and a water model, with size of 1840 by 280mm, were used to study the fluid flow phenomena happening in continuous casting of steel. Two types of solidified shell, smooth and rough, were employed to simulate different liquid-solid interfacial condition. The effect on flow behavior of nozzle angle and submergence, as well as casting speed, were investigated quantitatively. The results show that: 1) There exist two large separate re-circulation loops above and below the fluid jet in the mold under the smooth interface condition. However, in the case of a rough surface, representing a coarse dendritic structure at the interface, it was found that not only was the velocity of fluid decreased, but that there were additional small vortices in the upper region of the mold. 2) The angle and submergence depth of the nozzle are two important factors that affect the flow pattern; they can even change the flow direction, 3) The higher the casting speed, the higher the velocity of the jets emerging from the nozzle ports (and traversing the liquid pool to the narrow face of the steel shell) and the higher impacting point on that face. Higher casting speed gives rise to large fluctuations of the meniscus with resulting defects. A Large Eddy Simulation (LES) model was used to compute the fluid flow.

9:40 AM
Effect of Initial Sample Temperature on the Boiling Water Heat Transfer During Quenching of a Stainless Steel Plate: *Dianfeng Li*[1]; Mary A. Wells[1]; Steve L. Cockcroft[1]; Gary T. Lockhart[1]; [1]The University of British Columbia, Dept. of Metals & Matls. Eng., Frank A. Forward Bldg., 6350 Stores Rd., Vancouver, BC V6T1Z4 Canada

A 2-D Inverse Heat Conduction (IHC) model was developed and verified to quantitatively determine the boiling water heat transfer during water quenching. Using a custom-built rig, with a single nozzle above the sample, a series of tests to examine the effect of initial sample temperature on the boiling water heat transfer during quenching of an AISI 316 plate were done. Sample starting temperatures ranged from 1000∞C-600∞C and the study demonstrated that when the starting temperature of the sample was above the Leidenfrost point no effect of temperature on the boiling water curve was seen. However, when the starting temperature of the sample was below the Leidenfrost point the temperature had a strong effect on the calculated boiling curve. This work shows that boiling water heat transfer is not only a function of the sample surface temperature but is also dependent on the thermal history experienced by the sample.

10:00 AM
Experimental Study of the Bubble-Burst Phenomenon at the Surface of a Liquid Steel Bath: Anne-GwÈnaÎlle GuÈzennec[1]; Jean-Christophe Huber[2]; *Fabrice Patisson*[1]; Philippe Sessiecq[1]; Jean-Pierre Birat[2]; Denis Ablitzer[1]; [1]Ecole des Mines, LSG2M, UMR 7584 CNRS-INPL, Parc de Saurupt, Nancy 54042 France; [2]IRSID, voie romaine, BP 30320, MaiziËres-lËs-Metz 57283 France

We have developed an experimental device for studying the main mechanism of dust formation in electric arc furnace steelmaking: the burst of gas bubbles at the liquid steel surface. As in the case of the air-water system, the bubble-burst process takes place in three steps: breaking of the film cap, projection of film drops, and projection of jet drops. The film breaking and the jet drop formation are observed with high-speed video. The film drop aerosol enters a particle counter, which characterizes the drops in size and number. Results are presented and discussed. The quantification of both types of projections leads to the conclusion that the film drop projections represent the major source of dust. We currently are investigating the influence of the bubble size on the amount of film drop projections, with the aim of checking the theoretical existence of an opimal bubble size that minimizes dust emission.

10:20 AM Break

10:40 AM Cancelled
Energy Efficiency of Fluidized Systems Used for Charge Preheating Before Electric Furnaces: *Kamal Adham*[1]; Cassandra Lee[1]; [1]Hatch Associates, 2800 Speakman Dr., Sheridan Sci. & Tech. Park, Mississauga, Ontario L5K 2R7 Canada

11:00 AM
Fundamental Study of the Interaction of Liquids with Rotating Disks: *Panos Tsakiropoulos*[1]; Yawei Wang[1]; [1]University of Surrey, Sch. of Eng. (H6), Mechl., Matls. & Aeros. Eng., Guildford, Surrey GU2 7XH England

The flow of a liquid on a stationary or rotating disk is widely encountered in many engineering practices. For example, the semiconductor industry uses this process to produce very thin and uniform coatings of photo resist films. In optical and magnetic recording media a thin layer of lubricant is used to prevent the slider from excessive wear during the start up and turnoff. TV manufacturers coat the TV screen with a thin film of coating. A thin film produced by a high speed-rotating disk provides a very important means for evaporation and drying of milk, soaps, detergents etc. The space industry exploits the flow of liquids on rotating disks in vapour absorption refrigeration systems. The metallurgical industry produces elemental or alloyed powders using centrifugal atomisation (CA). Near net shape metallic components can be manufactured by combining CA and spray forming (SF). Clean processing of alloy melts, which is a requirement of the aerospace and power plant generation industries, is also possible by combining cold hearth clean melting with CA or CA/SF. The paper will discuss the interaction of liquids with rotating disks and the characteristics of the spray as a function of flow rate, disk speed, and distance from injector to the point of impact on rotating disk. The outlines of centrifugal atomised sprays, studied using digital camera, will be compared. Results on the distribution of droplet size and droplet velocity in the spray, obtained using a Laser Phase Doppler Particle Analyser, will be presented. A theoretical analysis of the dependence of droplet formation mechanisms and spray characteristics on flow rate, disk speed and height from injector to disk will also be presented.

11:20 AM
Modeling Chip Formation of an Aluminum Alloy Using a Continuum Hydro-Dynamic Code: *Jaton Nakia Wince*[1]; Judy Schneider[2]; [1]Mississippi State University, Mechl. Eng., PO Box 1942, Mississippi State, MS 39762 USA; [2]Mississippi State University, Mechl. Eng., MS 9552, 210 Carpenter Bldg., Mississippi State, MS 39762 USA

This paper presents the simulation of chip formation in orthogonal metal cutting utilizing the predictive capabilities of DYNA 3D. The Johnson and Cook constitutive model for materials, Aluminum 6061 T6 alloy, were incorporated into the simulation to account for the effects of strain hardening, strain rate hardening, and thermal softening effects of the materials being machined. The model was compared to experimentally measured metal cutting plasticity parameters: shear plane angle and shear front angle, to verify the simulations accuracy.

11:40 AM
High Temperature Fatigue and Mechanical Properties of a Die Steel H3: *Eun-Gu Yoh*[1]; Y.-S. Lee[2]; [1]Kookmin University, Auto. Eng., 861-1 Chungneung-Dong, Sungbuk-Gu, Seoul, Seoul 136-702 Korea; [2]Kookmin University, Mechl. Eng., 861-1 Chungneung-Dong, Sungbuk-Gu, Seoul 136-702 Korea

The temperature in hot forming of metallic materials, such as hot extrusion and hot forging, ranges from 300 to 1200°C. Correspondingly, the die also exhibits very high temperatures very close to that of a work piece and its life is limited generally by high temperature fatigue. Thus, the analysis of high temperature fatigue would need the mechanical properties over the wide ranges of temperature. However, very few studies on the high temperature fatigue of brittle materials have been reported. Especially, the study on the fatigue behavior over such transition temperature regime is very rare. In this paper, the stress-strain curves and stress-life curves of a die steel such as H3 are experimentally obtained. The wide ranges of temperature from 300 to 1200°C are considered in experiments. And the transition temperature zone is carefully examined.

Materials Processing Under the Influence of Electrical and Magnetic Fields - V

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Joanna R. Groza, University of California-Davis, Chemical Engineering Material Science Department, Davis, CA 95616 USA; George S. Dulikravich, The University of Texas at Arlington, Multidisciplinary Analysis, Inverse Design, and Optimization (MAIDO) Program, Department of Mechanical and Aerospace Engineering, Arlington, TX 76019 USA; Nagy H. El-Kaddah, University of Alabama, Department of Metallurgical & Materials Engineering, Tuscaloosa, AL 35487-0202 USA; James W. Evans, University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA; Zuhair Munir, University of California, College of Engineering, Davis, CA 95616-5294 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Wednesday AM — Room: 14A
March 5, 2003 — Location: San Diego Convention Center

Session Chair: Nagy H. El-Kaddah, University of Alabama, Dept. of Metallurgl. & Matls. Eng., Tuscallosa, AL 35487-0202 USA

8:30 AM Opening Remarks

8:40 AM
Solid Ceramic Particulate Behavior into Molten Steel Bath Heated by Induced Currents: *Ignacio Erauskin*[1]; IÒigo Agote[2]; Ibon Mitxelena[2]; Mikel Asensio[2]; [1]Fundacion Inasmet, Matls. & Processes, Paseo Mikeletegi, 2 Parque Tecnologico., San Sebastian Spain; [2]Fundacion Inasmet, Paseo Mikeletegi, 2. Parque TecnolÛgico, San Sebasti·n Spain

The improvements that could be obtained in the properties of cast structural alloys through the reinforcing its matrix with hard, microscopic and dispersed ceramic particles as TiC type carbides, are accepted and recognized. In this sense, the development of a liquid metallurgy process enabling the reinforcement by means of the addition of the ceramic material to the molten metal in the melting furnace would become an important advance in the metallic materials field. Nevertheless, these ceramic products are prone to the coalescence and have poor wettability into molten baths as steel, superalloys, etc. A factor that minimizes these problems is the bath stirring during the ceramic particle addition, this stirring being produced, for instance, by the Foucault currents in a induction melting furnace. We have empirically verified, however, that the stirring magnitude, as function of the furnace power and frequency, and material density and resistivity, has a high influence in the ceramic behavior, so that, the working out of a numerical simulation software about this matter would allow the development of structural materials with improved properties.

9:00 AM Invited
Magnetic Field Alignment of Polymers to Enhance Material Properties: *Mark E. Smith*[1]; Brian C. Benicewicz[2]; Elliot P. Douglas[3]; Jim D. Earls[4]; Ralph D. Priester[4]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., Polymers & Coatings, MS E 549, Los Alamos, NM 87545 USA; [2]Rensselaer Polytechnic Institute, Troy, NY 12180 USA; [3]University of Florida, Gainsville, FL 32611 USA; [4]The Dow Chemical Company, Freeport, TX 77541 USA

Liquid crystalline polymers can be orientated by use of electrical or magnetic fields; however the majority of high molecular weight thermoplastic resins typically suffer from high melt viscosities, which limits the utility of this process method for materials production. Thermoset liquid crystals allow the viable application of electrical and magnetic orientation for solid part production. This research is focused on the curing of a liquid crystalline epoxy with a diamine crosslinker in the presence of magnetic fields. The results of statistical design experiments will be presented on the three main processing variables: magnetic field strength, time in field, and extent of B-staging. The magnetic processing method allows a substantial manufacturing window in which material of tailored properties can be produced. Underlying principles of the orientation mechanism and the nature of the liquid crystalline formation and stability are also presented.

9:30 AM
The Aligned Solidification Structure of MnBi in Semi-Solidified Bi-Mn Alloy with a Static Magnetic Field: *Zhongming Ren*[1]; Hui Wang[1]; Kang Deng[1]; Kuangdi Xu[1]; [1]Shanghai University, Dept. Matls., 149 Yan Chang Rd., Shanghai 200072 China

The macrostructures of the Bi-3%Mn, 6%Mn, and 20%Mn alloys solidified under the influence of a high static magnetic field (up to 10T) have been investigated experimentally. It is shown that the magnetic field influenced behavior of the primary phase MnBi crystals significantly. The morphology and alignment direction of the MnBi crystals were depended on the melting temperature and magnetic field. In the case of semi-melting temperature, the MnBi crystals were aligned along with the direction of the magnetic field, and in the case of whole melting temperature, the MnBi crystals were aligned perpendicularly to the direction of the field. The alignment tendency and the average length of elongated MnBi crystals of MnBi increased with the increase of the applied field and the solidification time. A model was proposed to explain the alignment and orientation growth of MnBi crystals in a magnetic field in terms of the magnetic anisotropy of the crystals and the interaction between them.

9:50 AM
Directional Solidification of Immiscible Alloys Under an Applied Electromagnetic Force Field: *Nagy El-Kaddah*[1]; Lirong Tong[1]; [1]The University of Alabama, Dept. of Metallurgl. Eng., Box 870202, Tuscaloosa, AL 35487 USA

This paper describes an electromagnetic approach for solidification processing of immiscible alloy composites. It is based on the use of the electromagnetic force generated by passing electric current through the melt and crossing it by a uniform magnetic field to offset and modulate the buoyancy force on dispersed droplets during solidification. The technique was used to study the macrostructure development of Zn-10 wt% Bi immiscible alloy over a wide range of effective gravity values in a vertical unidirectional solidification system. A mathematical model for describing the growth of nucleated dispersed phase in the two-liquid phase region ahead of the solidification front and the entrapment of these droplets by the moving solid-liquid interface in vertical unidirectional solidification systems has been developed. The model is used to study the particle size distribution in unidirectional solidified Zn-Bi hypermonotectic alloys at reduced gravity conditions. It has been found that the particle size and distribution in the solidified alloy depends on solidification rate and the ratio of effective gravity to thermocapillary forces. The model was found to reasonably predict the experimentally measured particle size and distribution over the entire range of effective gravity investigated.

10:10 AM
Magnetized Fiber Orientation and Concentration Control in Solidifying Composites: *George S. Dulikravich*[1]; Marcelo J. Colaco[1]; Thomas J. Martin[2]; [1]University of Texas at Arlington, Mech. & Aeros. Eng. Dept., Multidisciplinary Analy., Inverse Design & Optimization (MAIDO) Prog., UTA Box 19018, Arlington, TX 76018 USA; [2]Pratt & Whitney Engine Company, Turbine Discipline Eng. & Optimization Grp., 400 Main St., MS 165-16, E. Hartford, CT 06108 USA

If short carbon fibers (5-10 microns in diameter and 200 microns long) are vapor-coated with a thin layer (2-3 microns) of a ferromagnetic material like nickel, the fibers will respond to the applied magnetic fields by rapidly rotating and translating so that they become aligned with the magnetic lines of force. We have developed an improved analytical model and a numerical algorithm for the prediction of magnetic force lines inside a flowing solidifying melt. This computer code was combined with our hybrid constrained optimizer that minimized a normalized sum of least square differences between the user-specified and the predicted local magnetic lines of force geometric patterns. We have utilized this software package and the interactive graphics software to evaluate the strengths, locations, and orientations of magnets needed for generate specified magnetic field lines in the solidifying composite thus verifying the feasibility of this manufacturing process.

10:30 AM Break

10:50 AM
Microstructure Formation During BiMn/Bi Eutectic Growth with Applied Alternative Electric Fields: *Lili Zheng*[1]; *Yuan Ma*[1]; David J. Larson[2]; [1]University at Stony Brook, Dept. of Mechl. Eng., Stony Brook, NY 11794-2300 USA; [2]University at Stony Brook, Dept. of Matls. Sci. & Eng., Stony Brook, NY 11794-2275 USA

This paper is to demonstrate that the presence of an electric field can be used to control materials microstructure formation. Special efforts have been made to identify the foremost process control parameters that affect the interface dynamics, and thermoelectric effects on materials microstructure formation during directional solidification. A computational model that integrates microscopic analysis to macroscopic model has been developed and applied to directional solidification of BiMn/Bi eutectic in the presence of electric fields. Numerical results demonstrated that in addition to process parameters, microstructure formation strongly depends on intensity, and frequency of applied current, and it changes spontaneously as an electric field is applied. Predicted patterns of microstructures have qualitatively agreed with the experiments. The results indicate the feasibility of utilizing electric fields to control microstructure formation during eutectic growth.

11:10 AM
Generating Strange Interactions in Particle Suspensions: Classical Molecules and Particle Foams: *James E. Martin*[1]; Robert A. Anderson[1]; Rodney L. Williamson[1]; [1]Sandia National Laboratories, Organization 1120, MS 1421, PO Box 5800, Albuquerque, NM 87185-1421 USA

We have discovered a method of generating strange interactions in magnetic particle suspensions. These interactions can be attractive or repulsive, and lead to a variety of unexpected effects, including stable particle clusters with molecular geometries, the emergence of a particle foam phase, and amusing collective particle dynamics. Through this interaction novel isotropic particle structures can be made that cannot be produced by any other known means, leading to a new class of composite materials with highly optimized properties.

11:30 AM
Optimization of Wall Electrodes for Electro-Hydrodynamic Control of Charged Particle Depositions During Solidification: *George S. Dulikravich*[1]; Marcelo J. Colaco[1]; [1]University of Texas at Arlington, Mech. & Aeros. Eng. Dept., Multidisciplinary Analysis, Inverse Design & Optimization (MAIDO) Program, UTA Box 19018, Arlington, TX 76019 USA

In the case of electro-hydrodynamics (EHD), flow-field of electrically charged particles in a melt is influenced by an externally applied electric field in the absence of a magnetic field. Solidification front shape, distribution of the charged particles in the accrued solid, and the amount of accrued solid phase in such processes can be influenced by an appropriate distribution and orientation of the electric field. We have developed numerical analysis software for EHD solidification that is based on a finite volume method. This algorithm was then combined with a constrained optimization algorithm to determine the best locations of wall electrodes that will provide the most uniform generation and distribution of electrically charged particles in the accrued solid phase.

Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction: Macroscale Modeling & Simulation

Sponsored by: Structural Materials Division,
Program Organizers: James M. Larsen, US Air Force, Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA; Leo Christodoulou, Defense Advanced Research Agency, Arlington, VA 22203-1714 USA; William J. Hardman, Naval Air Systems Command, Propulsion and Power Directorate (AIR 4.4.2), Patuxent River, MD 20670-1534 USA; Andrew J. Hess, Naval Air Systems Command, Propulsion and Power Directorate, Patuxent River, MD 20670-1534 USA; J. Wayne Jones, University of Michigan, College of Engineering, Department of Materials, Science and Engineering, Ann Arbor, MI 48109 USA; Stephan M. Russ, Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA

Wednesday AM — Room: 16A
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Jay R. Jira, US Air Force, AF Rsrch. Lab. Matls & Mfg Direct., WPAFB, OH 45433-7817 USA; Robert A. Brockman, University of Dayton Research Institute, Dayton, OH 45469-0110 USA

8:30 AM
A USN Strategy for Mechanical and Propulsion Systems Prognostics with Demonstration Results: *William J. Hardman*[1]; [1]NAVAIR, AIR 4.4.2, B106, Unit 4, 22195 Elmer Rd., Patuxent River, MD 20670-1534 USA

A US Navy strategy has been generated to develop, integrate and demonstrate diagnostics, prognostics, health monitoring and life management for propulsion and mechanical systems. How this overall strategy has evolved and its current status will be presented. The SH-60 program was initiated as the first proof-of-concept effort to develop, demonstrate, and integrate available and advanced mechanical diagnostic technologies for propulsion and power drive system monitoring. Included in these technologies were various rule and model based analysis techniques that were applied to demonstrate and validate various levels of diagnostic and trending capabilities. Recently there has been increased emphasis on prognostic capabilities. As used in this paper, prognostics is the capability to provide early detection of the precursor and/or incipient fault condition to a component or sub-element failure condition; and to have the technology and means to manage and predict the progression of this fault condition to component failure. The benefit of this prognostic approach is increased safety and significantly reduced supportability costs over the aircraft life cycle; enabling better management of both existing and potential aircraft system faults. This prognostic philosophy, its benefits, and envisioned implementation will be further embellished. These will be discussed and updated with particular attention to recent gear fault detection results and capability demonstrations.

9:00 AM Invited
Calibration of Failure Mechanism-Based Prognosis with Vibratory State Awareness Applied to the H-60 Gearbox: *Gregory J. Kacprzynski*[1]; Avinash Sarlaskar[1]; Andrew J. Hess[2]; [1]Impact Technologies, LLC, 125 Tech Park Dr., Rochester, NY 14623 USA; [2]Naval Air Systems Command, 22195 Elmer Rd., Patuxent River, MD 20670 USA

This paper describes a generic prognostic module architeture configured for use on the H-60 IGB spiral bevel pinion gear. The software module integrates advanced stochastic failure mode modeling, failure progression information from vibration features, and run-to-failure experience bases to enable IGB pinion gear failure predictions in the H-60 critical drive train. The state-of-the-art structural/material level modeling aspects of the module includes the utilization of both a stochastic sub-zone crack initiation model and a 3D linear fracture mechanics model. Secondly, the module is engrained with an adaptive model updating techniques for ìtuningî key failure mode variables at a local material/damage-site based on fused vibration features. The failure rate prediction strategies are implemented within a probabilistic framework to directly identify confidence bounds associated with IGB pinion failure progression. The overall modeling scheme is aimed at minimizing inherent modeling and operational uncertainties by updating material/fatigue properties, crack propagation rates etc. via sensed system measurements that evolve as damage progresses. The results of seeded fault, run-to-failure tests on the IGB pinion gear will be provided and compared to prognostic module predictions.

9:25 AM Invited
Modeling of a Gear with EDM Crack: *Alan C. Leung*[1]; Virginia G. DeGiorgi[2]; [1]Nova Research (Naval Research Laboratory), Multifunctl. Matls.-6353, 1900 Elkin St., Ste. 230, Alexandria, VA 22308 USA; [2]Naval Research Laboratory, Multifunctl. Matls.-6353, 4555 Overlook Ave. SW, Washington, DC 20375 USA

Comparison of damage states during a structureís operation and prediction of the effects structural performance is a necessary first step in the process known as prognostics. The process developed must provide meaningful engineering data and be sensitive to small changes in material performance tracked by the material state awareness metric that monitors damage at the micro-structural scale. Such structures of interest are power trains. Gears, a key component to power trains, are the primary focus of this work. A 2-D FE model of a gear set was developed. A crack is placed on one gear and varied in length. The gears are subjected to varying amounts of torque. Displacements between gear teeth during gear contact are calculated. Relations between the displacement of gear teeth and the increasing crack length allow a means of assessing and monitoring gear damage with associated deformations. Comparisons are made with experimental data from a gear.

9:50 AM Break

10:20 AM Invited
Integrating Structural Analysis and Experimental Methods: *Robert A. Brockman*[1]; [1]University of Dayton Research Institute, 300 College Park Ave., MS 0110, Dayton, OH 45469-0110 USA

Analytical models of turbine engine components are reused in the event of changes in mission requirements, analysis and life prediction methodology, or fleet management philosophy. However, existing models of fielded systems contain no means of integrating state awareness information into the reanalysis process. Achieving the goal of comprehensive damage prognosis requires that measured data from numerous sources be assembled and used continually to sharpen analytical predictions of performance and potential failure. The problems involved in integrating state awareness data into structural analysis methods are similar to those encountered in integrating laboratory experiments and analytical models. Both require some reorganization of the analytical models and processes. This paper focuses on the role of structural analysis methods and software in materials prognosis, and how selected methods may need to be restructured to integrate known diagnostic data into refined predictions of component performance and life.

10:45 AM Invited
Prediction of Elevated Temperature Crack Growth in Spin Pit Tests: *Robert H. Van Stone*[1]; [1]GE, Aircraft Engines, 1 Neumann Way, MD K105, Cincinnati, OH 45215 USA

Accurate crack growth prediction methods are a necessary element in the use of advanced crack detection prognostic methods to extend the lives of rotating gas turbine components. Fracture mechanics methods used to predict the cyclic crack growth lives (without time dependent or surface enhancement effects) are well established. Linear elastic fracture mechanics methods have now been developed which accurately predict the acceleration associated with hold times at elevated temperatures using a linear superposition of cyclic and static crack growth rates. The beneficial effects of surface residual compressive stress such as those induced by shot peening can also be predicted using the superposition of the stress intensities calculated from the applied stress and a portion of the residual stress. The accuracy of these methods are assessed by comparing predicted and experimentally measured crack propagation lives for laboratory tests. These same methods will be used to predict the growth of cracks in spit pit tests planned to demonstrate the utility of advanced crack detection prognostics.

11:10 AM Invited
Materials Prognosis for Legacy Engines: *Bryon J. Wicks*[1]; [1]Department of Defence, DSTO-PSL-AVD, Box 4331, GPO, Melbourne, Victoria 3001 Australia

The main factor affecting the airworthiness, availability and through life support cost of legacy engines is the removal and replacement of critical rotating components. A safe life method is conventionally used to determine this life for components subject to low cycle fatigue, based on a statistical database. Subsequently, during engine service, unanticipated flaws develop which were not included in the data set, or the materials themselves degrade in a manner which was not originally expected, or alternatively additional data is generated and subsequently included in the design data set following service experience which frequently leads to life reductions which can have a dramatic effect on fleet availability. This paper examines several examples where the use of a safe life for determining component replacement intervals could be seen to be inappropriate and often unduly conservative. An alternative method for determining component lives is proposed, involving an assessment of the deterioration of materials during service from all mechanisms.

Measurement and Interpretation of Internal/ Residual Stresses: Neutron and SXRD

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Shaping and Forming Committee
Program Organizers: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Bimal K. Kad, University of California, Ames Laboratory, La Jolla, CA 92093-0085 USA

Wednesday AM Room: 17B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Aaron Krawitz, University of Missouri, Dept. of Mech. & Aeros. Eng., Columbia, MO 65211 USA; Hahn Choo, University of Tennessee, Matls. Sci. & Eng., Knoxville, TN 37996 USA

8:30 AM Invited
X-Ray Microbeam Measurements of Subgrain Stress Distributions in Polycrystalline Materials: *Gene E. Ice*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., One Bethel Valley Rd., Oak Ridge, TN 37831-6118 USA

The behavior of polycrystalline materials depends on many factors, including the grain size/morphology, elastic anisotropy of misoriented crystal grains, non-uniform elastic stress tensor distributions and deformation induced stored energy. We describe the worldís first dedicated polychromatic 3-dimensional (3D) scanning x-ray microscope: the 3D X-ray Crystal Microscope. This new class of x-ray instrument nondestructively measures intra-granular distributions of strain, crystallographic phase, local orientation and deformation with submicron spatial resolution in all three dimensions. The microscope simultaneously collects Laue patterns from every grain along the incident beam. With a beam size smaller than the typical grain size in most materials, only a few grains are simultaneously illuminated and special techniques can be used to disentangle the overlapping Laue patterns. Early results indicate the promise of this emerging class of instrumentation. New initiatives to improve the spatial resolution by an additional order of magnitude and to build a neutron analog are discussed.

9:00 AM Invited

Diffraction Measurement of Load Partitioning in Aluminum-Ceramic Microsphere Syntactic Foams: Dorian K. Balch[1]; *David C. Dunand*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

Liquid aluminum was infiltrated into packed alumina-mullite microspheres, resulting in a syntactic foam consisting of hollow ceramic spheres embedded in a pure aluminum matrix. Load transfer during uniaxial compression was studied by neutron and synchrotron x-ray diffraction. As for composites with solid reinforcement, load transfer was observed between the continuous metallic matrix and the hollow microspheres. The extent of load transfer is modelled and discussed in both the elastic and plastic regimes.

9:30 AM Invited

Differentiating Between Beams and Plates Using Microbeam X-Ray Diffraction: *Cevdet I. Noyan*[1]; [1]IBM, T. J. Watson Lab., Rt. 134, Kitchawan Rd., Yorktown Heights, NY 10598 USA

Pure(4-point) bending of beams and plates are fundamental topics in elementary mechanics. In the case of beams, transverse strains are generally neglected and pure bending results in a uniform stress distribution between the two inner supports. In the case of plates, such an assumption is not strong, and non-uniform stress distributions are theoretically predicted. In addition, anticlastic bending, which requires the plate to assume a ìsaddleî shape, becomes less important. In this study, we present microbeam curvature mapping of the surface of homogeneous Si single crystal strips subjected to pure bending. These measurements show that beams and plates can not be differentiated on the basis of their dimensions alone. We provide experimental proof of the Searle/Ashwell analysis (1908/1952) which should be used for such cases.

10:00 AM

Smarts: A Neutron Spectrometer for Studies of Engineering Materials: *Mark A.M. Bourke*[1]; Ersan Ustundag[3]; David C. Dunand[2]; Bjorn Clausen[1]; Sven Vogel[1]; Thomas Sisneros[1]; [1]Los Alamos National Laboratory, MST, MS H805, Los Alamos, NM 87545 USA; [2]Northwestern University, Dept. Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [3]California Institute of Technology, MC 138-78, Pasadena, CA 91125 USA

In August of 2001 a new neutron scattering instrument called SMARTS (Spectrometer for Materials Research at Temperature and Strain) took first beam. This instrument is the first of a new generation of instruments at pulsed neutron sources dedicated for the study of engineering materials problems. It was designed with two goals in mind i) to make spatially resolved measurements on engineered components and ii) to perform in situ loading studies at both ambient and elevated temperatures. In doing so it can address both macroscopic residual strain measurements and fundamental polycrystalline deformation problems. Funded by DOEís Office of Basic Energy Sciences, SMARTS was built by a development team that included but was not limited to the authors of this paper. Capabilities include 1mm3 spatial resolution, 230KN loading capacity, 1500C furnace and a translator that can manipulate loads up to 1500Kg. This paper will illustrate, by example, the range of problems that have been addressed to date.

10:20 AM Break

10:35 AM Invited

On the Effects of Residual Stresses on Fatigue Behavior in Deep-Rolled Ti-6Al-4V Alloys at Ambient and Elevated Temperatures: I. Altenberger[2]; R. K. Nalla[1]; U. Noster[2]; B. Scholtes[2]; *R. O. Ritchie*[1]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720-1760 USA; [2]University of Gh Kassel, Inst. of Matls. Tech., Kassel 34125 Germany

Mechanical surface treatments, such as deep rolling, shot peening and laser shock peening, can significantly improve the fatigue behavior of highly stressed metallic components commonly used in engineering applications. However, the utility of these treatments for higher temperature service is often brought into question. In the present study, we examine the effect of deep rolling and laser shock peening on the fatigue properties of a wrought Ti-6Al-4V at ambient and 450°C. Near-surface residual stresses were characterized using synchronous x-ray diffraction, and microstructural gradients using transmission electron microscopy, before and after mechanical/thermal exposure. It was found that despite the almost complete relaxation of residual stresses at the higher temperature, there was still a significant benefit for fatigue resistance from the surface treatments. This was attributed to the formation of a stable, near-surface work-hardened layer, with a nano-scale grain size, which acts to diminish the plastic strain amplitude in fatigue, thereby inhibiting both fatigue crack initiation and propagation and hence improving lifetimes.

11:05 AM Invited

The Role of Residual Stress Distributions in Engineering Applications by Synchrotron Probe: *Thomas Tsakalakos*[1]; [1]Rutgers University, Dept. of Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854-8065 USA

Quantitative understanding of the internal stresses field distribution under load is fundamental in the design engineering of static/cyclic load-bearing components. The manufacture/processing or duty cycle of such components can lead a residual stress distribution which can dramatically alter (for good or ill) a components load capacity, and resistance to failure. For example, compressive (tensile) surface stresses tend to retard (accelerate) the surface-initiation and growth of cracks. To compound the problem the stress distribution is extremely difficult to experimentally characterize, offers little or no external evidence of its existence, and is often recognized only a posteriori after failure. In order to make reliable estimates of component performance it is necessary to have an accurate knowledge of these stresses. In this paper, two powerful synchrotron x-ray scattering techniques for residual strain depth-profiling and tomography-like scatter-intensity profiling of materials are presented. The techniques utilize energy dispersive x-ray scattering, from a fixed micro-volume, with micro-scanning of the specimen being used to profile its interior. The tomography-like profiles exploit scattering-cross-section variations, and can be contrast-enhanced by separately monitoring scattering from different crystal structures. The strain profiling technique is shown to finely chronicle the internal strain variation over several mm of steel. Detailed strain profiling for a cantilever spring demonstrates the interplay of residual and external stresses in elastic/plastic deformation. Since surface compression, by shot peening, is a classic method to fortify against fatigue failure, the strain profile for shot-peened, surface-toughened material is determined and discussed in terms of a simple elastic-plastic stress/strain model. The residual stress profiling of fatigue deformation processes on WC/Co and Al2O3/TiO2 nanocoatings showed different internal stress relaxations and internal fatigue damages suggesting different mechanisms. Other residual stress distributions by surface treatment processes such as laser shot peening, low plasticity burnishing, and friction welding will be also reviewed. The correlation of the internal stresses (stress gradients) with both abrasive and sliding wear properties in Nanostructured and microsize-grain coatings will be presented. These results could explain the observed enhancement of wear resistance of nanocoatings vs. conventional. Finally, macro and micro stress distributions of super strength (2.5- 4 GPa) nano steel pearlitic wires will be presented along with modeling and theory. A fresh approach to the fatigue crack retardation by overload and other transient effects will be also presented. Overload effects, underload effects and fatigue crack growth resistance with increasing K are all relatable to the internal stresses arising from the dislocations in the plastic zone. Preliminary experiments have shown that the probing sensitivity of the EDXRD method is sufficient to elucidate the origin of this internal stress Kint by mapping the internal stress tensor in and around the plastic zone of a crack in steels and other alloys. Thus, it is demonstrated that, provided an accurate knowledge of the residual stress profiles generated is available and allowance is made for stress redistribution and the multiaxial nature of residual stresses, reliable predictions of fatigue damage and performance can be made. We gratefully acknowledge the support of the Office of Naval Research under grant N000149910424. Part of this work is also supported by the Defense University Research Initiative on NanoTechnology (DURINT), which is funded by a subcontract to Rutgers University through the Massachusetts Institute of Technology by the Office of Naval Research under Grant.

11:35 AM
Strain and Texture Analysis of Pb(Zr,Ti)O3 Under Compression: *Robert C. Rogan*[1]; Ersan Ustundag[1]; Bjorn Clausen[1]; Mark R. Daymond[2]; Volker Knoblauch[3]; [1]California Institute of Technology, Matls. Sci., 1200 E. California, MC 138-78, Pasadena, CA 91125 USA; [2]Rutherford-Appleton Laboratory, ISIS Neutron Scattering Fac., Chilton, Didcot OX11 0QX UK; [3]Robert Bosch GmbH, Rsrch. 1-Matls., PO Box 10 60 50, Stuttgart D-70049 Germany

In-situ neutron diffraction studies were performed on various piezoelectric Pb(Zr,Ti)O3, PZT, samples under compressive stress. Single phase tetragonal and rhombohedral systems were studied, as well as morphotropic compositions containing both phases. Rietveld analysis of the diffraction patterns allowed for observation of the onset and culmination of domain switching through modeling of the sample texture using the March-Dollase model. Calculated lattice parameters indicated significant residual stresses due to locked in domains, as well as significant strain anisotropy indicative of a complicated internal stress state. The effectiveness of electrical poling and mechanical depoling was evaluated.

Mercury Management: Treatment Technologies

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizer: Larry Twidwell, Montana Tech of the University of Montana, Metallurgical and Materials Engineering, Butte, MT 59701 USA

Wednesday AM Room: 1B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Larry Twidwell, Montana Tech of University of Montana, Metallurgl. & Matls. Eng. Dept., Butte, MT 59701 USA; Corby G. Anderson, Montana Tech of University of Montana, Ctr. for Adv. Minl. & Metallurgl. Procg. (CAMP), Butte, MT 59701 USA

8:30 AM Welcome

8:40 AM Cancelled
Evaluation of Treatment of Bulk Mercury and High Mercury Surrogate Waste: *Mary Cunningham*[1]; [1]US Environmental Protection Agency, Washington, DC USA

9:05 AM Invited
Toward Mercury Management on US Department of Energy Sites: Development and Selection of Technologies: *Michael I. Morris*[1]; Greg A. Hulet[2]; [1]Oak Ridge National Laboratory, Nucl. Sci. & Tech. Div., Oak Ridge, TN 37831-6179 USA; [2]Idaho Nuclear Environmental Engineering Laboratory, Idaho Falls, ID 83415-3875 USA

The US Department of Energy (DOE) TRU and Mixed Waste Focus Area (TMFA) is tasked with finding solutions for the mixed waste treatment problems of the DOE complex. During TMFAís initial technical baseline development process, three of the top four technology deficiencies identified were the need for amalgamation, stabilization, and separation/removal technologies for the treatment of mercury-contaminated mixed waste. The Mercury Working Group (HgWG), a selected group of representatives from DOE sites with significant mercury waste inventories, is assisting TMFA in soliciting, identifying, initiating, and managing efforts to address these areas. Solicitations and contract awards have been made to the private sector to demonstrate both the amalgamation and stabilization processes using actual mixed wastes. Development efforts that will address DOEís needs for separation and removal processes are currently being funded. This paper discusses the technology selection process, development activities, and the accomplishments of TMFA to date through these various activities.

9:30 AM Invited
Mercury Recovery from Chlor-Alkali Wastewater Processes Using REMERC: *Berndt Moeller*[1]; [1]Pioneer Americas, LLC, PO Box 23, St. Gabriel, LA 70776 USA

Mercury-cell chlor-alkali production plants generate mercury contaminated wastewater streams from which mercury must be precipitated and filtered, resulting in a sludge containing 2% mercury (designated K106) that cannot be landfilled in the United States without treatment. REMERC is a chemical treatment process used to recover 99% of the mercury from wastewater sludge generated at these plants. The process uses leaching to oxidize mercury compounds to mercuric chloride, washing and filtration to separate mercuric chloride from the treated sludge, and cementation to reduce the mercuric chloride to metallic mercury. Mercury not recovered in cementation is recycled to the wastewater process for precipitation and filtration. Treated sludge contains less than 260 ppm total mercury and 0.2 ppm TCLP, meeting the USEPA low mercury subcategory specifications that allow landfill disposal. REMERC uses readily available chemicals, is straightforward to control and inexpensive to operate, has no mercury emissions to air, and recovers metallic mercury. REMERC enabled significant cost savings including lower solids disposal costs, recovery and reuse of metallic mercury in the chlor-alkali process, and ability to treat non-wastewater sludge. No new EPA permits were required. REMERC was shown to be a viable process for recovery of metallic mercury from K106 designated sludge.

9:55 AM Break

10:10 AM Invited
Thermodynamic Investigation of the Stability of HgTe with Subsequent Recovery of Mercury from Copper Smelter Acid-Plant Sludge Using a Vacuum Retort Reactor: John P. Hager[1]; *Devon A. Harman*[1]; [1]Colorado School of Mines, Dept. of Metallurgl./Matls. Eng., 1500 Illinois St., Golden, CO 80401 USA

Thermodynamic modeling calculations were undertaken for the recovery of mercury from HgTe with a vacuum retort reactor. Considerable discrepancy was noted in the available thermodynamic data. A series of transpiration experiments were undertaken on the thermal decomposition of HgTe. From the measured Gibbs Free Energy of decomposition, a 2nd law analysis was conducted to yield the Heat of Formation and Entropy at 298K, which provided for the development of a new database for HgTe in HSC Chemistry format. Modeling calculations were then conducted with HSC Chemistry. The minimum temperature for 100% volatilization of the mercury was determined as a function of retort pressure and the molar ratio of flush gas to HgTe. It was found that HgTe is more stable than was indicated from earlier references and that higher temperatures are required for the recovery of the mercury. It was determined that the temperature for 100% volatilization could be decreased from over 1180K to less than 790K by the use of a low residual pressure and the use of a high ratio of flush gas to HgTe.

10:35 AM Invited
Industrially Proven Methods for Mercury Removal from Gases: *Klas G. Hultbom*[1]; [1]Boliden Contech AB, Environml. Tech., PO Box 5097, Helsingborg, Boliden SE-250 05 Sweden

Mercury is present as a trace contaminant in many raw materials from metallurgical plants. When such materials are treated with pyrometallurgical processes the mercury is volatilised. Mercury has a high vapour pressure, even at low temperatures, and therefore it is removed only partly by normal gas cleaning processes. The Boliden Norzink scrubbing process removes elemental mercury by converting it to calomel. It is widely used in the base metals industry. This paper gives examples of the application, and also outlines the background for the development of the process. Another technology developed by Boliden for mercury removal is the Selenium filter. This is a fixed bed filter based on the reaction between elemental gaseous mercury and selenium, by which mercury selenide, HgSe, is formed. The mercury selenide is a highly stable compound with extremely low solubility in water and very low vapour pressure. Mercury is removed selectively without any side reactions. Special applications for the selenium filter technology include mercury removal from gases containing sulphur dioxide and geothermal off-gases.

11:00 AM Invited
Extraction and Recovery of Mercury from Concentrated Sulfuric Acid Streams Using Molecular Recognition Technology: Steven R. Izatt[1]; Neil E. Izatt[1]; Ronald L. Bruening[1]; *John B. Dale*[1]; Petrus J. Cilliers[2]; [1]IBC Advanced Technologies, Inc., 856 E. Utah Valley Dr., American Fork, UT 84003 USA; [2]Molecular Recognition Technology Africa (Pty), Ltd., Park Place E., 104 N. Rand Rd., Hughes, Boksburg 1460 S. Africa

IBC Advanced Technologies, Inc. (IBC) has developed a mercury selective product, which can be used by sulfuric acid producers as either a polisher, or as the primary technology for mercury removal from sulfuric acid. The Molecular Recognition Technology (MRT) separations material selectively binds Hg^{2+}, and to a lesser extent Hg^{+}, out of concentrated sulfuric acid. Mercury output levels of less than 0.1 ppm can be attained by the MRT system. A number of pilot plant tests have been successfully conducted at a number of base metal smelting operations. A large scale commercial plant is due for commissioning in 2003. Results from these projects will be discussed.

11:25 AM Invited
Treatment of Smelter Acid Plant Blow-Down Sludge: Ron Knott[1]; *Roshan Bhappu*[1]; [1]Mountain States R&D International, 13801 E. Benson Hwy., Vail, AZ 85641 USA

This paper describes a novel and proprietary treatment for the recovery of mercury, lead and associated by-products from the acid plant blow-down sludge. The proposed flow sheet includes a hydrometallurgical process for first recovering the contained mercury by an oxygen pre-treatment step in conjunction with the chlorine leach. The mercury is then recovered from the pregnant solution by cementation, first with iron to precipitate copper and then with aluminum powder to precipitate Hg, Au, Ag, etc. The leach residue, after Hg removal, is then leached with NaSH to convert lead oxides and sulfates to synthetic galena which is then floated off into a marketable product. The proposed process appears to be economically attractive and environmentally friendly.

Microstructural Processes in Irradiated Materials: Stainless Steels, Radiation Induced Segregation, Ion Irradiation and Other Materials I

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

Wednesday AM Room: 11A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Maria Jose Caturla, Lawrence Livermore National Laboratory, Chem. & Matls. Sci., Livermore, CA 94550 USA; Rodney Ewing, University of Michigan, Nucl. Eng. & Radiologl. Scis., Ann Arbor, MI 48109 USA

8:30 AM Invited
Radiation Effects in Nuclear Waste Glasses: *Rodney C. Ewing*[1]; Kai Sun[1]; Lumin Wang[1]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., Cooley Bldg., Ann Arbor, MI 48109 USA

Glasses used as nuclear waste forms will suffer radiation damage from the beta decay of fission products and the alpha decay of actinide elements. Since the ultimate radiation damage of a crystalline material is amorphization and glasses are in amorphous state, there is a misconception that glasses are radiation ìresistantî. However, experiments with neutrons, ions and electrons have demonstrated various radiation damages in glasses of various compositions. The typical radiation damage observed in glasses include: swelling associated with bubble formation, secondary phase precipitation due to radiation-induced chemical segregation, and increased leach rates of damage cascades. This talk will summarize our knowledge in radiation effects in glasses and present our most recent experiment results on this subject. In our experiments, the alpha and beta decay events in phosphate and silicate glasses are simulated with electron and ion beam irradiations at various temperatures. Advanced analytical and high resolution transmission electron microscopy (TEM) techniques are used to study the micro-structural-structural and microchemical evolution of the glasses. These techniques include in situ TEM during energetic beam bombardment at various temperatures, electron energy loss spectroscopy (EELS), high angle annular dark field z-contrast scanning TEM (HAADF STEM), energy filtered and fluctuation TEM which provides additional information beyond the first-order pair statistics obtained from diffraction techniques. The results will be compared to radiation effects observed in crystalline ceramics.

9:15 AM
Ion-Irradiation of Electronic Materials: Defects and Microstructures: *Jim S. Williams*[1]; [1]Autralian National University, Canberra, ACT 0020 Australia

After a brief review of ion disordering processes in semiconductors, this presentation focuses on some specific defect and microstructural issues of importance in electronic and optoelectronic applications. Two aspects of silicon irradiation are treated. Firstly, ion irradiation and consequential lattice displacements can lead to a spatial separation of excess interstitials and vacancies. In some cases, annealing of ion implanted silicon can result in the coalescence of such point defects into both extended defects of interstitial character and voids. Secondly, larger voids, or nanocavities, can be produced by hydrogen or helium ion irradiation of silicon and these metastable defects can exhibit some interesting properties, including the trapping of defects and fast diffusing impurities. In addition, the disordering of compound semiconductors is also treated since ion disorder can influence and prohibit the application of ion implantation in these materials for the fabrication of devices. Selected disordering behaviour in GaN and ZnO is examined, particularly the ability of these materials to dynamically anneal displacement damage during irradiation. Some intriguing microstructures (e.g. aligned defect arrays and porous structures) are also observed in these materials. Finally, ion-disordered semiconductors can exhibit unusual deformation behaviour during nanoindentation measurements. Some phase transformation effects in silicon and dramatic structural changes in GaN are also illustrated.

10:00 AM Break

10:30 AM
Microstructure of Alumina Irradiated with Energetic Ions Under Applied Electric Fields: *Toru Higuchi*[1]; Kazuhiro Yasuda[1]; Kuninori Tanaka[1]; Kenichi Shiiyama[1]; Chiken Kinoshita[1]; [1]Kyushu University, Dept. of Appl. Quantum Physics & Nucl. Eng., Hakozaki 6-10-1, Higashi-ku, Fukuoka Japan

Insulating ceramics for fusion reactors are expected to be irradiated with neutrons and ions under electric fields. The electric field influences the kinetics of point defects in those materials. In the present study, we have irradiated alumina with 100 keV He-ions at 760 and 870 K under electric fields of 100 and 300 kV/m to a displacement damage of around 0.5 dpa and observed microstructure changes by using transmission electron microscopy. The electric field suppresses the formation of interstitial-type dislocation loops and enhances their growth in thinner specimens than 300 nm. A higher fraction of interstitials are found to annihirate at the surface under the electric fields, comparing those without the field. The effect of electric fields on the kinetics of point defects is discussed in terms of the interaction between charged defects and electric fields.

10:50 AM
Annealing Stages in Neutron-Irradiated Austenitic Stainless Steels: *Edward Paul Simonen*[1]; Dan J. Edwards[1]; Bruce W. Arey[1]; Stephen M. Bruemmer[1]; Jeremy T. Busby[2]; Gary S. Was[2]; [1]Pacific Northwest National Laboratory, Matls., PO Box 999, MS P8-15, Richland, WA 99352 USA; [2]University of Michigan, Nucl. Eng. & Rad. Scis., 2355 Bonistal Blvd., Ann Arbor, MI 48108 USA

Post-irradiation annealing kinetics in neutron-irradiated austenitic stainless steels exhibit distinct stages in spite of the fact that the detectable microstructure consists dominantly of a distribution of Frank loops and no other significant component of damage. Three distinct annealing stages measured in microstructure and hardness recovery and comparison with model predictions suggest that neutron and proton radiation damage is more complex than is evident in characterization of Frank loop size distributions. Identified stages include a component susceptible to short-term annealing, a component consistent with predicted Frank loop dissolution and a component resistant to longer-term annealing. Specimens of solution-annealed 304SS and 316SS were irradiated at 275C in a light-water reactor to 1 and 5 dpa. Transmission electron microscopy established distributions of Frank loops that extended down to 0.5 nm in diameter. This work was supported by the Materials Sciences Branch, BES, US Department of Energy, under Contract DE-AC06-76RLO 1830.

11:10 AM
Radiation Damage: What Can We Learn from Nanocrystalline Metals: *Max Victoria*[1]; [1]EPFL-CRPP, Fusion Tech. Matls., CH-5232 Villigen-PSI 5232 Switzerland

The volume density of grain boundaries in nanocrystalline (nc) materials is high and defects produced by displacement cascades are expected to be easily absorbed into such sinks. In the present contribution we analyze results from molecular dynamics simulations (MD) in Ni of different grain sizes and for different PKA energies and compare them to experimental observations, in terms of the type of defect produced, their density and the mode of absorption at the grain boundaries. The original microstructure of the nc Ni has been well characterized, particularly in terms of the presence of dislocations, so that the observed radiation hardening will be discussed both in terms of possible dislocation mechanisms or the relaxation at the grain boundaries (A. Hasnaoui, H. Van Swygenhoven and P. Derlet, accepted for publication Acta Mater. (2002)) introduced by the absorbed defects.

11:30 AM Cancelled
Perspective on Experimental and Computer Simulation Studies of Radiation Effects in Ceramics: *William J. Weber*[1]; Fei Gao[1]; L. Rene Corrales[1]; Weilin Jiang[1]; Ram Devanathan[1]; Constantin Meis[2]; Alain Chartier[2]; Yanwen Zhang[3]; [1]Pacific Northwest National Laboratory, Fundl. Sci. Direct., PO Box 999, MS K8-93, Richland, WA 99352 USA; [2]CEA-Saclay, Direction de líEnergie NuclÈaire, DPC/SCPA B,t 450 Sud., Gif-sur-Yvette 91191 France; [3]Uppsala University, Div. of Ion Physics, Angstrom Lab., Uppsala Sweden

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - V

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Alloy Phases Committee
Program Organizers: Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Suzanne E. Mohney, Pennsylvania State University, Department of Materials Science & Engineering, University Park, PA 16802 USA; Michael R. Notis, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18015 USA; Douglas J. Swenson, Michigan Technological University, Department of Materials Science & Engineering, Houghton, MI 49931 USA

Wednesday AM Room: 12
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Michael R. Notis, Lehigh University, Dept. of Matls. Sci. & Eng., Bethlehem, PA 18015 USA; Sinn-Wen Chen, National Tsing-Hua University, Dept. of Cheml. Eng., Hsinchu 300 Taiwan

8:30 AM Invited
Calculated Phase Equilibria for Solder Alloys: *Ursula R. Kattner*[1]; [1]NIST, Metall. Div., Stop 8555, 100 Bureau Dr., Gaithersburg, MD 20899 USA
Phase equilibria information is needed for the evaluation of melting behavior and possible effects of contamination by other elements for candidate solder alloys. Parameterization of phase equilibria data by thermodynamic modeling allows compact storage and retrieval of this information for any composition or temperature of interest. A thermodynamic database for solder alloys has been assembled for 7 components: Sn, Ag, Bi, Cu, In, Pb, Sb. For the construction of this database, available thermodynamic assessments of the constituent binary and ternary systems were reviewed for consistency and, when necessary, individual phase descriptions were revised. One important criterion for the evaluation of these descriptions is the requirement that calculated reaction temperatures need to be known with relatively high accuracy. A summary of the evaluation of available thermodynamic descriptions and results from assembling this database will be discussed.

8:50 AM Invited
Development of Thermodynamic Database on Micro-Solders and Cu Base Alloy Systems: Xing Jun Liu[1]; Ikuo Ohnuma[1]; Cui Ping Wang[1]; Ryosuke Kainuma[1]; *Kiyohito Ishida*[1]; [1]Tohoku University, Matls. Sci., Grad. Sch. of Eng., Aoba-yama 02, Sendai 980-8579 Japan
Recent progress on the database of calculated phase diagrams in micro-solders and Cu-base alloy systems, useful for the development of Pb-free solders and the interfacial phenomena between solder and Cu substrate in electronics packaging technology, is presented. A thermodynamic tool, ADAMIS (Alloy Database for Micro-Solders), is based on comprehensive experimental and thermodynamic data accumulated with the CALPHAD (Calculation of Phase Diagrams) method and contains 8 elements of Ag, Bi, Cu, In, Sb, Sn, Zn and Pb. It can handle all combinations of these elements and all composition ranges. The element of Al is also added to ADAMIS within a limited range of compositions. Furthermore, the database of Cu-base alloys including the Cu-X binary system and Cu-Fe, Cu-Ni, Cu-Cr base ternary systems, has also been developed. Typical examples of the calculation and application of these databases will be presented.

9:10 AM Invited
Assessment of Phase Equilibria in Ag-Cu-Sn Near Eutectic System: *Choong-Un Kim*[1]; Jaeyong Park[1]; Ted Carper[2]; Puligandla Viswam[2]; [1]The University of Texas at Arlington, Matls. Sci. & Eng., Woolf Hall, #325C, Arlington, TX 76019-0031 USA; [2]Nokia Mobile Phones, Rsrch. & Tech. Access, 6000 Connection Dr., Irving, TX 75039 USA
Accurate assessment of phase equilibria is one of the most critical tasks in developing and applying new engineering alloys. Phase equlibria of the Ag-Cu-Sn (ACS) alloy system, a potential replacement for Pb-Sn solder alloys, however, is not well known, especially in near-eutectic compositions. The difficulty in assessing phase equilibria of the near-eutectic ACS system stems from the fact that multiple phase fields exist within a very narrow range of composition and temperature, making them difficult to resolve by ordinary experimental techniques. This study presents the near-eutectic phase equilibria of the ACS system as determined by a selective cooling method in conjunction with theoretical calculation. Selective cooling induces growth of the equilibrium solid phase at a given temperature and forces the remaining liquid phase solidify into a finer microstructure by rapid cooling for better identification of phase equilibria.

9:30 AM
Soldering-Induced Cu Diffusion and Intermetallic Compound Formation Between Ni/Cu UBM and SnPb Flip Chip Solder Bumps: *Chien Sheng Huang*[1]; Jenq Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 101 Sec., 2 Kuang Fu Rd., Hsinchu 300 Taiwan
Ni-based under bump metallization has been widely used as a diffusion barrier to prevent the rapid reaction between Cu conductors and Sn-based solders. In this study, electroplated Ni with different thickness was employed to evaluate the interfacial reaction after reflow, annealing and aging between Ni/Cu UBM and eutectic Sn-Pb solders in the 63Sn-37Pb/Ni/Cu/Ti/Si3N4/Si multiplayer structure. In the as-reflowed sample, an 1-mm (Ni1-x,Cux)3Sn4 IMC was found between solders and Ni/Cu UBM. During multiple reflow or annealing at 240∞C for a period of time, another type of IMC with the form (Cu1-y,Niy)6Sn5 was found between solders and (Ni1-x,Cux)3Sn4, whereas the thickness of (Ni1-x,Cux)3Sn4 was still around 1 mm. However, after aging at 150∞C for 1000 hours, the thickness of (Ni1-x,Cux)3Sn4 IMC increased to 3mm, yet no (Cu1-y,Niy)6Sn5 was revealed. It is argued that formation of (Cu1-y,Niy)6Sn5 IMC was due to the Cu diffusion through (Ni1-x,Cux)3Sn4 IMC into solders along with the further reaction with Ni and Sn. The degrees of Cu diffusion could be correlated to the heat treatment conditions and morphological evolution of (Ni1-x,Cux)3Sn4 IMC.

9:45 AM
Computational Model for Multi-Component Base Metal Dissolution and Intermetallic Compound Formation with Molten Solder Alloys: *Kenneth L. Erickson*[1]; Polly L. Hopkins[1]; Paul T. Vianco[1]; Jerome A. Rejent[1]; [1]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA
Results from experiments examining multi-component base metal dissolution by molten solder alloys were reported previously. Preferential dissolution of base metal constituents appeared to influence short-term base metal erosion and long-term inter-metallic compound (IMC) growth. Experiments with 76Au-21Pt-3Pd (wt%) alloy sheet and molten 63Sn-37Pb solder indicated that preferential dissolution of Au produced Pt-rich IMC layers that could limit base metal erosion and cause induction periods observed during subsequent IMC growth. To examine these issues quantitatively and develop predictive tools for process and long-term reliability analyses, a general model describing multi-component dissolution and IMC growth was developed based on solid-solid and liquid-solid reactions coupled with solid-phase and liquid-phase multi-component diffusion. The resulting equations now require phase-boundary conditions involving time-dependent concentration and flux discontinuities, rather than the constant-concentration ìjumpsî used with binary systems. This paper summarizes the multi-component model, its implementation in previously developed codes, and comparisons between calculated and experimental results.

10:00 AM Break

10:25 AM Invited
Directional Solidification of AgCuSn Eutectic Alloys: *Daniel Joseph Lewis*[1]; [1]NIST, Metall. Div., 100 Bureau Dr., Stop 8555, Gaithersburg, MD 20899 USA
Directional solidification has been used to examine microstructures in AgCuSn, CuSn, and AgSn eutectic alloys. The eutectic alloy in this system is of industrial importance as a replacement solder alloy for tin-lead. Using directional solidification it is possible to study solidifi-

cation rates relevant to soldering processes as well as solidification rates on the order of one micrometer per second. Microstructures collected over a range of solidification rates will help to understand the presence of a large volume fraction of tin dendrites seen in industrially assembled solder joints as well as provide information on the solidification velocities where three phase coupled growth is possible. Microstructures and quantitative image analysis will be presented for the three alloys considered.

10:45 AM Invited

Fluxless Plasma Soldering of Pb-Free Solders on Si-Wafer: *Jae-Pil Jung*[1]; Joon-Kwon Moon[1]; Soon-Min Hong[2]; [1]University of Seoul, Dept. of Matls. Sci. & Eng., 90 Cheonnong-dong, Dongdaemun-gu, Seoul 130-743 Korea; [2]Samsung Electronics Company, Ltd., Micro-Joining Lab., Inst. of Intelligent Sys., Mechatronics Ctr., 416 Maetan-3dong, Paldal-gu, Suwon City, Kyungki-do 442-742 Korea

In the field of electronic industry, a soldering flux is inevitable to get a reliable solder joint. However, with growing concerns about environment, the flux is avoided occasionally for environmental or technical problems. Flux residue on the fine-pitch device cannot be removed easily and may cause corrosion on the joint. For this reason many researchers have interests in fluxless soldering process. On this study fluxless solderability of Sn-3.5Ag, Sn-3.5Ag-0.7Cu and Sn-37Pb alloys were investigated. The solder balls were set on a UBM-coated Si-wafer to achieve fluxless reflow soldering in plasma. Ar-10%H2 plasma was used for the experiment. In the fluxless plasma reflow, the self-alignment of solder bumps and intermetallic compound on the interface were observed.

11:05 AM

Electromigration of Sn95/Sb5 Flip Chip Solder Bumps on Cr/Cr-Cu/Cu Under Bump Metallization: *T. L. Shao*[1]; Chih Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Eng., 1001 Ta Hsueh Rd., Hsinchu 30050 Taiwan

Increasing awareness of environmental and health impact of electronic materials has highly demanded the lead-free product of IC package recently. Lead-free solder of Sn95/Sb5 with low α particle property was used to avoid the soft error of memory IC flip chip package. The electromigration behavior of Sn95/Sb5 flip chip solder bumps was studied. The under bump metallization (UBM) on the chip side was Cr/Cr-Cu/Cu, and the metallurgy layer on the substrate side was Ni/Au. The chip was mounted on a BT substrate and then filled with underfill. The flip chip package was stressed at the current density of 2 * 10E4 A/cm^2 at 100°C. The current crowding effect and the stressing polarity effect were examined. Void formation on the cathode side was observed. The failure of the rerouting and thin film structure on chip side were examined.

11:20 AM

Relationship Between Mechanical Properties and Intermetallic Compound Formation at the Sn-0.7(0.5, 0.3, 0.1)wt%Cu/Ni Joints: *Sinn-Wen Chen*[1]; Shou-Wei Lee[1]; Chao-Hong Wang[1]; Ming-Chuen Yip[2]; [1]National Tsing Hua University, Dept. of Cheml. Eng., #101 Sec., 2 Kuang-Fu Rd., Hsin-Chu 300 Taiwan; [2]National Tsing Hua University, Dept. of Power Mechl. Eng., Hsin-Chu 300 Taiwan

Sn-0.7wt%Cu is one of the most promising Pb-free solders. However, the compound formation at the solder/Ni joint is very sensitive to the Cu amounts in the solders. This study determined intermetallic compound formation in the Sn-0.7(0.5, 0.3, 0.1)wt%Cu/Ni joints at 200°C and 250°C and also the tensile properties of these joints. At 250°C, only Ni3Sn4 phase was found in the Sn/Ni, Sn-0.1wt%Cu/Ni, Sn-0.3wt%Cu/Ni couples; while Cu6Sn5 was found in the Sn-0.5wt%Cu/Ni and Sn-0.7wt%Cu/Ni couples. The critical compositions for the Cu6Sn5 formation were determined to be 0.4wt%Cu and 0.1wt%Cu at 250°C and 200°C, respectively. Both Ni3Sn4 and Cu6Sn5 phases were found in the solid/solid couples (200°C), while the Ni3Sn4 was hardly detected in the solid/liquid couples (250°C) when the Cu6Sn5 existed. No significant differences of tensile properties were observed for these Sn-Cu/Ni joints made with solders of various Cu amounts, even if the intermetallic compounds formed at the interfaces were different.

11:35 AM

Interfacial Segregation of Bismuth in Pb-Free Solder Interconnects on Copper Metallization: P. L. Liu[1]; C. Z. Liu[2]; A. P. Xian[2]; *J. K. Shang*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA; [2]Chinese Academy of Sciences, Inst. of Metals Rsrch., Shenyang Natl. Lab. for Matls. Sci., 72 Wenhua Rd., Shenyang 110016 China

Bismuth is used as a major alloying element in Pb-free solder alloys because it stabilizes tetragonal tin and reduces the melting temperature of tin-base alloys. In this study, microchemistry of the interface in a tin-bismuth solder interconnect was examined by scanning Auger microscope equipped with an in-situ fracture stage. Segregation of bismuth was found on the interface between copper and copper-tin intermetallic phase when a copper/tin-bismuth solder interconnect was thermally aged. The segregation was confined to about a couple of atomic layers from the interface, resulting in embrittlement of the interface. Aging conditions for Bi-induced interfacial embrittlement were systematically determined.

Products, Applications, and Services Showcase: Furnaces & Casting Technology

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: David V. Neff, Metaullics Systems Company, Solon, OH 44139 USA

Wednesday AM — Room: 5A
March 5, 2003 — Location: San Diego Convention Center

Session Chair: TBA

8:30 AM

Furnaces for the New Business Trends: *Jarda Urbanek*[1]; Dirk Menzler[2]; [1]Junker, Inc., 2015B Rte. 34, Oswego, IL 60543 USA; [2]Otto Junker GmbH, PO Box 1180, Simmerath 52417 Germany

Brief intoduction of our company and services provided,followed by an overview of several heat treating furnace designs for various semifinished products. Furnace design emphasis is on varying lot size and product dimensions for either the strips, plates, coils, ingots or billets. Design features to support this business trends will be pointed out. Presentation of typical cycle times, efficiencies and temperature tolerances from actual process data for various products will be discussed.

8:50 AM

Heat Treating and Thermal Processing for the Aluminum Industry: *Thomas Zamanski*[1]; [1]LOI, Inc., 333 Technology Dr. S. 109, Canonsburg, PA 15317 USA

Today, LOI, Inc. continues to be a leader in heat-treating and thermal processing equipment with a large selection of equipment designs. Long recognized in the Steel Industry, LOI is making its presence felt in the Aluminum Industry as well, with its melting and heat-treating technology. Currently, the company is marketing technology in the following areas: -A Solution Anneal and Age Hardening Line for Aluminum wheels -Single coil Annealing Furnaces for Aluminum Strip or Foil -Twin Chamber Melting Furnace for Recycling Aluminum Scrap. LOI is able to perform projects that are on the cutting edge of technology because of the research and development team that is supported by the parent of the LOI Group, LOI Thermprocess. This relationship has allowed LOI, Inc. to remain technically strong enough to meet the demands of the North American market, yet flexible enough to handle both large and small projects.

9:10 AM

Recent Technological Advancements in Electro-Magnetic Pumping Solutions for the Aluminium Industry: *Alan Peel*[1]; [1]EMP Technologies Ltd., Beedes House Easton Ave., Stretton Burton-on-Trent, Staffordshire VE13 OVB UK

The presentation will discuss new enhancements to the EMP System particularly focusing on the new generation of vortex system based on the LOTUSS principles for ultra light gauge scrap submergence at very high charging rates in excess of 20,000 lbs/hour. Results of the improvements gained by clients following installation of the new vortex will be discussed. Combining metal transfer and de-gassing as an integral part of the circulation and charging system within the EMP System will also be discussed. In addition new developments based around the electro-magnetic pump for different applications from small die casting operations with furnace capacities less than 20,000 lbs allowing these operations to recycle in house their own scrap arisings and carry out their own efficient alloying.

9:30 AM

Advanced Monolithic Refractories for Lining Aluminium Melting and Holding Furnaces: *Duncan Jones*[1]; [1]Thermal Ceramics, Tembay Rd., Bromborough, Wirral CH62 3PH UK

Monolithic refractories are now becoming more widely used in aluminium melting and holding furnaces. In particular, specialist high strength castables with non wetting additives have been developed which are now replacing traditional brick linings in these furnaces. Thermal Ceramics, have focused their efforts on developing a range of specialist monolithic refractories to meet the necessary demands placed on refractory linings in modern aluminium furnaces. This paper reviews the various Tri-Mor monolithic refractories available for a complete aluminium furnace lining and in particular details the properties and application of some aluminium contact castables. These materials have been designed to be non-wetted by aluminium and resistant to corundum growth even under extreme furnace operating conditions. Additionally a range of maintenance materials suitable for both hot and cold repairs is discussed.

9:50 AM
High Temperature Alternatives to Refractory Ceramic Fiber (RCF): *Lance J. Caspersen*[1]; [1]Thermal Ceramics, 2102 Old Savannah Rd., Augusta, GA 30906 USA

In recent years there have been some discussion about the health and safety aspects of refractory ceramic fiber or RCF, particularly among aluminum companies. Although Thermal Ceramics, the largest producer of RCF material in the world, does not take the stance that these products are harmful if handled properly, we recognized the desire by some companies for alternative materials. Through a research project that began in the early 80ís, our company introduced a high temperature, non-RCF insulating product called Superwool 607 with low biopersistance, or capability to persist in the body. Thermal Ceramics has since developed many other innovations in product chemistries, capabilities and forms in these class of amorphous wool materials. This presentation gives a broad overview of the health and safety matters related to RCF and Superwool products, the various high temperature Superwool products available and the many applications that theyíve been used for.

10:10 AM
The Real Cost of Alloying in the Casthouse: *Edgar C. Burhop*[1]; Paul Cooper[2]; [1]Metallurg Aluminium, PO Box 768, Newfield, NJ 08344 USA; [2]Metallurg Aluminium, Fullerton Rd., Rotherham, S. Yorkshire S60 1DL UK

The cost of alloying in the casthouse is generally poorly defined, and the process of alloying is not always given the consideration that it deserves. Too much focus is usually placed on the price of an alloying additive, and not enough on other factors that affect the true cost of alloying. This paper presents these issues and gives an insight into the dissolution processes occurring in the furnace, and problems associated with different addition methods. A review is made of why poor elemental recoveries are sometimes observed. Recommendations for best alloying practice are also provided. The report concludes that the real cost of alloying is very complicated and not readily defined. Decisions on which alloying method to adopt, therefore, require studied consideration, and must be based on a close study and understanding of the melting and casting operations and the alloying methods available.

10:30 AM Break

10:50 AM
Recent Developments in the Application of Brochotís Casting Wheel in Ingot Production: *Jean-Jacques Grunspan*[1]; [1]Brochot SA, 52 Ave. Marcel Paul, Tremblay en France, Cedex 93297 France

Tomago has retro-fitted Brochotís advanced casting wheel to achieve both increased throughput and high quality, dross free ingots. The wheel eliminates the oxidation that causes dross by reducing turbulence at all critical points. This means that dross is not just hidden, but really absent. To meet Tomagoís desire of increasing capacity, the wheel has been slightly redesigned to ensure operation at 27 tonnes per hour. Other advantages include constant casting level in the moulds and the automatic lifting of both wheel and launder to avoid spillage of molten metal in the event of a stoppage downstream or an unstripped ingot. Other development already achieved in ingot production will also be detailed.

11:10 AM
The Manufacturing, Use and Plant Test Results of TF Combo Bags for DC Sheet Ingot Casting: *Sylvain Tremblay*[1]; M. Ruel[2]; [1]Pyrotek High Temperature Industrial Products, Inc., 1623 Manic St., Chicoutimi, Quebec G7K 1G8 Canada; [2]Pyrotek High-Temperature Industrial Products, Inc., 2400 Blvd. Lemire, Drummondville, Quebec J2B 6X9 Canada

In the last decade, the molten aluminum distribution in the ingot heat of dc sheet casting ingots has been achieved using mostly a combo bag made of fiberglass fabrics. Some of these fabrics are open-weave materials while others are solid fiberglass fabrics sewn together. This bag is usually soft and flexible and extensive sewing operations are required to manufacture it. Usually the bag deforms and this can affect not only the distribution but also the molten metal temperature profile around the mold and at the end, the final ingot quality. This paper will review the production and use of a new combo bag for dc sheet ingot casting. The paper is divided in two parts. The first part will deal with the manufacturing of the TF combo bag (acronym for thermal formed). Description of the fabrics used as well as the new production techniques to manufacture that bag will complete this first part. Part two (2) will present the test results of the TF combo bag used at different plants to cast AA-1045, AA-3003 and AA-5052 alloys. The following variables and measures will be discussed: Cast start-up, actual metal flow observations, ingot surface finish, temperature profile around the mold, and finally, results from the rolling plant. Comments will also be presented from an operation point of view using this new molten aluminum distributor.

11:30 AM
Conform TechnologyñDevelopments in Feedstock Materials: *Phillip M. Thomas*[1]; [1]Holton Conform, Business Dvlp. & Tech., Albany House Elliott Rd., W. Howe, Bournemouth, Dorset BH11 8JH UK

The Conform process is an established technology for the production of a wide range of end products from a range of non ferrous materials. The feedstock material has traditionally been rod which has been continuously cast and rolled although for some products - notably grain refining rod - the feedstock has been continuously cast bar. Over the past few years the range of alloys that can be processed has been extended and 2xxx and 7xxx alloys have been successfully processed as well as a number of composite materials. The type of feedstock materials has also been increased and now includes powders, granules and a variety of other particulate materials. The presentation will describe these developments by means of a series of case studies.

11:50 AM
Wagstaff Direct Chill Casting Update: *Craig Johnson*[1]; [1]Wagstaff, Inc., 3910 N. Flora Rd., Spokane, WA 99216 USA

Wagstaff will provide an update on the a latest developments on our ingot casting technology, billet casting technology, and automated casting controls.

12:10 PM
Advanced Welding Techniques for Aluminum Alloys: *Menachem Kimchi*[1]; [1]Edison Welding Institute, Resistance & Solid-State Welding, 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

This paper describes two new joining techniques for joining aluminum. One technique, called magnetic pulse welding (MPW), is applicable to joining of aluminum sheet. These processes both offer numerous advantages over conventional arc welding processes especially in automotive and aerospace applications. MPW is a very high-speed process which produces solid-state (cold) welds with minimal distortion and degradation to mechanical properties, offers potential for significant manufacturing cost reduction through increased weld speed, and is very good for welding dissimilar metal combinations (i.e., aluminum to steel). Conductive heat seam welding (CHRSEW) is a resistance welding process that offers the potential to reduce welding cost through increased welding speed, reduced joint preparation and consumable cost, the ability to successfully weld difficult-to-weld aluminum alloys such as the 7xxx series alloys, the ability to join dissimilar sheet thickness, and to produce good mechanical properties compared to arc welding. This paper will highlight process details and variables, mechanical properties, and potential applications for both welding techniques.

Recycling - General Sessions: Aluminum

Sponsored by: Light Metals Division, Jt. LMD/EPD-Recycling Committee
Program Organizers: Han Spoel, Spalco Metals Inc., Toronto, Ontario M5R 1W8 Canada; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday AM — Room: 3
March 5, 2003 — Location: San Diego Convention Center

Session Chair: Han Spoel, Spalco Metals Inc., Toronto, Ontario M5R 1W8 Canada

8:30 AM Announcements

8:45 AM
Determining Recyclability, A Working Method: *W. Bryan Steverson*[1]; Donald L. Stewart[2]; [1]Alcoa Inc, 2300 N. Wright Rd., N290, Knoxville, TN 37701-3141 USA; [2]Alcoa Inc., Casting Tech. Div., Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

How do you determine recyclability? When a customer approaches and asks if their change, addition, coating modification, etc. affects the recyclability of your product, how do you answer? How are concerns about safety, hygiene, hazardous contaminants, melt loss, cost, metal quality, and operational issues reviewed and answered? How are these ongoing questions answered in a thorough manner which both protects the recycler while providing the supplier the necessary flexibility to grow and improve their product. The purpose of this paper is to identify the concerns and discuss one current method in place within a UBC recycling environment.

9:15 AM
The Secondary Metal Supplier and Foundry Metal Quality: *Ray D. Peterson*[1]; [1]IMCO Recycling, Inc., 397 Black Hollow Rd., Rockwood, TN 37854-4021 USA

Foundry customers can purchase ingot or molten metal from many metal supplier sources. The primary concern of the customer should be receiving an alloy with a composition within the specified limits. If the chemical limits are correct, the source of the elements should not matter. Overly tight compositional specifications can lead to higher material costs. Once compositional concerns are met, other customer concerns should include hydrogen and alkali metals concentrations, and inclusions (both the type of inclusion and the quantity). Foundry metal from secondary metal sources can be equivalent to or superior to primary metal sources on the basis of both chemistry and impurities. The melting equipment used for processing the secondary metal sources can have a significant impact on the metal quality. Care must also be taken at the customerís plant not to degrade metal quality once it is received. Every plant producing castings should practice proper handling and treatment of molten metal to ensure quality and reliability.

9:45 AM
Controlled Melting of Secondary Aluminum in Rotary Furnaces: *Henrik Gripenberg*[1]; [1]Linde Gas Division, Lidingˆ S-181 81 Sweden

Secondary plants strive to optimize the cost of scrap raw materials. However lower cost scraps contain more organic contaminations like lacquer and oils. These cause emissions that have a negative influence on productivity and environmental conditions thus limiting the amount of organic components that can be allowed in the charge. By using controlled melting and optimized hardware solutions the negative impact of organic contaminations can be reduced and the economy of the melting is improved. The new WASTOX process involves controlled oxygen lancing and burner management for combustion of the emissions inside the rotary furnace. The composition of the flue-gas is analyzed using a new laser technology concept and is together with other parameters used for on-line process control. The WASTOX process was installed at the new Universal Rotary Tiltable Furnace (URTF) plant at Stena Aluminium AB. The paper will discuss theory, hardware solutions and industrial results from the WASTOX concept and the URTF plant.

10:15 AM Break

10:30 AM
Gases Evolved During Decoating of Aluminium Scrap in Inert and Oxidizing Atmospheres: *Anne Kvithyld*[1]; Thorvald Abel Engh[1]; Piotr Kowalewski[3]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., Trondheim N-7491 Norway; [3]Warsaw University of Technology, The Fac. of Matls. Sci. & Eng., Warsaw 00-661 Poland

Thermal removal of coatings on aluminium scrap in a delacquering unit prior to melting makes it possible to lower the emissions and utilize the energy released by the combustion process. Polyester coated on aluminium was studied using both inert and oxidizing atmospheres. The evolved gases were registered using quadrupole mass spectroscopy (MS) during the coat decomposition in a thermogravimetric/differential thermal analysis (TG/DTA) furnace. Ion gas intensity scans collected during the main weight losses are presented, together with the enthalpy changes. The losses, degradation products and enthalpy produced depend both upon the atmospheres and the heating rates. The data have been used to develop kinetic models of pyrolysis and combustion of the coating. These can be used to choose suitable atmosphere and temperature profiles in industrial decoating units so as to reduce and control emissions.

11:00 AM
A Study on Titanium Hydride Formation of Used Titanium Scrap: *Bo Young Hur*[1]; *Duck Kyu Ahn*[1]; *Sang Youl Kim*[1]; *Hiroshi Arai*[1]; *Soo Han Park*[1]; [1]Gyeongsang National University, ULSFoM-NRL, ReCAPT, Div. of Matl. Eng., 900, Kajoa-Dong, Chinju, Kyungnam 660-701 S. Korea

Metallic hydride is often used as foaming agent during fabrication of porous metal. Most of the titanium hydride made from sponge Ti, but in this study, It made from scrap Ti to reduce the cost. Scrap Ti with various shapes such as plate, chip, and bulk etc. was hydride in self-made apparatus and compared with the standard sample made from sponge Ti. The temperature of hydrogenation was about 400° in the case of pure sponge titanium, but return scrap titanium alloy were increased. The hydrides of titanium alloy were crushed by ball mill for 3 hours. It was determined by heating and cooling curve in reaction chamber. The result of XRD was titanium hydride peak only that it was similar to pure titanium.

11:30 AM
In Situ, Real Time Measurment of Aluminum Melt Chemistry: *Robert De Saro*[1]; Arel Weisberg[1]; Joseph Craparo[1]; [1]Energy Research Company, 2571-A Arthur Kill Rd., Staten Island, NY 10309 USA

Energy Research Company is developing an instrument to measure the chemistry of molten aluminum. Termed LIBS, for Laser Induced Breakdown Spectroscopy, the probe can measure in situ and in real time the elemental concentrations of aluminum. The laser, operating in the visible spectrum, is repetitively fired through a fiber optic cable, which is placed in the melt via a proprietary probe. A small amount of melt absorbs the laser light and is rapidly ionized. The returning light is resolved by a spectrometer, which uniquely identifies the elements in the melt. The light amplitude determines the concentration. Laboratory experimental results have confirmed that the concept works. Full-scale field tests at commercially operating plants will begin this year. This work was funded, in part, by the Department of Energyís Office of Industrial Technology.

Science and Technology of Magnetic and Electronic Nanostructures: Nanostructures in Functional Materials: Magnetic

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Ramamoorthy Ramesh, University of Maryland, Department of Materials and Nuclear Engineering, College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Wednesday AM Room: 15A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Robert D. Shull, NIST, Magnetic Matls., Gaithersburg, MD 20899-0001 USA; Xiaoqing Pan, University of Michigan, Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109 USA

8:30 AM
Multi Component Functional Nanostructures: Fabrication and Local Properties: *Dawn A. Bonnell*[1]; [1]University of Pennsylvania, Matls. Sci., 3231 Walnut St., Philadelphia, PA 19104 USA

The potential to assemble dissimilar molecular or nanostructural elements into structures with complex functionality has motivated considerable activity in several scientific disciplines. The library of new molecules with electrical, optical or chemical activity grows quickly and now includes synthetic polypeptides, conjugated organic molecules, nanotubes, metallic and semiconducting nanowires, etc. However, functionality has been achieved in only a few select systems. In order to realize the potential inherent in nanodevices, methods of assembling a wide range of dissimilar elements and connecting the resulting complex structures must be developed. Furthermore, the processes must be informed by a knowledge of the properties of individual components. We report here a novel approach that utilizes local reactivity of ferroelectric surfaces due to variations in atomic polarization. It will be demonstrated that chemical reactivity is domain specific based on control of local electronic structure. When combined with chemical assembly nanostructures consisting of oxide substrates, metal nanoparticles, and organic/biological molecules can be fabricated. The procedure can be iterated to develop structures in which multiple types of metal particles, and electronically or optically active molecules can be assembled in predetermined configurations. New techniques for quantifying the local properties of nanostructures and even individual defects will be illustrated.

9:15 AM

Future Data Storage and Nanoscience: *Joachim Walter Ahner*[1]; [1]Seagate Technology, 1251 Waterfront Pl., Pittsburgh, PA 15222 USA

It is well appreciated, that as the size of material objects approaches nanometer dimensions, the materials structural and electronic properties change. This is related to a number of factors including quantum size effects and the enhancement of the surface/volume ratio with diminishing size. The investigation of these effects and the impact in future recording media will become crucial in the next future, when recording approaches Terabit/sq.inch densities. I will report about the development of two novel probing instruments for imaging, manipulation and analyzing nanometer scaled materials. I will propose concepts of applications for future data storage devices and present first results on ferromagnetic and ferroelectric recording media.

10:00 AM

Magnetic Properties of Lithographically Defined Nanomagnets: *Fernando J. Castano*[1]; Yaowu Hao[1]; Caroline A. Ross[1]; Bernhard Vogeli[2]; Henry I. Smith[2]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Cambridge, MA 02139 USA; [2]Massachusetts Institute of Technology, Dept. of Electl. Eng. & Compu. Sci., 77 Massachusetts Ave., Cambridge, MA 02139 USA

The present work reviews progress in the fabrication and characterization of sub-100 nm nanomagnets made using lithography techniques, assessing the possibility of integrating these small structures in magneto-electronic devices. The starting materials are evaporated or sputtered multilayer structures and arrays of nanomagnets, with a wide variety of different shapes, are defined using either subtractive or additive fabrication approaches. The magnetization reversal in compositionally modulated rectangular and ring shaped elements will be discussed. Issues such as the element variability, as well as the interaction among nanomagnets will be addressed. Furthermore, the magnetoresistance exhibited by these small magnets will be examined as a function of their shape.

10:45 AM

Magnetism, Structure and the Characterization of Antisite Disorder in the Predicted Half-Metals Co_2MnSi and Co_2MnGe: *J. A. Christodoulides*[1]; M. P. Raphael[1]; B. Ravel[1]; S. F. Cheng[1]; B. N. Das[1]; Q. Huang[2]; R. Ramesh[3]; G. A. Prinz[1]; V. G. Harris[1]; [1]Naval Research Laboratory, Washington, DC 20375 USA; [2]National Institute for Standards and Technology, Ctr. for Neutron Rsrch., Gaithersburg, MD 20899 USA; [3]University of Maryland, College Park, MD 20742 USA

Magnetoelectronic devices rely upon an imbalance in the number of majority and minority carriers to add additional degrees of freedom to traditional logic devices. The ideal magnetoelectronic device consists of ferromagnetic materials that exhibit complete spin polarization at the Fermi surface (i.e. half-metallic ferromagnets). One promising class of materials is the Heusler alloys, some of which have been predicted by first principles band theory to be half-metallic. Here we report on the magnetic, structural and electrical transport properties of Co_2MnSi and Co_2MnGe. We have studied single crystals, poly-crystalline arc-melted buttons and thin films grown by d.c. and r.f. magnetron sputter deposition. Samples have been characterized for antisite disorder by neutron diffraction and anomalous x-ray diffraction techniques. For Co_2MnSi, the thin films and arc-melted buttons have lattice constants, magnetic properties, and room temperature resistivities that approach those of the single crystal. On the other hand, the residual resistivity ratio, ρ_{300K} / ρ_{5K}, of these samples shows sharp contrasts with the single crystal; the crystal, the arc-melted button and the best thin films having ratios of 6.5, 2.7 and 1.4, respectively. Disorder studies on thin film and arc-melted samples show that the antisite disorder is typically zero for Mn-Si antisite disorder, but between 10% and 14% for Co-Mn antisite disorder. We postulate this to be the cause for the increased amount of defect-induced scattering in the electrical transport measurements of these samples. The results of a similar study on Co_2MnGe will also be presented.

Surface Engineering in Materials Science - II: Manufacturing via Surface Engineering

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Surface Engineering Committee

Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA; A. Agarwal, Plasma Processes, Inc., Huntsville, AL 25811-1558 USA; Narendra B. Dahotre, University of Tennessee-Knoxville, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; C. Suryanarayana, University of Central Florida, Mechanical, Materials & Aerospace Engineering, Orlando, FL 32816 USA

Wednesday AM — Room: 7A
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: John J. Moore, Colorado School of Mines, Dept. of Matls. Sci. & Eng., Golden, CO 80401 USA; R. Mishra, University of Missouri-Rolla, MSE, Rolla, MO 65409-0340 USA

8:30 AM Invited

Laser Surface Modification of Aluminum for Automobile Application: *J. Mazumder*[1]; J. Caroll[1]; Y. Lu[1]; J. Kelly[1]; [1]University of Michigan, Dept. of Mechl. Eng., & Matls. Sci. & Eng., 2350 Hayward St., Ann Arbor, MI 48109-2125 USA

There is a considerable interest in using Aluminum in automobile for reducing the weight of a car for improved fuel efficiency. This paper reports various laser surface modification applications for possible use in various engine and power train components. Laser Cladded Mn-Al Bronze coatings on cast alloy AA 333 were successfully produced using a 6kw continuous wave CO2 laser with Oscillating Beam. The coatings were dense, well bonded, free of cracks and relatively much harder with sufficient toughness. Copper based alloys can be used to improve resistance for pump and valve seats for Aluminum engine heads. Laser surface alloying of Iron on Aluminum cast alloys were also investigated for improved wear properties within the Aluminum engine bore. Preliminary evaluation of the selected coatings microstructures was carried out using optical and scanning microscopy, x-ray energy dispersive spectroscopy and x-ray diffractometry. It revealed columnar type grains of varying sizes, homogenous distribution of the alloying elements and FCC Cu-based solid solution with enhanced lattice parameters, depending on the processing condition. The coating/substrate interface microstructure was found to contain a-Al (FCC) and tetragonal Cu-Al2. The dry sliding tribological tests revealed encouraging results of 1/3 of the wear rate for coatings compared to the cast aluminum AA333 substrate. The coating was much less prone to seizure under identical test conditions of 10 lbs normal load at 600 rpm and 30.3 mm radius of sliding. Significant improvements of thermal diffusivity were reported for valve seats cladding compared to inserts. For Al-Fe alloys predominant phase were a-Aluminum solid solution with a range of distribution of Al-Fe-Si intermetallics. Improved wear resistance was also observed for this alloy.

9:00 AM

Formation of Metal-Ceramic Layer on Aluminiun Alloy (Al-12Si) by Laser Processing: *Dillibabu Sastikumar*[1]; M. Jamal Mohamed Jaffar[2]; R. Jagdheesh[1]; A. K. Nath[3]; [1]Regional Engineering College, Dept. of Physics, Tiruchirappalli, Tamil Nadu 620 015 India; [2]Jamal Mohamed College, Dept. of Physics, Tirichirappalli, Tamil Nadu 620 020 India; [3]Center for Advanced Technology, Industl. CO2 Laser Sect., Indore 452 013 India

The hardness and wear resistance of various engineering materials have been reported to be greatly enhanced by laser surface treatment with different metal-ceramic powder mixtures (Ti-SiC, Ni-WC, Ni-TiC and Ni-Cr3C2/(Ni-Cr). The observed increase in the surface hardness was in the range of 600-1500 HV. Al-12Si is widely used in automobile industry for manufacturing engine pistons. However, its hardness and wear resistance are found to be insufficient for many engineering applications. The fundamental cause of poor wear characteristics of Al-12Si is its low hardness (100 HV). In the present investigation, attempts are made to form metal-ceramic layers over Al-12Si with various compositions of Ni-SiC using high power CW CO2 laser to improve its hardness and wear resistance. Different laser powers and scan speeds were used. The sample laser surface treated with 25Ni-75SiC (% wt) composition exhibited formation of metal-ceramic layers. The layers exhibited appreciable increase in the microhardness

(500-1200 HV). Microstructure of laser melted zone exhibited dendrite structures. Different phases formed in the laser treated region were identified by XRD.

9:20 AM
Growth of TiN/AlN Superlattice by Pulsed Laser Deposition: *Haiyan Wang*[1]; Ashutosh Tiwari[1]; Abhishek Gupta[1]; Xinghang Zhang[1]; Jagdish Narayan[1]; [1]North Carolina State University, Matls. Sci. & Eng., 2141 Burlington Lab., CB 7916, Raleigh, NC 27695-7916 USA

TiN-AlN binary-components have attracted a lot of interests in coatings of high speed cutting tools, due to their higher oxidation resistance, higher hardness, lower internal stresses and better adhesion. Especially, nanometer-scale multilayer structures of AlN/TiN show superior structural and mechanical properties due to their tremendous interface area and become one of the promising candidates for super-hard coatings. Here we present a novel method to grow highly aligned TiN/AlN superlattice by pulsed laser deposition. In this method TiN and AlN targets are arranged in a special configuration that they can be ablated in sequence, giving alternate layer by layer growth of TiN(1nm)/AlN(4nm). X-ray diffraction and transmission electron microscopy (TEM) analysis showed the structure to be cubic for both TiN and AlN in the nanoscale multilayers. Microstructure and uniformity for the superlattice structure were studied by TEM and Scanning transmission electron microscopy with Z-contrast (STEM). Nanoindentation results indicated a higher hardness for this new structure than pure TiN and AlN and four point probe electrical resistivity measurements showed overall insulating behavior.

9:40 AM
Deposition of Yttria Stabilized Zirconia with Ion-Plating Apparatus: *Guo Chun Xu*[1]; Kazuhisa Fujita[1]; Taiji Torigoe[2]; Yutaka Hibino[1]; [1]Ion Engineering Research Institute Corporation, The 2nd Rsrch. Dept., Tsuda-Yamate 2-8-1, Hirakata, Osaka 573-0128 Japan; [2]Mitsubishi Heavy Industries, Ltd., Matls. & Strength Lab., 2-1-1 Shinhama, Arai-Cho, Takasago, Hyogo 676-8686 Japan

Currently air plasma spraying (APS) and electron-beam physical vapor deposition (EB-PVD) are two prominent processes to fabricate the thermal barrier coating (typically yttria stabilized zirconia (YSZ)) on turbine engine blades. The APS process results in a porous coating with lower thermal conductivity, and the EB-PVD process provides a columnar microstructure with superior tolerance against thermal expansion mismatch. Because the formation of the columnar microstructure requires the substrate temperature over 1000°, a low temperature process is proposed using an ion-plating apparatus equipped with ionization and bias power supplies for the deposition of YSZ. In this presentation we discuss the structural influences of process parameters including the bias voltage, the ionization power as well as annealing after the deposition. The microstructure of the YSZ formed are characterized with Raman spectrometer, X-ray diffraction detector, and scanning electronic microscopy.

10:00 AM
Development of Grain Size Functionally Gradient Materials-Application to Alumina: H. Keshavan[1]; S. Bal[2]; *K. Morsi*[1]; [1]University of Missouri, Mechl. & Aeros. Eng., E3411 Eng. Bldg. E., Columbia, MO 65211 USA; [2]University of Missouri, Dept. of Orthopedic Surgery, Columbia, MO 65211 USA

Nanoceramics have emerged as materials of high technological importance owing to their enhanced properties compared to conventional ceramics. A major draw back for the application of such materials is the higher cost of the precursor nano-powders used compared to conventional ceramic powders. Most mechanical and wear properties of materials are however dictated by the material condition at the surface rather than the bulk. The work presented covers sequential slip casting and optimized hot pressing experiments to produce a continuously decreasing grain size from the bulk to the surface. In other words functionally gradient materials with respect to grain size. The effect of processing parameters on the product microstructure are discussed. The applications for such a technology would be wide, ranging from cutting tools to orthopedic implants.

10:20 AM Break

10:40 AM Invited
Friction Stir Surface Composite Fabrication: *R. S. Mishra*[1]; Z. Y. Ma[1]; [1]University of Missouri, Dept. of Metallurgl. Eng., 218 McNutt Hall, Rolla, MO 65409 USA

Friction stir processing is a new solid state technique for microstructural modification in metallic materials. This has been used to develop a new method for surface composite fabrication. Examples of various SiC reinforced aluminum matrix surface composite will be shown. This relatively fast solid state process avoids any phase stability issues. The transition interface from unreinforced to reinforced region shows excellent bonding. The effects of processing parameters on microstructure and hardness will be presented.

11:10 AM
The Influence of Pulsing and Helium Additions on Particle Energy Distributions on the Structure and Properties of Reactively Sputtered Titanium Oxide and Titanium Nitride Thin Films: Chris Muratore[1]; J. A. Rees[2]; Brajendra Mishra[1]; *John J. Moore*[1]; [1]Colorado School of Mines, Adv. Coatings & Surface Eng. Lab., 1500 Illinois St., Golden, CO 80401 USA; [2]Hiden Analytical, Ltd., 420 Europa Blvd., Warrington, WA5 5UN UK

A plasma probe consisting of an electrostatic ion energy analyzer in series with a quadrupole mass spectrometer was used to study the effect of pulsing the power supply when using inductively coupled plasma (ICP) enhanced reactive deposition of titanium nitride and titanium oxide thin films. The deposition processes for both materials employed an unbalanced magnetron to generate the metal flux, and utilized the ICP as a remote plasma source to excite the reactive gas before it entered the processing chamber. The relative abundances of various oxygen and nitrogen species arriving at the substrate were manipulated by adding small amounts (less than 20% total reactive gas flow) of helium to the reactive gas before excitation by the ICP source. Additionally, higher energy molecular and atomic ions of nitrogen were observed in the presence of helium gas. The variation of plasma chemistry and/or energy distributions associated with helium additions resulted in a 44% increase in hardness and a 22% increase in the smoothness of titanium nitride films. It was also possible to increase the transmission of titanium oxide films in the range of visible light wavelengths by adding helium. Pulsing the plasma source also resulted in denser films, increased hardness and decreased resisistivity of the films. Such modifications of material properties are explained in terms of measured plasma properties.

11:30 AM
Direct Laser Deposition of In Situ Boride Reinforced Titanium Alloy Composites: *Rajarshi Banerjee*[1]; Peter C. Collins[1]; Sean Connors[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Due to their wide applicability, there is considerable interest in the development of metal-matrix composites consisting of hard precipitates, such as transition-metal borides, dispersed in a metallic/alloy matrix. One such system that has generated considerable interest in recent years is the titanium boride in titanium alloy matrix system. Despite the development of a variety of different processing routes for these composites, including some in situ ones, there are relatively few technologies capable of processing a fully dense, near-net shape component with a relatively fine dispersion of boride precipitates. A recent advancement in the field of near-net shape manufacturing techniques is laser engineered net-shaping (LENSô) which falls in the class of direct laser deposition processes using powder feedstock. Using a feedstock consisting of a blend of Ti (or Ti-6Al-4V) and elemental boron powders, TiB reinforced Ti-alloy composites have been deposited via the LENSô process. These as-deposited composites exhibit a refined homogeneous distribution of TiB precipitates within the alloy matrix, a consequence of the rapid solidification rates inherent to the LENSô process. The TiB precipitates are thermodynamically stable and exhibit minimal coarsening even after prolonged exposures to high temperatures. The ability to deposit these composites in situ at precise locations using the LENSô process affords the possibility of using this technology for surface engineering and repairing expensive components of intricate geometry, such as turbine blades used in aircraft jet engines.

The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research: Amorphous Materials, Radiation Effects, Composites, Quasicrystals

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Japan Institute of Metals, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Julia R. Weertman, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Katherine T. Faber, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Morris E. Fine, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Wayne King, Lawrence Livermore National Laboratory, San Ramon, CA 94583-2496 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Ben Mori, Tokyo 168-0081 Japan

Wednesday AM Room: 9
March 5, 2003 Location: San Diego Convention Center

Session Chairs: M. Yamamoto, Osaka University, Matls. Sci. & Eng. Dept., Osaka 565 Japan; Wayne King, Lawrence Livermore National Laboratory, San Ramon, CA 94583 USA

8:30 AM Invited
Microstructure Evolution During Irradiation: *Michio Kiritani*[1]; [1]Hiroshima Institute of Technology, Sch. of Eng., Miyake 2-1-1, Saeki-ku, Hiroshima 731-5193 Japan

Microstructural evolution in metals and alloys during energetic particle irradiation is reviewed with emphasis on the underlying defect reaction processes. The microstructures produced by electon irradiation with a high-voltage electron microscope, fusion-neutron irradiation with a D-T fusion neutron source, fission-neutron irradiation with reactors, and ion irradiation with accelerators are compared and contrasted. By electron irradiation, analyses of defect microstructure developments extend to include the measurement of point defect properties, and the detection of a variety of point defect processes such as radiation-induced diffusion, stochastic fluctuation of point defect reactions, and one dimensional easy motion of small interstitial clusters. By neutron irradiation, analysis of defects produced by cascades and subcascades leads to the recoil energy spectrum analysis. A comparison is made of the damage produced by fission- and fusion-neutron irradiation, to find differences depending on the defect processes involved.

9:00 AM
Nucleation of Vacancy Loops in Quenched Pure Aluminum: *Kuan Yeul Victor Chen*[1]; [1]Formerly of Northwestern University, Matls. Rsrch. Ctr. & Dept. of Matls. Sci. & Eng., Evanston, IL, Presently at 2715 Cazadero Dr., Carlsbad, CA 92009 USA

The nucleation of vacancy loops has been studied in nominally 99.999% pure aluminum using transmission electron microscopy and electrical resistivity measurements. The clustering of supersaturated vacancies after quenching was examined with a two-step aging treatment. The temperatures characterizing the nucleation of vacancy loops in aluminum are analyzed. For aluminum quenched from 610°C into liquid nitrogen and preaged at temperature -60°C or lower, the rate controlling process for the nucleation of vacancy loops is the migration of single vacancies at the beginning of preaging. Toward the end of preaging, the nucleation rate is dominated by the migration of divacancies. At temperature higher than -40°C, dissociation of subcritical clusters is noticeable. The nucleation rate is found to vary with the concentration of quenched-in vacancies and the aging temperature. This investigation suggests that the migration energy, E_m^2, of a divacancy is $E_m^2 = 0.47 \pm 0.02$ eV.

9:20 AM
In-Situ Observation of Brownian Motion and Dynamical Response to Irradiation of Helium Bubbles in Al and Cu: *Kotaro Ono*[1]; Kazuto Arakawa[1]; Robert C. Birtcher[2]; [1]Shimane University, Dept. of Matls. Sci., 1060 Nishi-Kawatsu, Matsue, Shimane Prefecture 690-8504 Japan; [2]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA

The dynamical response to irradiation of helium bubbles in pure Al has been studied in-situ using high-energy self -ions irradiation in an electron microscope. Helium bubbles were introduced in pure Al by pre-irradiation of He+ ions. TEM specimens were then warmed up stepwise to higher temperatures. The dynamical behaviors of the bubbles during beam on and beam off periods were continuously monitored by TEM and recorded with a video recording system. At enough high temperature, Brownian type motion of helium bubbles was quantitatively demonstrated during the beam off periods. While, most of helium bubbles were retarded and a few bubbles were accelerated during the beam on period. At low temperatures when bubbles are thermally immobile, irradiation of high energy self-ions caused intermittent bubble motion. These results are interpreted in terms of competitive point defects absorption to bubbles and easy migration of bubbles along interstitial type dislocation loops during the irradiation with high energy self-ions.

9:40 AM Invited
Is Segregation-Induced Grain Boundary Embrittlement a Kinetically-Constrained Melting Process?: *Paul R. Okamoto*[1]; Jonas K. Heuer[2]; Nghi Q. Lam[1]; [1]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., Argonne, IL 60540 USA; [2]Bettis Atomic Power Laboratory, Matls. Tech., PO Box 79, W. Miffin, PA 15122 USA

The kinetics of intergranular embrittlement of dilute polycrystalline Ni-S alloys and that of S-implantation-induced amorphization of single crystal Ni were investigated. Plots of % intergranular fracture versus S-grain boundary concentration and that of the amorphous volume fraction versus S-implant concentration were found to be virtually identical, implying that (1) the implanted S-atoms and those segregating to grain boundary sites are subject to the same kinetic-constraint (i.e. drastically reduced S-diffusivity), and (2) that their distributions obey the same Poisson statistics. Hence, polymorphous melting may be the physical origin of both amorphization and segregation-induced embrittlement, an interpretation which is supported by the fact that the critical S-concentration for 50% intergranular fracture and 50% amorphization are equal to the maximum nonequilibrium solubility limit of 17 at. % S defined by the polymorphous melting curve on the Ni-S phase diagram. The extension of the polymorphous melting concept to two co-segregating impurities provides a simple explanation for the well-known synergistic effects of hydrogen and sulfur co-segregation on embrittlement.

10:10 AM
Friction-Induced Solid-State Amorphization from Non-Equilibrium Solid Solution Phase in Fe-Cr-B-Ni-Mo Spray Coatings: *C. G. Park*[1]; H. W. Jin[1]; M. C. Kim[2]; [1]Pohang University of Science & Technology, Ctr. for Adv. Aeros. Matls., Pohang 790-784 Korea; [2]Research Institute of Industrial Science & Technology, Pohang 790-600 Korea

Experimental evidence of friction-induced amorphization is presented from cross-sectional TEM investigations. Rapid quenching of Fe-Cr-B-Ni-Mo alloys resulted in the non-equilibrium microstructure composed of micro-crystalline $(Cr,Fe)_2B$ borides and nanocrystalline α-(Fe,Cr) solid solution phase supersaturated with B and Si. Cross-sectional TEM observations revealed that the crystalline-to-amorphous phase transition occurred in the outer surface layer of the α-(Fe,Cr) phase. The spray coatings exhibited significantly enhanced wear resistance with a significantly low friction coefficient (~0.04) with a presence of thin (~150 nm) amorphous surface layer. Theoretical calculations based on thermodynamic instability of crystalline phase suggest that the super-saturation of the α-(Fe,Cr) solid solution phase and the introduction of lattice defects due to frictional work are the major factors to trigger the amorphization reaction.

10:30 AM
A High-Resolution TEM Study of Deformation-Induced Shear-Band Formation and Crystallization in Amorphous $Al_{90}Fe_5Gd_5$: Wenhui Jiang[1]; *Michael Atzmon*[1]; [1]University of Michigan, Cooley Bldg./N. Campus, NERS, Ann Arbor, MI 48109-2104 USA

In several amorphous, Al-based, alloys, Al-rich nanocrystallites have been reported to precipitate at shear bands formed by plastic deformation. The mechanism of this room-temperature process is still a subject of debate. While large, local, temperature spikes are expected at shear bands, the cooling rates are very high, suggesting that the crystallization cannot be explained by a temperature rise. In an effort toward explaining mechanically assisted nanocrystalliztion in these alloys, we have investigated the nanoscale structure using images constructed from the small-angle signal in reciprocal space, excluding thickness effects. We observe nanoscale voids to form at shear bands in the tensile, but not in the compressive region. On the other hand, Al nanocrystallites at shear bands are observed only in the compressive region. These results support a crystallization mechanism based on defect production in the solid state.

10:50 AM
Sea Urchin Mineralized Tissue: *Stuart R. Stock*[1]; [1]Northwestern University, IBNAM, Tarry 16-717, 303 E. Chicago Ave., Chicago, IL 60611-3008 USA

Sea urchin ossicles are structural analogs of mammalian bones and a model biomineral system. Relatively new x-ray methods complement electron microscopy and allow study of mineralized tissue with voxels (volume elements) approaching 1 µm3 in millimeter-sized samples. This talk focuses on a multi-mode x-ray investigation of mineralized tissue using microCT, both synchrotron and laboratory sources, phase contrast radiography and transmission microbeam diffraction mapping. Sea urchins employ as wide a range of composite reinforcement strategies as are seen in engineering composites, and, studied as materials, teeth (and other ossicles) from different echinoid families illustrate combinations of reinforcement parameters and toughening mechanisms providing good functionality. This probe of the design space available to sea urchins offers important guidance for engineering of structural tissue. The results on sea urchin teeth and pyramids (jaws) also illustrate what might be accomplished by such an integrated approach in bone.

11:10 AM
Microstructures in an Al-Cu-Fe Quasi-Crystal After Deformation at Room Temperature: *S. Miyazaki*[1]; C. Hirose[2]; S. Kumai[1]; A. Sato[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., 4259 Nagasuta, Midori-ku, Yokohama 226-8502 Japan; [2]LSI Logic Japan Semiconductor Corporation, 10 Kitahara, Tsukuba-shi, Ibaraki 300-0032 Japan

Plastic deformation was given to a specimen containing Al-Cu-Fe quasi-crystals. The specimen thickness as well as strain rate were altered in order to seek a deformable condition at room temperature. It is found that the quasi-crystals embedded in Al do not deform in the usual compression test of a bulk specimen but in a thin foil specimen of the order of the quasi-crystal size they deform plastically under the high stress acting on them directly. A large number of line contrasts, presumably planar defects across a foil specimen, were introduced by a compressive stress applied on the foil. A high density of dislocations was introduced simultaneously by the compression test. Burgers vectors of the glide dislocations were determined by visibility criterion. The dislocation type was different from those reported in the literature after high temperature deformation.

11:30 AM
Interactive Effect of Temperature, Stress, Moisture, and Physical Aging on Creep and Creep Rupture of a Polymer Composite Matrix: *Raghavan Jayaraman*[1]; [1]University of Manitoba, Mech. & Indl. Eng., 348D, Eng. Bldg., 15, Gillson St., Winnipeg, MB R3T 5V6 Canada

Individual and interactive effect of temperature, stress, moisture, and physical aging on creep and creep rupture of Hexcel F263 epoxy were experimentally evaluated and modeled. Individually, increase in temperature, stress, and moisture accelerated creep and creep rupture of the epoxy and increase in physical aging retarded them. Under combined loading conditions, there was no interaction among temperature, stress, and moisture; however, there was significant interaction among physical aging, moisture, and stress. A creep model based on superposition principle and a creep rupture model based on a critical fracture energy criterion have been used to model the experimental data. Fractographs are presented and discussed, in support of the critical fracture energy criterion used in this study.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Iron and Steel Making Fundamentals I

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors
Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday AM — Room: Leucadia
March 5, 2003 — Location: San Diego Marriott Hotel

Session Chairs: V. Sahajwalla, The University of New South Wales, Sch. of Matls. Sci. & Eng., Sydney, NSW Australia; Takashi Nakamura, Tohoku University, Dept. of Metall. Grad. Sch. of Eng., Sendai Japan

8:30 AM Invited
A Basic Study on the Effective Utilization of Chromium-Containing Steel Slag: *Etsuro Shibata*[1]; Takashi Nakamura[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 1,1 Katahira, 2-Chome, Aobaku, Sendai 980-8577 Japan

The solubility of chromium oxide and valance of chromium in slag are important to consider how to utilize effectively the chromium-containing slag. Those experimental data of various types of slag cited in literatures were arranged using optical basicity, and the effects of oxygen potential and slag composition were investigated. The saturated solubility of chromium oxide and the ratio of Cr2+ to Cr3+ tend to decrease with increase in optical basicity of slag at the oxygen potential below 10(-5) atm. On the other hand, at the oxygen potential of 0.21 atm, the saturated solubility and the ratio of Cr6+ to Cr3+ tend to increase with increase in optical basicity. For a practical approach to reduce chromium in stainless steel slag and simultaneously recover chromium as a ferroalloy, the direct smelting reduction of chromium oxide in molten slag using some reductant was also investigated using a small furnace.

8:55 AM Invited
Kinetics of Carbon Injection into Metallurgical Slag: F. Ji[1]; M. Barati[1]; *K. S. Coley*[1]; G. A. Irons[1]; [1]McMaster University, McMaster Steel Rsrch. Ctr., Hamilton, Ontario L8S 4L6 Canada

Carbon injection into metallurgical slags is important in EAF steelmaking for slag foaming and in nonferrous slag cleaning. The current publication presents kinetic data for coal injection into electric arc furnace slags to draw some general conclusions about the interaction between injected carbon and metallurgical slags. Coal particles were injected into EAF slags and the kinetics were followed by gas and slag analysis. The results were analyzed, in terms of fundamental rate equations for each possible reaction step, as a function of temperature and slag composition.

9:20 AM
Semi-Stochastic Optimization of Chemical Composition of High-Temperature Austenitic Steels for Desired Mechanical Properties: *George S. Dulikravich*[1]; Igor N. Egorov[2]; Vinod K. Sikka[3]; Govindarjan Muralidharan[3]; [1]University of Texas at Arlington, Mech. & Aeros. Eng. Dept., Multidisciplinary Analy., Inverse Design & Optimization (MAIDO) Prog., UTA Box 19018, Arlington, TX 76019 USA; [2]IOSO Technology Center, Milashenkova 10-201, Moscow 127322 Russia; [3]Oak Ridge National Laboratory, Matls. Procg. Grp., PO Box 2008, MS6083, Metals & Ceram. Div., Oak Ridge, TN 37831-6083 USA

The methodology for steel optimization subject to several simultaneous objectives consists in organization an iterative optimized experiment. The result of these studies is the Pareto-optimal set of steel compositions that simultaneously optimizes the chosen objectives. The multi-objective optimization algorithm is based upon the use of a response surface that is created from the available experimental data with the help of radial-basis functions and artificial neural networks.

During the conduct of research the information is being stored concerning the properties of steel in the vicinity of the Pareto set. This allows us to improve the accuracy of the results. As the independent design variables we considered the percentage of following components: C, Mn, Si, Ni, Cr, N. Ranges of their variation were set in accordance with lower and upper bounds of the available set of experimental data. As the main optimization objective we considered the strength of the H-type steel after 100 hours under the temperature of 1800F. Other objectives have been chosen based on the necessity to reduce the cost of the steel. In this work, the additional three objectives were to simultaneously minimize the percentages of Mn, Ni, Cr. Thus, the multi-objective optimization problem had 6 independent design variables and 4 objectives. We defined the desirable number of Pareto optimal solutions as 10 points. Every iteration of this methodology results in a formulation of a set of steel compositions, which are Pareto optimal and need experimental evaluations to obtain the true values of the objectives.

9:45 AM
ìIn-Situî Observation of Iron Carburization During Smelting Reduction: *Ko-ichiro Ohno*[1]; Tetsuya Nagasaka[2]; Mitsutaka Hino[2]; [1]Tohoku University, Dept. of Metall., Aza-Aoba 02, Aramaki, Aoba-ku, Sendai 980-8579 Japan; [2]Tohoku University, Dept. of Metall., Grad. Sch. of Eng., Aoba-yama 02, Sendai 980-8579 Japan

It was recently recognized that the reduction, melting of iron and slag separation in the composite of granular iron ore and coal are completed in a very short period of about 10 minutes when the composite is heated rapidly up to approximately 1673K. Utilization of these phenomena is attempted for new iron-making process. The mechanism of iron ore reduction and iron-slag separation during rapid heating has not yet been realized, and the fundamental research has just been started. Carburization of the reduced iron is regarded as an important factor in this process. In the present work, the composite was prepared from four kinds of coal or graphite and electrolytic iron powder. The temperature at which liquid were formed and iron was carburized was monitored at various heating speeds. Direct observation of smelting reduction behavior was made by a laser microscope combined with the infrared image heating furnace to clarify the effect of molten ash on iron carburization. The meltdown temperature of composite fell as melting temperature of coal ash fell. From the observed result, it was presumed that liquid formation in composite triggered carburization of iron. Carburization mechanism during smelting reduction was considered as follows. When slag containing iron oxide melted down and contacted with carbon, iron oxide in slag was reduced and metallic iron particle was formed and carburization of the iron particle occurred simultaneously at slag-carbon interface. Carburized iron particle was carried from slag-carbon to slag-iron interfaces due to slag convection flow caused by the difference of interface tension between carbon-slag and iron-slag interfaces. Repeating the above-mentioned process continuously carburized the reduced iron.

10:10 AM Break

10:20 AM Invited
Fundamental Investigation of Basic Mechanisms of Carbon Dissolution in Molten Iron: *V. Sahajwalla*[1]; R. Khanna[1]; [1]The University of New South Wales, Sch. of Matls. Sci. & Eng., Sydney, NSW 2052 Australia

Based on experimental and computer simulation results from our group, we report a systematic, atomic level analysis of carbon dissolution from carbonaceous materials in molten iron. Experimental results were obtained using carbon dissolution studies, wetting, XRD and FESEM investigations, and Monte Carlo simulations on Graphite/Fe-C-S system. Three basic mechanisms, namely carbon atom dissociation rate, interfacial phenomena and mass transfer in the melt were found to be responsible for the overall dissolution process. While mass transfer in the melt is governed by the concentration gradients, impurities such as sulphur and temperature, sulphur and oxide impurities present in ash play a significant role in the interfacial phenomena. Carbon atom dissociation kinetics depends strongly on the degree of long-range order of carbonaceous materials and is an important rate controlling mechanism in the overall dissolution kinetics of less ordered forms of carbon. A systematic comparison of dissolution rates for a var iety of coals, cokes, chars, natural and synthetic graphite has been carried out in an attempt to identify rate-controlling mechanisms. The structure of the carbonaceous material appears to be an important underlying factor in carbon dissolution.

10:45 AM Invited
Dissolution of Alumina Particles in CaO-Based Fluxes: *W. D. Cho*[1]; Peter Fan[1]; [1]University of Utah, Dept. of Metallurgl. Eng., Salt Lake City, UT 84112 USA

The slag chemistry in steel processing plays an important role in terms of the removal of impurities including alumina particles. In the present study, the dissolution of solid alumina particles in CaO-based fluxes at the temperatures between 1450 and 1550°C has been investigated to determine dissolution kinetics and mechanism. Alumina particles were added directly in the slags and the size of alumina particles was measured as a function of time using optical and scanning electron microscopes. The dissolution rate has also been obtained as a function of SiO2, CaF2, MgO and MnO contents in the slags. The diffusion boundary layer between alumina and bulk slag phase has been observed and analyzed using SEM and EDX. In addition, some experiments have been performed for the dissolution of alumina in BaO-based fluxes.

11:10 AM
The Effect of MgO and Al2O3 Additions on the Liquidus for the CaO-SiO2-FeOx Systems at Various Partial Pressure of Oxygen: Hisao Kimura[1]; *Fumitaka Tsukihashi*[2]; [1]The University of Tokyo, Grad. Sch. of Eng., 7-3-1 Hongo, Bunkyo, Tokyo 113-8656 Japan; [2]The University of Tokyo, Grad. Sch. of Frontier Scis., 7-3-1 Hongo, Bunkyo, Tokyo 113-0033 Japan

Phase diagrams for the CaO-SiO2-FeOx-MgO, Al2O3 systems are necessary for the analysis of copper smelting reaction and sintering process of iron ore. The effect of MgO and Al2O3 addition on the liquidus of phase diagram for the CaO-SiO2-FeOx systems at low oxygen partial pressure was observed. The liquid phase area was changed by the addition of MgO and Al2O3 with changing oxygen partial pressure from 10^{-8} to 10^{-3} atm at 1573K. The effect of Fe2+/Fe3+ ratio on the melting mechanism is discussed.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Elemental Losses and Distributions

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday AM Room: Solana
March 5, 2003 Location: San Diego Marriott Hotel

Session Chairs: Yoichi Takeda, Iwate University, Fac. of Eng., Morioka 020-8551 Japan; Kimio Itagaki, Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Sendai, Miyagi 980-8577 Japan

8:30 AM Keynote
Distribution of Minor Elements in Sulfide Smelting: *Kimio Itagaki*[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi 980-8577 Japan

Various kinds of molten sulfide systems of Cu2S-FeS, Ni3S2-FeS, Ni3S2-Cu2S-FeS, ZnS-Cu2S, PbS-Cu2S, Ni-S and Pb-S were equilibrated with molten FeOx-SiO2, FeOx-CaO and Al2O3-CaO based slags in a magnesia crucible under controlled partial pressures of SO2, O2 and S2 to determine the distribution ratios of some valuable(Ag, Au, Co, Ni etc.) and detrimental(Pb, As, Sb, Bi etc.) minor elements in smelting various sulfide concentrates. The experimental results were analyzed on the basis of thermodynamics and the effects of partial pressures of oxygen and sulfur as well as slag basicity were discussed in this study.

9:00 AM
Thermodynamic Evaluation of Arsenic and Antimony on the Bessemer Matte and Calcium Ferrite Slag: *Jonkion M. Font*[1]; Mitsuhisa Hino[2]; Kimio Itagaki[2]; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Sendai, Miyagi 980-8577 Japan

Due to the known disadvantages of the use of iron silicate slag in the nickel converting stage, i.e., high metal losses, high magnetite content, and low fixation of impurities in the slag, the development of the new proposed ìferrous calcium silicateî slag for smelting and converting processes of nickel is very promising. Hence, considering the successful experimental results of using CaO base slag for the nickel smelting process, a study based on a detailed comparison for the phase equilibrium, and the minor elements distribution between the Ni_3S_2-FeS matte with either FeO_X-CaO or FeO_X-SiO_2 base slag was investigated. The main results are summarized as: no difference in the nickel losses between both slags was found at high nickel content in the matte, and the fractional distribution analysis for arsenic and antimony pointed out their preferential fixation in the calcium ferrite slag rather than in the iron silicate slag. The technical feasibility of using calcium ferrite slag in a converting process of the Bessemer matte will have a prominent future for the nickel industry when the energy and pollution issues are taken into account.

9:25 AM

Thermodynamics of PbO, ZnO and CuO0.5 in CaO-SiO2-Al2O3 Melts: *Takaaki Ishikawa*[2]; Kenji Matshuzaki[3]; Takayuki Tsukada[1]; Kimihisa Ito[1]; [1]Waseda University, Matls. Sci. & Eng., 3-4-1, Okubo, Shinjuku-ku, Tokyo 169-8555 Japan; [2]Chiba Prefectural Machinery & Metallurgy Research Institute, Chiba 263-0016 Japan; [3]Mitsui Mining & Smelting, Rsrch. Ctr., Saitama 362-0021 Japan

The distribution equilibria of lead, zinc and copper between CaO-SiO2-Al2O3 melts and liquid copper were measured at 1623K under a controlled H2-CO2 atmosphere. The distribution ratios were plotted against the oxygen partial pressure, and reasonable oxide forms dissolved in the melts were estimated from the slopes of the plots. The activity coefficients of lead oxide (PbO), zinc oxide (ZnO) and cuprous oxide (CuO0.5) increased with increasing slag basicity, defined by XCaO/XSiO2. The temperature dependence of the activity coefficients was also measured.

9:50 AM

Distribution of Minor Elements Between Ni-S Melt and Al_2O_3-CaO-MgO Slag at 1873 K: *Hector Mario Henao*[1]; Mitsuhisa Hino[1]; Kimio Itagaki[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls. (IMRAM), 1,1 Katahira, 2-Chome, Sendai, Miyagi 980-8577 Japan

To provide thermodynamic data for converting the nickel matte to liquid nickel, distribution ratios of some minor elements (Au, Ag, Cu, Co, Fe, P) between the Ni-S alloy and the Al_2O_3-CaO-MgO slag in a magnesia crucible were determined at 1873 K under controlled PSO_2 of 10.1 kPa and PO_2 in a range between 8.0 and 0.1 Pa by using CO-CO_2-SO_2 gas mixtures. It was found that the distribution ratios, $L_X^{S/Ni}$, defined by (mass%X in slag)/{mass%X in alloy}, for P, Fe are larger than unity, while less that unity for Au, Ag, Cu, Co.

10:15 AM Break

10:25 AM Keynote

Thermodynamic Evaluation of Copper Loss in Slag Equilibrated with Matte: *Yoichi Takeda*[1]; [1]Iwate University, Fac. of Eng., Ueda 4-3-5, Morioka 020-8551 Japan

Main focus is the determination of copper solubility in SiO2-CaO-FeOx slag equilibrated with matte under specified SO2 atmosphere. A classical thermodynamic model postulating CuO0.5 and CuS0.5 molecules applies to calculate copper solubility in slag. A large number of experiments provide proper parameters: activity coefficients of copper oxide and sulfide, oxygen and sulfur potentials and activity of copper in the slag-matte system. Copper loss , that is function of copper solubility and slag volume, in the silica saturated SiO2-FeOx binary slag is minimally equilibrated with less than 66% copper matte while copper loss in the 50% FeOx-SiO2-CaO is minimally equilibrated with over 66% copper matte. This slag composition has potential for continuous copper converting, direct copper production or high-grade matte production.

10:55 AM Invited

Copper Losses in Copper Smelting Slags: *Ivan Imris*[1]; [1]Technical University of Kosice, Dept. of Power Eng., Fac. of Mechl. Eng., Letna 9, Kosice 041 87 Slovak Republic

The common problem of new and conventional technology of copper production is the copper losses in the slags, which may be divided into mechanical and physico-chemical losses. In the first group can be included particles of mechanically entrained or floated unsedimented matte particles which coexist with slag. On the other hand, the physico-chemical losses are caused by solubility of copper in the slag in the form of sulphide and oxide. The copper losses in the slags from different processes has been predicted by calculation from thermodynamic data and compared with those determined by microscopic examination and quantitative electron microprobe analysis. Depending on the forms of copper losses in the slags the reduction of copper losses in the slag or the slag cleaning process could be suggested.

11:20 AM

Distribution Behavior of Arsenic, Antimony and Bismuth: *Supachai Surapunt*[1]; Nozomu Hasegawa[2]; [1]Thammasat University, Fac. of Eng., 99 Phaholyothin Rd., Klong-Luang, Pathumthani 12121 Thailand; [2]Naoshima Smelter and Refinery, Mitsubishi Metal Corp., Naoshima, Kagawa Japan

The distribution behavior of VA minor elements (As, Sb and Bi) in the smelting stage of the Mitsubishi process were evaluated thermodynamically based on thermodynamic data and operating data by using the method of calculation proposed by Itagaki and Yazawa. T h e fractional distributions between the gas, slag and matte phases considered as the degree of vapor saturation of 0.8 are as follows: 53.8, 27.2 and 19% for As, 23.6, 56.6 and 19.8% for Sb, and 91.3, 1.1 and 7.6% for Bi, respectively. Arsenic is mostly distributed to the gas and slag phases. It is effectively eliminated by volatilizing and slagging. Antimony is mainly in the slag phase which is suitably removed by slagging. Bismuth is easily vaporized to gas phase. The degree of vapor saturation has a large effect on the distribution behaviors of the three elements between the three phases. The amount of a minor element in the charge has an effect on the distribution of As but no effect on the distributions of Sb and Bi. Increasing matte grade and temperature results in the change of distributions for As and Sb, significantly. The change in the distribution ratio between slag and matte phases also considerably affects the distributions of As and Sb. The distribution behavior of Bi is not significantly changed by the change in matte grade, temperature and distribution ratio.

11:45 AM

The Influence of Reverb Slag Composition on Copper Losses: *Natasa Mitevska*[1]; *Zivan D. Zivkovic*[1]; [1]Copper Institute, Zeleni bulevar 35, Bor 19210 Yugoslavia

The statistical analysis of the slag composition influence on copper losses to discard slag in the reverberatory furnaces No. 1 and No. 2 in the RTB BOR, Copper Smelter and Refinery (Yugoslavia) is presented in this paper. The model for the slag basicity calculation is determined, and connected to the copper distribution coefficients between the matte and slag phase. The influence of all components on the slag structure is also illustrated.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Materials Processing Technologies: General I

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday AM Room: Point Loma
March 5, 2003 Location: San Diego Marriott Hotel

Session Chairs: Toshiyuki Kozuka, Kumamoto University, Dept. of Matls. Sys., Kumamoto 860-8555 Japan; Y. B. Hahn, Chonbuk National University, Chonju 561-756 Korea

8:30 AM Invited

Fabrication of Blue Light-Emitting Diodes Using GaN-Based Multiple Quantum Wells Grown by Metal Organic Chemical Vapour Deposition: *Yoon-Bong Hahn*[1]; Rak-Jun Choi[1]; Hyung Jae Lee[1]; [1]Chonbuk National University, Sch. of Cheml. Eng. & Tech., Semiconductor Physics Rsrch. Ctr., 664-14 Duckjin-Dong 1 Ga, Chonju 561-756 Korea

Gan-based blue light emitting diodes (LEDs) were fabricated by utilizing nanoscale triangular- and rectangular-type multiple quantum wells and their characteristics were compared in terms of structural, electrical and optical properties. Optimization of the LED fabrication process was also investigated by minimizing a plasma-induced damage. The InGaN/GaN multiple quantum wells (MQWs) were grown by a low-pressure metalorganic chemical vapour deposition method. The size of quantum dots formed in the active layer of InGaN was in a range of 2 to 50 nm, and the dots in the triangular QWs showed very uniform spatial distribution compared to those in the rectangular QWs. Both photoluminescence (PL) and electroluminescence (EL) showed higher emssion and smaller full-width at half maximum in QWs and LED structures with triangular QWs than those with rectangular QWs. Especially, EL spectrum as a function of injection current showed that the peak energy is nearly independent of the injection current in the triangular-QW -based LEDs. In the course of LED fabrication a physical degradation of sidewall along with rough surface morphology of n-GaN caused by increased ion scattering induced the deterioration of forward and reverse voltages. It was found that the turn-on voltage is sensitive to the surface roughness of the etched n-GaN and the breakdown voltage is strongly affected by the sidewall contamination. Annealing under nitrogen after the mesa etching improved the electrical properties of the InGaN/GaN MQW LEDs.

9:00 AM

Sintering of Pb{Zr,Ti,(Mg1/3Nb2/3)}O3 Ceramics by Spark Plasma Sintering and their Compositional Distribution: *Kazuyuki Kakegawa*[1]; Satoru Sawahara[2]; Naofumi Uekawa[3]; [1]Chiba University, Grad. Sch. of Sci. & Tech. & Dept. of Matl. Tech., Fac. of Eng. & Ctr. for Frontier Elect. & Photonics, 1-33 Yayoi-cho, Inage-ku, Chiba-shi, Chiba 263-8522 Japan; [2]Chiba University, Grad. Sch. of Sci. & Tech., Chiba 263-8522 Japan; [3]Chiba University, Dept. of Matl. Tech., Fac. of Eng. & Ctr. for Frontier Elect. & Photonics, Chiba 263-8522 Japan

Both sintering and homogenization of ceramic solid solutions are the result of diffusion during the heating at high temperatures. Thus homogenization generally accompanies with sintering. It is a normal fact that distribution of composition decreases as the sintered density of the sample increases. We found an interesting fact that dense sintered body could be obtained with almost no change in the compositional distribution, when spark plasma sintering (SPS) method was employed. This paper shows such results in Pb{Zr,Ti,(Mg1/3Nb2/3)}O3 solid solution. The change in the compositional distribution of the sintered body by SPS was compared with that by the normal sintering. By normal sintering, the compositional distribution decreased as the sintered density increased as normally expected. On the contrary, the compositional change by SPS was much smaller than that by the normal sintering. This sintering characteristic of SPS enabled a fabrication of sintered material having a desired compositional distribution.

9:25 AM

Mg Alloy Composite Materials with Solid-State Synthesized Mg2Si Dispersions: *Katsuyoshi Kondoh*[1]; Wenbo Du[1]; Ritsuko Tsuzuki[1]; [1]The University of Tokyo, Rsrch. Ctr. for Adv. Sci. & Tech. (RCAST), 4-6-1, Komaba, Meguro-ku, Tokyo 153-8904 Japan

The synthesis processing of Mg_2Si or Mg_2Si/MgO at lower temperature from the elemental magnesium alloy chips and Si or SiO_2 powder mixture was established via the cyclic plastic working based on Powder Metallurgy (P/M) process, which is effective on the mechanical breakage of MgO surface films preventing from the solid-state reaction between Mg and Si powder. The Mg_2Si grain of 200-500nm is extremely fine, compared to that via the conventional casting process, because of not coarsening during the solid-state synthesis of Mg_2Si. By using in-situ forming process of Mg_2Si via the cyclic plastic working, the magnesium composite material with Mg_2Si/MgO dispersions, which particle size was less than 1-3μm, was developed. For example, when employing AZ31 chips as raw materials of the matrix, it has low density of 1.85g/cm3 and extremely superior mechanical properties, such as tensile strength over 350MPa and Youngís modulus of 50GPa, to the conventional Mg-Al-Zn alloys. Concerning to the tribological property under the wet lubricant condition, it shows a low friction coefficient less than 0.04 and no seizure phenomenon with contacting to the S35C steel counter material. In particular, when including the formed MgO particles, the coefficient reduced to 0.02 due to its mild offensive property on the counter part. Its wire and pipe were produced via the mass hot extrusion production in manufacturing plant, and also showed excellent mechanical properties. The optimization of breezing and welding conditions on this developed Mg composite are going in this study.

9:50 AM

Reaction of Sn-Containing Solders with Nickel Based Under Bump Metallizations: *Guojun Qi*[1]; Min He[2]; Zhong Chen[2]; [1]Singapore Institute of Manufacturing Technology, Process Tech. Div., 71 Nanyang Dr. 638075 Singapore; [2]Nanyang Technological University, Sch. of Matls. Eng. 639789 Singapore

This work relates to wafer bumping technology development for flip chip packaging applications in the electronics industry. Nickel is an alternative under bump metallization (UBM) material because of its slower reaction rate with Sn-based solders as compared to Cu-based UBMs. Two types of Ni-based UBMs are widely used: sputtered nickel and electrolessly plated Ni-P alloys (EN). In this study we compared the interfacial morphology of Sn-containing solders (Sn-Ag and eutectic Sn-Pb) with these two types of nickel based under bump metallizations. Both chunky and needle type of intermetallics were observed between the EN UBM and the solders. Their morphology changed with different cooling rates below the melting temperatures of the solders. In the case of the sputtered nickel UBM, there was only a layer of scallop-type intermetallics formed. These differences are discussed in terms of soldering reaction at the interface. Kinetics of the intermetallic growth was also examined on the two UBM systems.

10:15 AM Break

10:35 AM Invited

Effect of Intense Magnetic Field on CdTe Electro-Deposition: *Toshiyuki Kozuka*[1]; Yoshinori Sugita[1]; Masayasu Kawahara[1]; [1]Kumamoto University, Dept. of Matls. Sys., 2-39-1, Kurokami, Kumamoto 860-8555 Japan

CdTe metal composite semi-conducting material has high potential for good performance of energy conversion efficiency, so that there is much room for improvement of energy conversion efficiency according to the processing of making thin CdTe film. In this paper, the imposition of intense magnetic field on CdTe electro-deposition is proposed, which is one of possibility for improving the energy conversion efficiency. In the experiment, cryogen-free superconducting magnet up to 5T was used and CdTe film was deposited electrically in -0.72V vs. SHE. The electrolyte was Cd and Te ammonia alkali solution with controlled temperature in the acrylic cell of 70mm diameter. Intense magnetic field up to 5T can not only make the deposition surface smooth, but also the size of CdTe crystal.

11:05 AM Invited

Ceramic-Metal Composites Obtained by Reactive Pressureless Counterflow Infiltration/Penetration (RPCI) of a Ceramic Substrate with Two Different Metallic Infiltrants: *V. M. Kevorkijan*[1]; [1]Independent Researching, Betnavska cesta 6, Maribor 2000 Slovenia

A new method for faster production of functionally graded metal-ceramic laminates is based on reactive pressureless counterflow infiltration/penetration of a ceramic substrate with two different metallic infiltrants. The porous or dense ceramic preform is pressurelessly infiltrated or penetrated from one side by the first metallic infiltrant to the desired cross section of the preform, producing the top layer made up of the solidified infiltrant and the underlayer consisting of reactive infiltrated preform. The partly infiltrated preform is then completely infiltrated starting from the other side by the second metallic infiltrant, in this way fabricating the internal layer, consisting of the ceramic preform infiltrated with the second infiltrant and the bottom layer formed by the solidified second infiltrant. Systems already investigated include carbide, nitride and boride ceramic performs infiltrated with aluminum and magnesium alloys. The combination of ferrous and non-ferrous infiltrants (aluminum alloys and cast gray iron) was also experimentally studied.

11:35 AM

Preparation of Vanadium-Doped SnO2 Nanocrystallites: *Huaming Yang*[1]; Weiqin Ao[1]; Chenghuan Wang[1]; Guanzhou Qiu[1]; [1]Central South University, Dept. of Minl. Eng., Changsha 410083 China

The vanadium-doped SnO2 nanoparticle was synthesized by the co-precipitation reaction and subsequent calcination from the vanadium(III) chloride and tin(IV) chloride. The crystal size, pore size distribution, micrograph and properties of the nanocrystalline powders were examined by differential thermal analysis (DTA), thermogravimetric analysis (TGA), X-ray diffraction (XRD), desorption isotherm (Barrett-Joyner-Halenda method) and transmission electron microscopy(TEM). Thermal treatment of the precipitate powder at 600°C led to the formation of V-SnO2 nanoparticle of ~10nm in crystal size. Most of the pores in the nanoparticle are about 5~15nm in diameter. Effect of doped vanadium on the crystal size of the nanoparticle was discussed.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Aqueous and Electrochemical Processing IV

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday AM — Room: Pacific
March 5, 2003 — Location: San Diego Marriott Hotel

Session Chairs: Tsutomu Yamamura, Tohoku University, Sendai, Miyagi Prefecture 980-8579 Japan; Dingfan Qiu, Beijing General Research Institute of Mining and Metallurgy, Beijing 100044 China

8:30 AM Invited

Extraction and Separation of Metals from Sulfide by Slurry Electrolysis Process (SEP): *Qiu Dingfan*[1]; Wang Jikun[1]; [1]Beijing General Research Institute of Mining and Metallurgy, Wenxing St., Xizhirncnwai, Beijing 100044 China

Slurry Electrolysis Process (SEP) is a new hydrometallurgical method and clean technology. Sulfide ores can be treated by SEP without S02 emission, and the sulfur from the sulfides will be recovered in element form which is easy to stockpile and can be transported at low cost. In a special slurry electrolysis cell, it can achieve leaching of elements from the feed and electrowinning of metals from the electrolyte at the same time. Because the anode reactions are utilized for leaching some elements, the consumption of the techological energy is decreased. Author has researched the behavior of different metals in the process and point out that separation of metals in some minerals is possible by SEP. Beijing General Research Institute of Mining and Metallurgy (BGRIMM) has researched SEP to treat bismuth concentrate contain S, Be and F from laboratory to pilot-plant and commercial plants. The commercial plant was set up in 1997; it has been in operation since that time. SEP also has advantage of separation some elements. It is especially suitable for treatment of complex concentrates, which could hardly be accepted by any smelter. This kind of mineral contains Pb; Cu, Au, Ag and S. According to the results of lab experiment and pilot-plant test, a new plant to treat complex gold minerals was set up in 1998 and was in operation in 1999.

9:05 AM

Gold Leaching by Using Ammonium Thiosulfate Solution and Gold Recovery by Solvent Extraction and Cementation: *Toyohisa Fujita*[1]; Liu Kejun[1]; Atsushi Shibayama[1]; Harunobu Arima[2]; Wan-Tai Yen[2]; [1]Akita University, Fac. of Eng. & Resource Sci., 1-1 Tegata Gakuencho, Akita 010-8502 Japan; [2]Queenís University, Dept. of Mining Eng., Kingston, Ontario K7L 3N6 Canada

The effect of variables on the gold extraction with ammonium thiosulfate was investigated on sponge gold and ore samples. The Effects of $CuSO_4$, $(NH_4)_2S_2O_3$, NH_4OH, $(NH_4)_2SO_4$, pH, stirring speed and retention on gold leaching rate and thiosulfate oxidation (consumption) have been studied. Almost 100% of gold was leached from sponge gold and over 96% from ore sample at optimum conditions. Trioctylmethyl ammonium chloride was used to recover gold from thiosulfate pregnant solution. It was found that more than 99% of gold was recovered by the extractant diluted with n-octane at O/A ratio of 1:1 without the re-adjustment of pH or others. Also, the gold cementation was conducted without de-aeration by using zinc, copper and aluminum powders. The result indicated that the gold was effectively recovered from a solution of low ammonia and copper concentrations and higher thiosulfate concentration. The gold recovery process by using ammonium thiosulfate is an environmental friendly method comparing to conventional cyanidation process.

9:30 AM

Corrosion Behaviors of the Pb-Ag-Ca Anodes for Zinc Electrowinning in Sulfuric-Acid Electrolyte: *Yasushi Takasaki*[1]; Hitoshi Watanabe[1]; Kazuo Koike[1]; [1]Akita University, Fac. of Eng. & Resource Sci., 1-1 Gakuen-cho, Tegata, Akita 010-8502 Japan

In the zinc hydrometallurgical extraction, minimizing of the electric power consumption of the electrowinning process is an important issue for energy saving. At the Zinc & Lead 2000 symposium, the authors suggested that using the Pb-0.5%Ag-0.6%Ca alloy decreased the electrolysis energy compared to the conventional Pb-0.9%Ag alloy. In this study, corrosion behaviors of the Pb-Ag-Ca anodes in sulfuric-acid electrolyte were investigated. Referring to the results for the Pb-0.5%Ag-0.6%Ca anode after 20-days of electrolysis, the bath voltage and the quantity of anode slime were decreased respectively as compared with the Pb-1%Ag anode. Subsequently, beta-PbO2 was detected by XRD and its peaks were sharper comparing to respective ones of the Pb-1%Ag anode. Moreover, dense beta-PbO2 layer on the Pb-0.5%Ag-0.6%Ca anode surface was observed. Therefore considering the results, it was suggested that the dense beta-PbO2 layer of the anode surface was a factor that leads to a lower bath voltage and decreasing quantity of anode slime.

9:55 AM

De-Oiling of Industrial Water Effluents Using Column Flotation: *F. J. Tavera*[1]; R. Escudero[1]; [1]Universidad Michoacana de San Nicol·s de Hidalgo, Inst. de Investigaciones Metal˙rgicas, Santiago Tapia 403, Morelia, Michoac·n 58000 MÈxico

Column flotation technology, at first developed for application in mineral dressing, has received special attention in order to process non-mineral dispersions as a result of its low costs, high efficiency, and relatively simple operation. This paper presents the results from the operation of a laboratory flotation column in order to separate vegetable oil and soap from aqueous industrial effluents. The flotation system was operated under continuous countercurrent conditions, without and with additions of a cationic collector. The experiments have shown that it is possible to separate about 80% of the organic phase in a rougher flotation stage; also, the results in this work suggest that there is an optimum bubble size from which either below or above the recovery of the organic decreases.

10:20 AM Break

10:30 AM Invited

Mechanism of the Electrolysis of Rare-Earth Chlorides in Molten Chloride Bath: *Tsutomu Yamamura*[1]; [1]Tohoku University, Grad. Sch. of Eng., 02 Aoba, Aramaki, Aoba-ku, Sendai, Miyagi Prefecture 980-8579 Japan

Recently, there has been a wide interest in the development of a process for producing rare earths and actinides, and their oxides by a pyro-process using molten salts as operational fluids in nuclear fuel reprocessing. The≈\@electrolytic reduction mechanism of rare-earth chlorides such as LaCl3, CeCl3, NdCl3, SmCl3 and DyCl3 in alkali chloride baths has been investigated in NaCl, KCl and eutectic LiCl-KCl by means of electrochemical transient methods. The thermodynamic behaviors of rare-earth containing species have been discussed in the frame of Electrode potential - pO2- diagram. Low current efficiency found in the case of Nd electro-winning has been attributed to the dissolution of Nd into the bath. The analyses of electrochemical measurement required the elucidation of the effects of the factors such as under-potential deposition, metal dissolution, moisture and oxygen.

11:05 AM Invited

The Interfacial Chemistry of Sulfur in the Pressure Leaching of Sulfide Minerals: *David Dreisinger*[1]; Zhimin Zheng[1]; Jianming Lu[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

The pressure leaching of sulfide minerals has become widely applied for the recovery of base and precious metals. The leaching of zinc sulfide concentrates at 150 ?aC in the Dynatec Zinc Pressure Leach process has been successfully applied commercially for over 20 years. Pressure oxidation of refractory gold ores containing pyrite and other sulfide minerals has been applied commercially since the 1980°¶s at T > 190 ?aC. It is widely anticipated that pressure leaching of copper sulfides and in particular chalcopyrite will be the next widespread application of pressure leaching of sulfide minerals. The behaviour of elemental sulfur and the role of surfactants in the pressure leaching of sulfide minerals have received relatively little fundamental study. Sulfur melts at ?a119 C and it is well known that liquid elemental sulfur will tend to wet sulfides and inhibit leaching reactions from proceeding to completion. Surfactants have been widely used in zinc pressure leaching, pressure oxidation of refractory gold ores and are being advocated for the pressure leaching of chalcopyrite. There are two aspects of the interfacial chemistry of sulfur that have been studied

WEDNESDAY AM

experimentally in a custom built high pressure and high temperature cell. First, the interfacial tension of the liquid sulfur °V aqueous solution system has been studied by photographing droplets of liquid sulfur in solution. The shape of the droplets is then used to calculate the interfacial tension. Second, the contact angle between liquid sulfur and various sulfides has been measured in the presence of various aqueous solutions by photographing droplets of liquid sulfur resting on horizontal mineral specimens. All of these measurements have been performed in the absence and presence of surfactants. In this paper, measurements of interfacial tension and contact angles in the liquid sulfur °V aqueous solution °V mineral system are reported and discussed in relation to current and future applications in zinc, gold and copper pressure leaching.

11:40 AM

Ecologically Safe Technology of Non-Ferrous Metals Obtaining from Sulfide Raw Materials by Means of Bacterial Leaching: *O. V. Slavkina*[1]; N. V. Fomchenko[1]; V. V. Biryukov[1]; [1]Moscow State University of Environmental Engineering, 21/4 Staraya Basmannaya St., Moscow 107066 Russia

The principally new two-stage technological realization of bacterial leaching process by means of bacteria Thiobacillus ferrooxidans is proposed. Optimal conditions for main electrochemical reactions of sulfides oxidizing must be created for the first stage (the active oxidizing agent supply and reaction products withdrawal). The active concentrate subdivision in two fractions (easy and hard oxidizable) happens here. Then these fractions are to be separated and hard oxidizable fraction is returning to the first stage or can be removed from the process. Easy oxidizable fraction is transferring to the second stage of bio leaching. The first stage duration and the suspension separation procedure are to be calculated according to developed method for every processing concentrate. The second stage of bio-leaching shall be carried-out under high activity of microorganisms. Such conditions are contributing to most complete non-ferrous metals transferring into solution and sulfide sulfur transformation into elemental sulfur with its partial oxidation to sulphate-ion. In addition, the sediment, containing mainly ferric iron, sulphate-ions and hydroxide-ions is forming on the second stage. The quantity of sediment is depending on the processing concentrate and pH at the second stage of bio-leaching process. At the same time the ferric iron equilibrium concentration in solution is determined by these parameters. Ferric iron, being an active oxidizer in bio-solution at optimal pH is returning to the first stage of bio-leaching. Process realization according to proposed technological strategy makes possible to achieve higher rates of bacterial leaching in comparison with traditional one-stage process. At the same time two-stage bio-leaching process is in rather good accordance with standard methods of non-ferrous metals recovery from solutions. It is advisable to organize the stage of non-ferrous metals recovery between the first and the second stage of bacterial leaching. Such way permits to obtain selective sediments of non-ferrous metals while the iron (in the form of the ferrous sulphate) is stayed in the solution.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: Copper II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday AM	Room: Santa Rosa
March 5, 2003	Location: San Diego Marriott Hotel

Session Chairs: Florian Kongoli, FLOGEN Technologies Inc., Matls. Tech. Dept., Montreal, Quebec H3S 2C3 Canada; Yasuo Ojima, Sumitomo Metal Mining, Toyo Smelter & Refinery, Saijo, Ehime 793-0005 Japan

8:30 AM Keynote

Future of Copper Converting Process: *Yasuo Ojima*[1]; [1]Sumitomo Metal Mining, Toyo Smelter & Refinery, Otu 145-1 Funaya, Saijo, Ehime 793-0005 Japan

The mainstream of copper smelting process in the world is the combination of Outokumpu type Flash smelting and PS converting process. Presently it will be estimated that in the world copper production about 50% is produced by Flash smelting process and 90% is by PS converting process. PS converting process has been widely used for many smelters more than 100 years owing to its operational flexibility in spite of the batch operation. However, an environmental issue has been pointed out because of the difficulty of fugitive gas handling. Recently several copper smelters in the world have been newly constructed and executed the major modification and modernization. Continuous converting process has emerged some of them instead of PS converting process. This paper describes the comparison of PS and emerging continuous converting process, and the prospect of the future of copper converting process.

9:00 AM

Efficiency of Porous Plugs in Fire Refining of Crude Copper: *C. M. Acuna*[1]; M. Sherrington[2]; [1]Codelco, Chuquicamata Div., Chuquicamata Chile; [2]Instituto Nacional de CapacitaciÛn, INACAP, Calama Chile

In the fire refining of crude copper the use of porous plugs, by stirring with an inert gas, has been mainly considered as a surface renewing agent to increase product quality and/or shortening the refining cycle. However its application is rather limited, specially because of different industrial practices as well as insuficient data on its aside effects on the refractory lining. Furthermore, the combined stirring-reaction mechanism has to be clarified. The standard procedure in fire refining consist of sulfur removal via air injected through tuyeres, arsenic reduction via soda-lime flux and oxygen control by substoichiometrical air or steam fuel mixtures. In the present study the effect of bottom stirring by use of porous plugs and nitrogen gas injection in the treatment of a dirty crude copper, in the range 400-150 ppm sulfur, approximately 2,500 ppm arsenic and 14,000-9,000 ppm oxygen, was investigated. The combined effect stirring-reducing air/steam:fuel mixtures follows a kinetics of order one where the kinetics constant can be represented by a polynomial of the form: K = A * R2 + B * R + C with K as the kinetics constant, A, B and C as constants depending on the stirring nitrogen flow rate and R as the ratio air-fuel or steam-fuel used in the reducing step. If just the oxidation, complexing and reducing steps are considered the process time may be shortened by 30%-40%, depending on the optimization of the air-fuel or steam-fuel mixture used.

9:25 AM Invited

ISASMELTñAn Update on Latest Developments: Philip Arthur[1]; Britt Butler[1]; James Edwards[1]; *Chris Fountain*[1]; Simon Hunt[1]; Philip Partington[1]; Jorma Tuppurainen[1]; [1]Mount Isa Mines, Ltd., Brisbane, QLD 4000 Australia

The Isasmelt process is making a significant contribution to the global metals industry. Isasmelt furnaces are now operating successfully in eight different countries and treating approximately 3 million tonnes of concentrates and secondary materials per year. The process is currently used in both lead and copper smelting. The most recently commissioned plants were in Germany and China, one for treating scrap copper and the other treating low-quality copper concentrates. The Copper Isasmelt furnace at Mount Isa Mines continues to set new operational records, with throughput of more than 1 million tonnes of concentrate per year and very low operating costs. This paper summarises the latest operational data from the Isasmelt plants and introduces plans for the latest process enhancement, a replacement for Peirce Smith Converters.

9:50 AM

Development of Sumitomo Premixed Concentrate Burner for Copper Flash Smelting: *Yasumasa Hattori*[1]; Yoshiaki Mori[1]; Yasuhiro Kondo[1]; Yukihito Sasaki[1]; Toyokazu Okubo[1]; Kozo Baba[1]; [1]Sumitomo Metal Mining Company, Ltd, Niihama Rsrch. Labs., 17-5, Isoura-Cho, Niihama, Ehime 792-0002 Japan

An extensive study including fluid dynamic calculation, cold model tests, pilot plant tests as well as measurements in a concentrate burner of a commercial flash furnace was carried out in order to develop a new Sumitomo flash smelting concentrate burner. The pressure measurements in the commercial burner indicated that the premix of concentrate and reaction air in the burner was insufficient. Pilot plant tests exhibit that the enhancement of the premix of concentrate and reaction air is very effective to improve burner performance. Using the additional pressure drop of the reaction air as an indicator of the premix, a new type Sumitomo premixing concentrate burner was developed.

10:15 AM Break

10:25 AM
Effect of the Oxygen Potential on the Viscosity of Copper Smelting Slags and its Relation to the Liquidus Surface: *F. Kongoli*[1]; I. McBow[1]; S. Llubani[1]; [1]FLOGEN Technologies, Inc., Matls. Tech. Dept., 5757 Decelles, Ste. 511, Montreal, Quebec H3S 2C3 Canada

Viscosity is an important parameter of copper smelting and converting processes. It affects the settling of matte or metal droplets in the slag, distribution of elements, etc. Despite this importance, considerable confusion exists on the viscosity of copper smelting slags as a result of many disagreements found among the measured viscosity data of several authors especially in the slags of high ferric iron content and close to magnetite precipitation. In this work the effect of oxygen potential on the viscosity of copper smelting slags has been quantified through a coupled quantification of viscosity and liquidus temperature. The confusion in the literature has been clarified and the importance of the relation between the viscosity and the liquidus temperature is discussed.

10:50 AM Invited
Two Copper Smelting Processes at Onsan: *In-Ho Song*[1]; Young-Chul Kang[1]; [1]LG-Nikko Copper, Inc., Daejung-Ri 70, Ulju-Gun, Onsan-Eup, Ulsan City 689-892 Korea

The Onsan flash smelter of LG-Nikko Copper Inc. was commissioned at the capacity of 80,000tpy from copper concentrate in 1979 and its capacity was increased up to 164,000tpy through the two timesí expansion in 1988 and 2001. As the Onsan smelter was started Mitsubishi continuous process in 1998, it has two processes within one plant. This paper outlines two processesí difference and operating dada, i.e., process flow, feed materials, output product quality, impurity distribution, man power, dust generation ratio, power consumption, productivity, steam production, etc.

11:15 AM
High-Intensive Operation of Flash Smelting Furnace at Saganoseki Smelter & Refinery: Masatoshi Ogasawara[1]; *Toshihiro Kamegai*[1]; Masatoshi Maeda[2]; [1]Nippon Mining & Metals Company, Ltd., Saganoseki Smelter & Refinery, Saganoseki-machi, Oita 879-2201 Japan; [2]Metal Economics Reseach Institute, Japan, Mori Bldg. 11, 2-6-4 Toranomon, Minato-ku, Tokyo Japan

Saganoseki smelter & refinery operated two flash smelting furnaces, producing a combined 330,000 metric tons per year (hereinafter mtpy) of copper. In 1996, however, it successfully shifted to single furnace operation while maintaining production at the same level. Since then, the production capacity has been further increased to the present 450,000 mtpy by technological improvements. Also, in order to reduce the converter load which represents a bottleneck in efforts to increase production, the matte grade was gradually increased and currently runs at 65 to 66% attaining the low slag loss of 0.7 to 0.8%. This paper introduces some improvements, recent operation results and thermodynamic analyses conducted.

11:40 AM
The Copper Loss in Slag of Flash Smelting Furnace in Tamano Smelter: *Tsuneo Maruyama*[1]; Nobuyuki Furui[1]; Makoto Hamamoto[1]; [1]Hibi Kyodo Smelting Company, Ltd., No 6-1-1 Hibi, Tamano City, Okayama 706-8511 Japan

In 1972, the Tamano Smelter of Hibi Kyodo Smelting Co., Ltd. went into operation. The Flash Smelting Furnace, used at the Tamano Smelter, was equipped with electrodes that were attached to the settler substitute of the slag cleaning furnace. After that we promoted the development of our own original coke combustion technology. A new Tamano Type Flash Smelting Furnace (T-FSF), without electrodes, was put into operation in 1988. We have continued to improve the technology of coke combustion and combustion the burner. As a result, we have been able to reduce the copper loss in slag as well as achieve stable operation. Furthermore, we have been able to accomplish this while increasing the concentration feed and matte grade. We will continue to strive for high matte grade operation and to raise production efficiency, while paying attention to the important subject of copper loss in slag. In this report, we will present the detailed data with regards to the copper loss in slag reduction, as well as of the influence of the thickness, settling time and coke. We would also like to report the recent operation conditions of the T-FSF.

Notes

15th International Symposium on Experimental Methods for Microgravity Materials Science - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee

Program Organizers: Robert Schiffman, R. S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica & Instituto Nazionale di Fisica della Materia, Bologna 40127 Italy

Wednesday PM March 5, 2003 — Room: 10 — Location: San Diego Convention Center

Session Chair: William H. Hofmeister, Vanderbilt University, Cheml. Eng., Nashville, TN 37235 USA

2:00 PM

Spherule Formation of Meteorites on Microgravity Condition by Collision: *Yasunori Miura*[1]; [1]Yamaguchi University, Fac. of Sci., Inst. of Earth Scis., Yoshida 1677-1, Yamaguchi 753-8512 Japan

New type of spherules with high pressure and high temperature is found at dynamic reaction with ultra-high velocity materials of meteoroidal parent body during atmospheric passing. Various types of spherules and melt fragments at meteorite falling site in rice-field after 104 years are found at Niho chondritic meteorite, Yamaguchi, Japan. Direct Simulation of Monte Carlo (DSMC) model can explain dynamic reaction of meteoritic projectile to produce various spherules and melt fragments during atmosphere of the Earth by dynamic fluid reaction under microgravity condition.

2:20 PM

Conduction-Limited Melting in Microgravity: *Afina Lupulescu*[1]; Martin E. Glicksman[1]; Matthew B. Koss[2]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng., 110 8th St. (CII-4223), Troy, NY 12180-3590 USA; [2]College of the Holy Cross, Physics Dept., PO Box 143A, Worcester, MA 01610-2395 USA

Conduction-limited melting is of importance in convection-free processes in low-Earth orbit, during mushy zone fusion, and in weldingñall examples where the length scales for thermal buoyancy are highly restricted. Steady-state crystal growth data reported earlier indicate that dendritic growth under microgravity conditions occurs by pure thermal conduction. We now report on melting process, observed for the first time using video images, where both freezing and melting sequences for pivalic acid (PVA) were observed. PVA dendrites generally melt in a stable manner following an accelerating square-root-of-time dependence. The theoretical kinetics against which these experiments are compared is based on quasi-static kinetic analysis for melting under shape-preserving conditions. Comparison between theory and experiment yield Stefan numbers (dimensionless superheating) in good agreement with thermal data telemetered from the space-borne thermostat. The experiments and their analysis raise several new questions concerning the roles of capillarity, kinetics, and convection during melting processes.

2:40 PM

Impact Macrostructures in the South West Egyptian Region: *E. Farabegoli*[1]; G. Onorevoli[1]; C. Patuelli[2]; R. Serra[2]; [1]Universit‡ di Bologna, Dipartimento di Scienze della Terra e Geologico-Ambientale, Piazza di Porta S. Donato 1, Bologna 40126 Italy; [2]Universit‡ di Bologna, Dipartimento di Fisica ed Istituto Nazionale di Fisica della Materia, Viale Berti Pichat 6/2, Bologna 40127 Italy

The area in the South West region of Egyptian Great Sand Sea was analysed by remote sensing using multi-spectra images in order to investigate the impact structures. The research was focused on old empty craters, old craters filled with alluvial-lake sediments and on elongated linear structures by abrasion on the rocky substrate. The results are discussed taking into account the recent impact or volcanic theories.

3:00 PM

The Measurement of Precise Solute Diffusion Coefficients in Molten Metals and Semiconductors: The Effect of Gravitational Fields: *Reginald William Smith*[1]; Weidong Huang[1]; B. J. Yang[1]; [1]Queenís University, Matls. Sci. of Microgravity Applic., Nicol Hall, Rm. 228, Kingston, Ontario K7L 3N6 Canada

This paper will describe the development and characterisation of a new shear cell for use with the Advanced Thermal Environment Facility(ATEN) being developed for the ISS by the Canadian Space Agency.

3:20 PM Break

3:40 PM

The Effects of Improved Heat Extraction on the Microstructure of Directionally Solidified Immiscible Alloys: *J. V. Puckett*[1]; J. B. Andrews[1]; [1]The University of Alabama-Birmingham, Microgravity Solidification Lab., 1150 10th Ave. S., BEC 361, Birmingham, AL 35294 USA

Immiscible alloy systems that possess a high-miscibility gap can generally be solidified under coupled growth conditions to produce an aligned, fibrous microstructure. However, under certain processing conditions, rows of aligned spheres are more likely to form in some alloys, instead of the more desirable fibrous microstructure. Speculative theories about the mechanisms leading to this aligned droplet structure include Rayleigh breakdown of the liquid ìfibersî into droplets near the solidification front and oscillatory variations in solidification rate that cause variations in fiber diameter that facilitate the breakdown of fibers into doubly orientated arrays of droplets. This investigation focuses on the influence of efficient heat extraction during solidification on the tendency of samples to form droplets as opposed to fibrous microstructures by virtue of reducing the time that is available for cylindrical instabilities or ripening to occur after primary solidification. Microstructures corresponding to different heat extraction rates as well as the influence of processing alloys slightly off monotectic composition will be presented.

4:00 PM

The Influence of Gravity on the Solid-Liquid Interfacial Free Energy: *Reginald William Smith*[1]; C. Patuelli[2]; R. Tognato[2]; [1]Queenís University, Matls. Sci. of Microgravity Applic., Nicol Hall, Rm. 228, Kingston, Ontario K7L 3N6 Canada; [2]Universita di Bologna, Dipto. di Fisica & Inst. Nazionale di Fisica della Materia, Alma Mater Studiorum, Bologna 40127 Italy

Theoretical analysis suggests that the free energy of a solid/liquid surface, and hence the local surface tension, may be influenced by the gravitational field. This paper reports recent experimental work which attempts to establish the physical significance of this analysis.

4:20 PM

Investigation of Libyan Desert Glass by X-Ray Micro-Diffraction Analysis: *C. Patuelli*[1]; R. Serra[1]; S. Coniglione[1]; M. Chiarini[2]; [1]Universitaí di Bologna, Dipartimento di Fisica ed Istituto Nazionale di Fisica della Materia, Viale Berti Pichat 6/2, Bologna 40127 Italy; [2]INFM Unit‡ di Bologna, Via Gobetti 101, Bologna 40129 Italy

Samples of Libyan Desert Glass (LDG) collected by Serra et al. (1) in the South West region of Egyptian Great Sand Sea were analyzed by X-ray micro-diffraction technique. A short order crystal structure was observed using a well-collimated X-ray Cu k beam of 30 m in diameter and interesting FCC metal alloys phases were identified by matching the whole LDG spectrum. The matched phases confirm that this natural tektite glass has an extraterrestrial origin. The important role played by micro-gravity environment during melting and solidification of this nearly pure silica (98% SiO2) is taken into account and it is advanced the hypothesis that SiO2 was still present in the comet core before the impact. BARAKAT A. A., DE MICHELE V., NEGRO G., PIACENZA B. and SERRA R. (1997). Some new data on the distribution of Libyan Desert Glass (Great Sand Sea, Egypt) in Proc. ′ Silica 96 ª Meeting on Libyan Desert Glass and related desert events. Bologna University, July 18 1996. Proceedings. Segrate (Milano), Pyramids, 29-36.

Actinide Materials: Processing, Characterization, and Behavior: Advanced Fuels and Materials II

Sponsored by: Light Metals Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS), LMD-Reactive Metals Committee

Program Organizers: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA; Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Wednesday PM March 5, 2003 — Room: 4 — Location: San Diego Convention Center

Session Chair: Sean M. McDeavitt, Argonne National Laboratory, Cheml. Tech. Div. Matls. Dvlp. Sect., Argonne, IL 60439-4837 USA

2:00 PM
Production of Urania/Thoria Microspheres by Spray Drying: *Alvin Solomon*[1]; S. Anthonysamy[1]; S. Kuchibhotla[1]; [1]Purdue University, Sch. of Nucl. Eng., W. Lafayette, IN 47907 USA

Spray drying has been used to produce (U,Th)O2 fuel microspheres for dispersion fuels. This methodology avoids the problem of non-recyclable waste streams, but the recyclable dry ìfinesî must be collected and controlled. A facility has been built for performing the spray drying using a commercial labscale spray dryer with an upward-facing two fluid nozzle and counter flow heated air. Dryer modifications include a vessel extension to increase sphere size, and a remote cleaning system. Safety is assured because the system operates at negative pressures during operation or automatically shuts down. The fines are collected in a sump/filtration system. Optimized slurries were obtained by controlling the Zeta Potential, slurry viscosity and rheology. Spray drying runs were carried out using 25 to 30% solids loading with various percentages of triethanolamine as dispersing agent and binder. Spheres of ~90%TD and a modal size of 200 microns were obtained with uniform porosity.

2:25 PM
Precursor Selection for Chemical Vapor Deposition of Niobium on Zirconia Microspheres: *Christine T. Snyder*[1]; Jude M. Runge[1]; Thomas C. Carter[1]; Andrew S. Hebden[1]; Bryce W. Campbell[1]; Sean M. McDeavitt[1]; [1]Argonne National Laboratory, Chem. Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA

As part of the effort to study non-fertile nuclear dispersion fuel containing (Pu, Am, Np)O2 for actinide transmutation systems, a chemical vapor deposition (CVD) method is under development for coating non-radioactive zirconia microspheres with niobium. In the CVD process, the solid film results from the decomposition and reaction of the chemical constituents of a precursor, or starting material. Precursor selection is one of the crucial steps in avoiding nucleation of solid particles that can result in films containing impurities, defects, particulates, as well as poor adhesion. In this investigation, precursor selection was evaluated using the following criteria: 1) chemistry of reaction (intermediate steps, by-products), 2) volatility and temperature of vaporization, 3) composition and stability at room temperature, and 4) hazards and toxicity.

2:50 PM
Compatibility of U-Mo Alloys and Al in RERTR Dispersion Fuels: *Dennis D. Keiser*[1]; Curtis C. Clark[1]; Mitchell K. Meyer[1]; [1]Argonne National Laboratory, Eng. Tech. Div., PO Box 2528, Idaho Falls, ID 83403-2528 USA

Argonne National Laboratory is developing fuels to convert reactors that employ fuels containing highly-enriched uranium to fuels that contain low-enriched uranium (less than 20% 235U). This work is being done as part of the Reduced Enrichment for Research and Test Reactors program (RERTR). The leading candidate fuel for this conversion is a plate-type fuel that has a fuel meat comprised of U-Mo alloy powders dispersed in an Al matrix. This fuel meat is clad in Al. When these dispersion fuels are irradiated in-reactor, reaction occurs between the Al matrix and the U-Mo alloy powders. As a result, out-of-reactor diffusion studies have been performed to investigate the interdiffusion behavior of U, Mo, and Al. This paper will discuss the results from diffusion tests that employed solid-solid diffusion couples that were annealed at temperatures from 400∞C to 640∞C for varying times. Couples were examined for diffusion structure development using a scanning electron microscope equipped with both energy-dispersive and wavelength-disepersive spectrometers (SEM/EDS/WDS). Point-by-point and linescan analysis was used to generate composition profiles and diffusion paths. The interdiffusion behavior of the various elements and development of interdiffusion zones will be discussed. Comparisons will be made to structures observed for fuels irradiated in a reactor.

3:15 PM
The Morphology and Structure of Annealed U-Nb-W Alloys: *E. Kahana*[1]; M. Talianker[1]; A. Landau[2]; A. Venkert[2]; [1]Ben-Gurion University of the Negev, Dept. of Matls. Eng., PO Box 653, Beer-Sheva 84105 Israel; [2]Nuclear Research Center-Negev, PO Box 9001, Beer-Sheva Israel

Dilute uranium alloys with small additions of the second transition metal solute such as molybdenum, niobium, titanium and zirconium, have been the subject of extensive investigations. In general, these alloying additions, extensively soluble in the high temperature uranium γ phase, are almost completely insoluble in the low temperature a phase. Therefore, on slow cooling rate the γ phase decomposes via diffusional phase transformation forming a two-phase structure of a phase and either an intermetallic phase or an alloy rich γ_o phase. This work presents the effect of addition of tungsten as a second solute element, on the structures formed in annealed ternary U-14at%Nb-W alloys. The solubility of tungsten in uranium is extremely limited, less than 1 at%, and there is no intermetallic compound in the uranium-tungsten system, however, there is full solubility of tungsten in niobium. The niobium content in the alloys was chosen near the eutectoid composition of U-Nb system. The range of the tungsten content was between 0.6at%W to 2.3at%W. In common with other U-Nb base alloys it was found that the microstructure of annealed U-Nb-W alloy consists of two main phases: orthorhombic α-uranium and γ_o tetragonal phases that form as a result of eutectoid reaction. However, in addition to these expected phases, the existence of dendrites and small particles containing U-Nb-W were found. Longer annealing periods resulted in the formation of only one type of small precipitates of a new ternary U-Nb-W phase. The composition of this phase as determined by EDS analysis in TEM was Nb=49.75at%, W=45.71at%, U=4.54at%, and the unit cell was identified as cubic BCC with the lattice parameter a=3.185≈.

3:40 PM Break

4:00 PM
Testing of Commercial Materials for Melt Containment of Uranium-Bearing Metal Waste Form: *Ken C. Marsden*[1]; [1]Argonne National Laboratory-West, PO 2528, Idaho Falls, ID 83403 USA

Electrometallurgical processing of metal fuel rods at ANL-W generates chopped stainless steel cladding coated with process salts and containing small amounts of residual uranium. This waste stream will be melted and alloyed with zirconium to form a dense, corrosion resistant waste form. The resulting alloy is approximately SS304-15Zr-7U and is formed by melting near 1600C. The waste form is allowed to solidify in the crucible. Development testing has proceeded in several furnaces to explore feasibility of commercially available materials. The materials problem is complicated by the presence of reactive chloride species in the process salt. Various commercially available materials have been explored, including coated graphite, yttria, boron nitride, and alumina. Tests have been conducted at charge masses between 3 and 69 kg. An inexpensive alumina refractory displays good resistance to the process when coated with a thin oxide coating.

4:25 PM
The Mechanical Response of Depleted Uranium as a Function of Strain Rate and Temperature: *Carl M. Cady*[1]; Shuh-Rong Chen[1]; George T. (Rusty) Gray[1]; David F. Teter[1]; Dan J. Thoma[1]; Gary K. Lewis[1]; Deniese R. Korzekwa[1]; Philip K. Tubesing[1]; Ann M. Kelly[1]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA

The compressive stress-strain response of several wrought depleted uranium (DU) ingots as a function of temperature and strain rate was investigated. The yield and flow stresses of DU was found to exhibit a pronounced sensitivity to strain rate, temperature and carbon content. The stress-strain response of uranium under quasi-static uniaxial tension will be compared to the compression data to show the sensitivity of stress-state. Constitutive modeling efforts are underway to predict the mechanical behavior of materials outside the regions where data for strain-rate and temperature already exists. The Mechanical Threshold Strength Model can accurately capture the constitutive response of DU. The Taylor impact test was used to validate the model and show how well the model fit works even outside the region where experimental data exists.

4:50 PM
Modern Day Alchemy: Development of Ceramic Fuels for the Transmutation of Nuclear Waste: *Ryan F. Hess*[1]; R. Margevicius[1]; K. McClellan[2]; M. Stan[2]; K. Sickafus[2]; S. Voit[1]; E. Henderson[2]; G. Egland[2]; [1]Los Alamos National Laboratory, NMT-11, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

Transmutation fuels work at Los Alamos National Laboratory is currently focused on mononitride ceramic fuel forms, and consists of closely coordinated actinide (hot) and inert and surrogate (cold) fuels work. This work involves three major components: 1) fuel material synthesis and fabrication, 2) fuel performance, and 3) fuel materials modeling. Results from all three components will be presented. The cold work focuses on the synthesis and ceramic processing of the proposed ZrN fuel matrix. The actinide (hot) fuels effort at Los Alamos emphasizes the synthesis and fabrication of actinide-bearing nitride fuel pellets. These pellets will be inserted into the Advanced Test Reactor early in 2003 and continue for the next couple of years. The nitride pellets are designed to contain varying amounts of Pu, Am, Cm, and Np. Results presented will outline fabrication techniques which are designed to reduce the volatility of americium.

Aluminum Reduction Technology: Advanced Processes

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Martin Segatz, VAW Aluminum Technology, Bonn D-53117 Germany; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday PM Room: 6B
March 5, 2003 Location: San Diego Convention Center

Session Chair: Harald Oye, Norwegian University of Science and Technology, Dept. of Chem., Trondheim N-7491 Norway

2:00 PM
Aluminum Carbothermic Technology: Comparison to Hall-Heroult Process: *Marshall Bruno*[1]; [1]Alcoa Inc., Next Generation Al. Processes, Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069-0001 USA

Production of aluminum by carbothermic reduction has been investigated by every major aluminum company over the past forty seven years. As an alternative to the Hall-Heroult process, carbothermic offers potential energy, cost and environmental advantages if solutions to critical technical hurdles could be developed. These include efficient delivery of energy to attain 2000-2200C, while minimzing loss of volatile aluminum-containing species; capture and recycle of those volatiles that do evolve to recover mass and energy values; effective decarbonization of the Al-C metal phase; and recovery of energy values in the by-product CO to reduce net energy consumption. To address these hurdles, Alcoa and Elkem, with support from DOE, initiated development of a new Advanced Reactor Process. Previous efforts by the industry will be reviewed; estimates will be given for energy, cost and environmental benefits from the new process; and process challenges will be identified.

2:25 PM
Aluminum Carbothermic Technology: Alcoa-Elkem Advanced Reactor Process: *Kai Johansen*[1]; Jan A. Aune[2]; Marshall Bruno[3]; Anders Schei[4]; [1]Elkem ASA Research, PO Box 8040 Vaagsbygd, Kristiansand N-4602 Norway; [2]Elkem Technology a/s, PO Box 4376, Torshov , Oslo N-0402 Norway; [3]Alcoa Inc., Next Generation Al. Processes, Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069-0001 USA; [4]Consultant, Formerly of Elkem ASA Research, Kristiansand N-4602 Norway

Alcoa and Elkem are jointly developing the Advanced Reactor Process for production of aluminum by carbothermic reduction of alumina. The new process is based on a continuous, staged, ultra high intensity electric slag resistance reactor utilizing Elkemís state-of-the-art high temperature smelting technology. The reactor design meets the specific process requirements for aluminum as defined by Alcoa, based on the thermodynamic properties of the Al-O-C system, an operating diagram of the chemistry involved, and fundamental studies of vapor recovery parameters including computor modeling, conducted with Carnegie Mellon University. The thermodynamics will be reviewed; the process will be described; progress and future program will be reported.

2:50 PM
A Finite Element Computational Fluid Dynamics Sensitivity Analysis for the Conceptual Design of a Carbothermic Aluminium Reactor: *Dimitrios I. Gerogiorgis*[1]; B. Erik Ydstie[1]; [1]Carnegie Mellon University, Dept. of Cheml. Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Carbothermic reduction is a nontraditional chemical process for aluminium production, based on the endothermic chemical reduction reaction occurring between aluminium oxide and carbon. This process has potential for drastic reduction of fixed and operation costs of the investment. Furthermore, it is environmentally benign and in principle significantly more energy-efficient, as the costly electrolytic ionization is effectively avoided by the direct chemical reduction pathway. This method is identified as a potential alternative to Hall-HÈroult electrochemical reduction by several studies, but its complexity still poses remarkable technical obstacles for implementation. A conceptual multistage electrothermic reactor recently proposed (Johansen, Aune et al., 2000) is a quite attractive idea for achieving reactor scaleup but entails significant design challenges. Actual carbothermic reduction of alumina occurs in the second stage of this multiphase reactor: a high-temperature molten slag formed at a temperature of ca. 1950∞C (1st stage) is fed into a submerged arc furnace (2nd stage), where it reacts at a temperature of ca. 2050∞C to CO and Al. Temperature and flow control are crucial in achieving high yield and minimal Al evaporation. The present study elaborates on a finite element CFD model developed for the conceptual reactor (Gerogiorgis et al., 2001), considering only a reactor sector to minimize computational expense. The rectangular computational domain comprises a cylindrical electrode and symmetry planes, and a pseudohomogeneous molten slag is assumed, so as to relax multiphase flow complications. Appreciable modeling complexity is caused by the interaction and nonlinear couplings among the Joule electrothermic phenomenon within the electric field between the inert DC electrodes, the endothermic reduction reaction (modeled by considering Arrhenius-type heat consumption) and the Boussinesq natural convection that is inducing a vertical recirculation of the molten slag. The complete triple-PDE problem (electric charge balance, heat balance and momentum balance) is solved using a commercial, modular PDE modeling suite (FEMLABÆ v. 2.2-COMSOL) in order to obtain the potential, temperature and velocity distributions, respectively, and thus probe the combination of Joule heating, endothermic reaction and Boussinesq natural recirculation. The main objective of the present CFD simulation study is to extract reactor design conclusions by explicitly addressing the effect of process parameters on the temperature and velocity fields. A sensitivity analysis is thus being conducted with respect to a number of design variables (reactor dimensions, electrode length, imposed voltage); profiles obtained reveal the nontrivial optimization problem arising when sufficient heating is to be maintained at adequate circulation. This delicate balance is obvious in preliminary experimental observations of a pre-pilot plant and is believed to govern Al production rates; thus, it is quintessential to feasibility and profitability.

3:15 PM Break

3:25 PM
An Improved Pyroconductivity Test of Spinel-Containing Cermet Inert Anodes in Aluminum Electrolysis: *Yanqing Lai*[1]; Qingwei Qin[1]; Zhongliang Tian[1]; Gang Zhang[1]; *Jie Li*[1]; Yexiang Liu[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

An improved pyroconductivity test device, consisting of a specially constructed closed furnace and a potentiostat, was constructed based on the conventional direct current four-point technique. Symmetrical current distribution in the specimen was obtained by keeping a fixed pressure and good contact between the specimen and clamps at any temperature. The potentiostat was used to supply direct current and continuously monitor the current intensity and voltage between two probes, which can be adjusted outside the heating furnace to well contact with the specimen. Test results of copper and graphite specimens show that the reliability and reproducibility were excellent. The electrical conductivity as a function of temperature for various spinel-containing cermet inert anodes was investigated spanning the Hall cell operating temperature. The factors influencing the electrical behaviour were studied, which included the particle size of row materials, manufacture process, phase composition and morphology et al.

3:50 PM
On Expansion of TiB2-Carbon Composites Used as Wettable Cathode in Aluminum Electrolysis Due to Sodium Penetration: *Qingyu Li*[1]; Yanqing Lai[1]; Jianhong Yang[1]; Jie Li[1]; Hengqin Zhao[2]; Yexiang Liu[1]; [1]Central South University, Sch.of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China; [2]Zhengzhou Institute of Multipurpose Utilization of Mineral Resources, CAGS, No. 328, Longhai W. Rd., Zhengzhou, Henan 450006 China

The current Hall-Heroult process could be renovated by introduction of new cell design based on the TiB2 wettable cathode, inert anode and other materials. In the present work, a convenient method for the preparation of the TiB2-Carbon Composites cathode materials composed of TiB2 thermosetting resins and carbon was studied by means of the cold press forming and sintering. The relations between the sintering temperature or the TiB2 content and sodium penetration into the composite were measured. The sodium penetration into the composite was much less than that into the current carbon materials. The increase of sintering temperature and the TiB2 content was beneficial to reduce sodium penetration and expansion of the composite resulted from sodium penetration.

4:15 PM
Modeling of the Solubilities of NiO/NiAl2O4 and FeO/FeAl2O4: Yunshu Zhang[1]; Xiaoxia Wu[1]; *Robert A. Rapp*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Based on the literature data, combined with a number of solubility data measured by the present authors, the solubility of $NiAl_2O_4$ in

WEDNESDAY PM

Al_2O_3-saturated cryolite melts at 1300K were modeled thermodynamically in terms of two Ni-containing acidic solutes Na_2NiF_4 and Na_4NiF_6 over a wide composition range of 2 ≤cryolite ratio r ≤12. Equilibrium constants and ΔG o/f values for the formation of these two solute species were thereby estimated. This two Ni-containing solute model provides an essentially perfect match with the experimental solubility values. The solubilities of $NiO/NiAl_2O_4$ in Al_2O_3-undersaturated cryolite melts in dependence upon dissolved Al_2O_3 concentration were calculated from the present model and plotted for a number of melt compositions. Similarly, the solubilities of $FeO/FeAl_2O_4$ in cryolite melts with dissolved Al_2O_3 at 1300K were modeled in terms of Fe-containing solute complexes.

4:40 PM

Result of Aluminum Electrolysis Test of Minitype Cermets Anode: *Qun Zhao*[1]; Yanli Xie[1]; Zhuxian Qiu[1]; [1]Northeastern University, Nonferrous Dept., 3 Ln., 11# Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China

A minitype NiFe2O4 based cermets was tested in low temperature electrolyte. Test cells operated under the following conditions. Anode current density was set at 1 A cm-1 and the current was kept at 22A accordingly. Alumina was added in bath every 1 hour, 5g for each time. Electrolysis was conducted at a temperature of 850° and last 11 hours. Gas ejection from the surface of anode was observed and a few amount of metal was obtained. The cracks were formed in the bottom of anode after test and problems were existed at electricity resource and anode joint. With EPM-810 type microscope, changes of microstructure and element distribution in cermets anode were studied and the corrosion process of anode during electrolysis was discuss.

Carbon Technology - IV

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Amir A. Mirchi, Alcan Inc., Arvida Research and Development Centre, Jonquiere, QC G7S 4K8 Canada; Don T. Walton, Alcoa Inc., Wenatchee Works, Malaga, WA 98828-9784 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday PM — Room: 6D
March 5, 2003 — Location: San Diego Convention Center

Session Chair: Philippe Beghein, SGL Carbon Group, Business Line Cathodes Techl. Serv., Passy 74190 France

2:00 PM

Operational Changes in Carbon Cathode of Aluminum Cell: *S. M. El-Raghy*[1]; M. O. Ibrahiem[2]; F. M. Ahmed[2]; H. A. Ahmed[1]; [1]Cairo University, Fac. of Eng., Cairo Egypt; [2]Aluminium Company of Egypt, Naga Hammad Egypt

Since its start, 1975, Aluminium Company of Egypt, EgyptAlum, is using two types of anthracite cathode blocks. Graphite content of these cathodes were either 20% or 50%. This paper is reporting EgyptAlumís experience over these years with respect to structural changes in the carbon cathode during operation. Graphitization, oxidation of carbon cathode as well as sodium and ash content have been studied as a function of cell life over 10 years for the two types of the cathode blocks mentioned above. The exothermic peak of carbon block, by DTA, decreased with increasing cathode life. Oxidation temperature of new carbon sample is 680°C while it comes down to 620°C and 615°C for samples of 1 and 4 years life respectively. Electrical resistivity decreased from 41.5 to 25 µohm.m for cathode blocks after 10 years service. The initial graphite content proved to have some influence on structural changes during operation.

2:25 PM

Multiaxial Mechanical Behavior of the Carbon Cathode: Understanding, Modeling and Identification: *Guillaume DíAmours*[1]; Mario Fafard[1]; Augustin Gakwaya[1]; Amir A. Mirchi[2]; Olivier Doucet[1]; [1]Laval University, Sci. & Eng. Fac., Adrien-Pouliot Bldg., Sainte-Foy, QuÈbec G1K 7P4 Canada; [2]Alcan Inc., Arvida R&D Ctr., PO Box 1250, JonquiËre, Quebec G7S 4K8 Canada

As part of the on-going project START-Cuve, involving the thermo-electro-mechanical modelling of a cell coke bed electrical preheating, a new mechanical constitutive model has been developed for the simulation of the multiaxial behavior of carbon cathode during preheating and heat-up of aluminum electrolysis cells. This model will be able to calculate strains and stresses that are generated in the cathode during thermally induced expansion of the lining. This paper presents the mechanical development of the model who was guided by recent experimental observations on laboratory specimens which were loaded under direct tension, uniaxial compression, shear and multiaxial compression field at different levels of lateral confinement. All tests have been done at ambient temperature. A single surface plasticity model was formulated to account for the dependence of both strength and ductility of carbon under confinement. The constitutive parameters of the model have been calibrated from the experimental results.

2:50 PM

Performance of Graphitized Carbon Cathode Blocks: *Andreas Schnittker*[1]; Holger Nawrocki[1]; [1]Erftcarbon GmbH & CoKG, Aluminiumstrasse 4, Grevenbroich 41515 Germany

The use of fully graphitized cathode blocks, particularly in modern high-amperage technology cells, is growing. It is important to understand the specific requirements and demands of individual reduction technologies, and to characterize the performance trend of graphitized blocks in existing applications in order to optimize the blockís properties for future projects. The most important characteristics are the Electrical Resistivity, wear rate (erosion) and Thermal Properties. The operational performance is determined by a collection of factors e.g. raw material, granulometry, forming technology, baking, impregnation and graphitization. Recently, impregnation and the customized fine tuning of the graphitization process are used to enhance critical properties targeted at specific operational and performance improvements. The aim of this paper is to give an overview of the state of the art and an outlook for future developments and applications.

3:15 PM

Improvement of Abrasion Resistance of Graphitized Cathode Block for Aluminum Reduction Cells: *Shinjiro Toda*[1]; Tsutomu Wakasa[1]; [1]Nippon Electrode Company, Ltd., R&D Dept., 5600 Kambara, Kambara-cho, Ihara-gun, Shizuoka-ken 421-3203 Japan

Abrasion resistance of graphitized cathode is a key parameter to attain longer cell life. An apparatus for abrasion resistance measurement was developed to evaluate cathode blocks in aluminum electrolysis cells. Abrasion resistance of various kinds of cathode blocks, including anthracite based carbon blocks, was measured with this apparatus. The abrasion rate of graphitized blocks was found to be 3 times larger than that of anthracite blocks. This result agrees with the erosion rate of each grade of carbon blocks in the actual cells. Several cokes were investigated as raw material for graphitized blocks. A coke from high quinoline insoluble content pitch showed good hardness. This coke has large areas of optical isotropic texture and graphitized carbon blocks with this coke show good abrasion resistance relative to conventional grade.

3:40 PM Break

3:50 PM

Laboratory Test Method for Measuring Wear Rates of Carbon Cathode Materials: *Frank Hiltmann*[1]; Henrik Gudbrandsen[2]; Sverre Rolseth[2]; Lisbet St□en[2]; Jomar Thonstad[3]; [1]SGL CARBON GmbH, CFL, Griesheim Plant, Stroofstrasse 27, Frankfurt 65933 Germany; [2]SINTEF, Matl. Tech., Trondheim 7465 Norway; [3]Norwegian University of Science and Technology, Trondheim Norway

The wear of graphitized cathode blocks used in industrial aluminium electrolysis limits the lifetime of the cells. To study this problem a laboratory cell was developed to determine chemical wear rates of carbon cathode materials. It is based on the assumption that the carbon lining reacts with aluminium and is subsequently transferred to the cryolite bath in the form of dissolved carbide, which is eventually oxidised at the anode. The test cell that simulates this sequence consists of a sintered alumina crucible, housing a carbon test sample underneath an aluminium pad, and an oxygen-evolving inert anode immersed in the bath. As the only source of carbon is the cathode specimen, the rate of carbon transfer is monitored via the amount of CO2 and CO in the anode off-gas. Resulting wear rates are of the same magnitude as wear rates experienced in industrial cells, i.e. 2-5 cm per year.

4:15 PM

A New Technology for Cathode Rodding Used in Aluminium Electrolytic Cells: *Martin GagnÈ*[1]; Chantal Labrecque[1]; Dany Lavoie[2]; Annie LÈvesque[2]; Brian Murphy[2]; [1]Rio Tinto Iron & Titanium, 770 Sherbrooke St. W., Ste. 180, Montreal, Quebec H3A 1G1 Canada; [2]Aluminerie Alouette, Sept-Iles, Quebec Canada

The quest for the reduction of electrical resistance of the cathode blocks of aluminium smelting electrolytic cells has put every component of these cells under scrutiny, including the high phosphorus grey iron (HPGI) used as sealant between the steel cathodic bar and the graphite block. During this program, a detailed characterization of the as-cast and after-service HPGI roddings was performed and their microstructure and properties compared to those of ferritic Ductile Iron

WEDNESDAY PM

(FDI), a potential substitution material. Once the feasibility of production of FDI rodding confirmed, FDI cathode roddings were cast. Up to now, 130 cathode blocks have been put in service and six full cells are operational, the oldest one since August 2000. In this paper, the promising performance of the cathodes in service, which is probably related to the higher electrical conductivity and thermal stability of FDI, is discussed in function of cell stability and cathodic distribution.

4:40 PM
Electrical Resistance of Al-Steel Transiton Joints vs. Time and Temperature: *John G. Banker*[1]; Antoine Nobili[2]; [1]DMC Clad Metal Division, 5405 Spine Rd., Boulder, CO 80301 USA; [2]DMC Nobelclad Division, 1 Allee Alfred Nobel, Rivesaltes 66600 France

Electric transition joints (ETJ) are used for welding aluminum to steel in reduction cells. ETJs can experience temperatures up to 600∞C which can cause strength loss and resistance increase. This results form formation of brittle, high resistance Fe-Al intermetallics at the bond. Over the time required for mechanical failure to occur, the resistance can increase dramatically. The increase can be altered by inserting a titanium or chromium interlayer. Compared to bi-clad, the temperature at which resistance begins to increase is raised by 200∫ with chrome, and 300∫C with titanium. As-manufactured, resistance of a standard bi-clad specimen is 38.8 m-ohm, chromium interlayer ETJ is 55.8, and titanium interlayer ETJ is 44.4. After 275 hours at 500∞C, the bi-clad had fallen apart, the chrome interlayer ETJ resistance had increased to 11,250 and the titanium interlayer ETJ exhibited no change.

Cast Shop Technology: Solidification and Foundry Technology I

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; David H. DeYoung, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday PM Room: 6C
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Peggy Jones, General Motors Corporation, Powertrain, Saginaw, MI 48602-2641 USA; Philippe Jarry, Pechiney Group, Ctr. de Recherches, Voreppe 38341 France

2:00 PM
Effects of Hydrogen/Solidification Rate/ and Ca on Porosity Formation in As-Cast Aluminum Alloy A356: *Prince N. Anyalebechi*[1]; [1]Grand Valley State University, Padnos Sch. of Eng., L. V. Eberhard Ctr., Ste. 718, 301 W. Fulton, Grand Rapids, MI 49504-6495 USA

The effects of solidification rate, relatively high levels of melt hydrogen content and Ca on the amount and characteristics of porosity formed during solidification of aluminum alloy A356 have been quantitatively determined. Unidirectionally cooled laboratory-size ingots with solidification rate range of 0.2-7.5 K/s, 0.27-0.63 cc/100 g melt hydrogen concentration, and 0-52 ppm of Ca were used for the study. Amount of porosity and average pore size increased with increase in hydrogen concentration, but decreased with increase in solidification rate. At the 0.27 cc/100 g hydrogen level, 59 ppm of Ca discernibly decreased the amount of porosity. This is provisionally attributed to the contention that, like alkali (Li and Na) and other alkaline earth (Mg) metals, Ca probably increases the solubility of hydrogen in aluminum and its alloys.

2:25 PM
Optimal Heat Treatment of A356.2 Alloy: *Daryoush Emadi*[1]; Laurence V. Whiting[1]; Mahi Sahoo[1]; Jerry H. Sokolowski[2]; Paul Burke[3]; Mitch Hart[3]; [1]CANMET, Matls. Tech. Lab., 568 Booth St., Ottawa, Ontario K1A 0G1 Canada; [2]University of Windsor, NSERC/Ford-Nemak Industl. Rsrch. Chair, Rm. 209, Essex Hall, 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [3]Grenville Castings, 2 Air Care Dr., PO Box 785, Smiths Falls, Ontario K7A 4W6 Canada

The factors necessary to obtain an optimal heat treatment of ASTM B108 test bars in A356.2 were investigated. Test bars were solutionized at various times and temperatures, and then quenched in different media and cooling rates. In addition, the length of natural aging prior to artificial aging was studied to examine the scatter in the properties. The mechanical properties, electrical conductivity and hardness were measured for various artificial aging times and temperatures. Dimensional changes during heat treatment were measured by dilatometry. For test bars, an optimal heat treatment consists of a 4 hour solutionizing at 540°C, followed by water quenching, and then 12-20 hours natural aging followed by 6 hours aging at 155°C. This treatment has the advantages of good properties, with good reproducibility (least scatter and standard deviation), lower energy per heat treatment cycle and higher furnace throughput.

2:50 PM
Precipitation of Dispersoids in DC-Cast AA3103 Alloy During Heat Treatment: *Yanjun Li*[1]; Lars Arnberg[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., 7491, Trondheim Norway

The precipitation behavior of dispersoids in DC cast AA3103 alloy during heating and homogenization at 600∞C has been studied by means of TEM, electrical conductivity measurement and image analysis. During heating, a-Al(Mn,Fe)Si is the first phase to precipitate in the alloy. When heated to higher temperature, long rod like and plate like Al6(Mn,Fe) dispersoids precipitate in the alloy. During homogenization, the size of Al6(Mn,Fe) dispersoids grows with homogenization time while a-Al(Mn,Fe)Si dispersoids dissolve quickly. The evolution of dispersoids during heat treatment is controlled by nucleation, growth, coarsening and dissolution. The size, size distribution and number density of dispersoids have been measured. The volume fraction of dispersoids formed during heating, measured from TEM images, is in good agreement with the volume fraction calculated from the electrical conductivity of the alloy.

3:15 PM
Modelling of the Thermo-Physical and Physical Properties for Solidification of Al-Alloys: *Nigel Saunders*[1]; Xiuqing Li[2]; Alfred Peter Miodownik[1]; Jean-Philippe Schille[2]; [1]Thermotech, Ltd., Surrey Tech. Ctr., The Surrey Rsrch. Park, Guildford, Surrey GU2 7YG UK; [2]Sente Software, Ltd., Surrey Tech. Ctr., The Surrey Rsrch. Park, Guildford, Surrey UK

The thermo-physical and physical properties of the liquid and solid phases are critical components in casting simulations. Such properties include the fraction solid transformed, enthalpy release, thermal conductivity, volume and density all as a function of temperature. Due to the difficulty in experimentally determining such properties at solidification temperatures, little information exists for multi-component alloys. As part of the development of a new computer programme for modelling of materials properties (JMatPro) extensive work has been carried out on the development of sound, physically based models for these properties. Wide ranging results will be presented for Al-based alloys, which will include more detailed information concerning the density change of the liquid that intrinsically occurs during solidification due to its change in composition.

3:40 PM Break

3:50 PM
Effects of Solidification Rate/Fe/ and Ca on the Cast Microstructure of Aluminum Alloy A356: *Prince N. Anyalebechi*[1]; [1]Grand Valley State University, Padnos Sch. of Eng., L. V. Eberhard Ctr., Ste. 718, 301 W. Fulton, Grand Rapids, MI 49504-6495 USA

The effects of solidification rate, Fe, and Ca on the characteristics of the constituent phases formed during solidification of aluminum alloy A356 have been investigated. This involved the use of unidirectionally cooled laboratory-size ingots with a range of solidification rate of 0.2-7.5 K/s. Two levels Fe (0.05 wt.% and 0.20 wt.%) and Ca (0 ppm and 59 ppm) were investigated. As expected, average dendrite cell and constituent phase particle size decreased exponentially with increase in solidification rate. Effect of Ca depended on the Fe content in the alloy. For example, at 0.20 wt.%Fe and 0.05 wt.%Fe, Ca slightly increased and decreased, respectively, the average constituent phase particle size. At the low levels investigated, Fe content did not affect the average size and/or volume fraction of the constituent phases.

4:15 PM
Microstructure Formation in Directionally Solidified Al-Cu Alloys: *Shan Liu*[1]; Je H. Lee[1]; Heath Walker[1]; Rohit K. Trivedi[1]; [1]Iowa State University, Ames Lab., USDOE, Ames, IA 50011 USA

Microstructure lengthscales determines the properties (mechanical, corrosion, heat-treatment, electro-magnetic, etc.) and thus the service life of a casting and directional solidification has been used to disclose the relationship of different lengthscales with the control parameters, e.g. alloy composition, temperature gradient and growth velocity. We have systematically studied the microstructure development during upward solidification of Al-Cu alloys with compositions ranging from 4.0~40.0 wt%Cu. It was found both numerically and experimentally that liquid convection dominates the microstructure evolution in a sample of size >1.0mm diameter, which causes serious problems when one tries to correlate the microstructure dimensions with the control parameters. A new experimental technique has been developed where diffusion controls the microstructure formation. A thorough comparative study on the microstructure evolution of Al-Cu alloys will be presented over the above-mentioned composition range where the interface morphology can be cellular/dendritic or eutectic.

4:40 PM
Squeeze Casting of a Conventionally Wrought Aluminium Alloy: *Chris D.J. Manson-Whitton*[1]; Brian Cantor[1]; Keyna OíReilly[1]; Peter Schumacher[1]; [1]Oxford University, Dept. of Matls., Begbroke Business & Sci. Park, Sandy Ln., Yarnton, Oxfordshire OX5 1PF UK

High structural integrity aluminium castings can be achieved through controlled, refined microstructures, low porosity and low levels of inclusions. Mechanical properties can be further improved through the use of high strength alloys, such as those used in conventionally wrought applications. Traditional casting techniques preclude the use of such alloys, however squeeze casting offers the opportunity for controlled microstructures in wrought alloys direct from the melt. 7xxx series components have been cast using an experimental direct squeeze caster. Filling and pressurisation parameters were controlled in order to understand key aspects of the solidification regime, particularly the formation of macrosegregation in long freezing range alloys. As-cast microstructures of gravity and pressurised castings were characterised in terms of structural scale and segregation, and discussed with reference to measured solidification profiles.

5:05 PM
The Mechanics of Casting Condition and Quality of As-Cast Products Interrelationships During High-Pressure Die-Casting of Al-Si Alloys: *Reza Ghomashchi*[1]; [1]University of Quebec at Chicoutimi, Ctr. for Univ. Rsrch. on Aluminium-CURAL, 555 University Blvd., Chicoutimi, Quebec G7H 2B1 Canada

The effects of casting conditions, including fluid flow rate, casting and ingate thickness, and melt and die temperatures, were studied on the microstructure of LM24 high pressure die casting alloy using a fully-controlled cold chamber high-pressure die-casting machine coupled with an experimental die set of a rectangular die cavity shape of 65x130x(2, 4, 8) mm. All castings showed a bimodal distribution of dendrites in which the morphology of dendrites was completely different to the classic tree shape characteristic. It was found that the casting parameters influence the quality of the coupons surface finish, porosity size and percentage, volume fraction and size of dendrites, while have no effect on the silicon morphology and percentage of Al-Si eutectic.

Computational Phase Transformations: Solidification and Crystallization

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)
Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Wednesday PM — Room: 11B
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Ralph E. Napolitano, Iowa State University, Ames Lab. Dept. of Matls. Sci. & Eng., Ames, IA 50011 USA; Alain Karma, Northeastern University, Ctr. for Interdisciplinary Rsrch. on Complex Sys., Boston, MA 02115 USA

2:00 PM Invited
Phase-Field Modeling of Dendritic Solidification with Coupled Heat and Solute Diffusion: Juan C. Ramirez[1]; *Christoph Beckermann*[1]; [1]University of Iowa, Dept. Mechl. Eng., 2412 SC, Iowa City, IA 52242 USA

A phase-field model for dendritic solidification of dilute alloys has been developed that accounts for the simultaneous diffusion of heat and solute, including unequal diffusivities. The model incorporates the thin-interface asymptotics developed by Karma and coworkers for the purely thermal and solutal cases, allowing calculations to be performed for coupled heat and solute transport using an interface thickness that is large compared to the capillary length and in the limit of vanishing interface kinetics. In addition, an extra term is included in the solute diffusion equation that eliminates the artifical solute trapping effect due to smearing of the interface. Simulations are performed for free dendritic growth into an undercooled melt of a dilute binary alloy. The effect of solute additions on the dendrite tip growth velocity, tip radius, and operating point selection are investigated.

2:30 PM Invited
Phase-Field Modeling of Alloy Directional Solidification: *Alain Karma*[1]; [1]Northeastern University, Ctr. for Interdisciplinary Rsrch. on Complex Sys., Boston, MA 02115 USA

Despite several decades of theoretical study of directional solidification since the pioneering analysis of Mullins and Sekerka on morphological stability, several fundamental aspects of the formation of cellular and dendritic array structures remain poorly understood. We have carried out a numerical study of these array structures that is based on a new phase-field formulation of the solidification of dilute binary alloys [A. Karma, Phys. Rev. Lett, {\bf 87}, 115701 (2001)]. This formulation makes it possible to simulate quantitatively microstructural evolution in the low velocity regime where the solid-liquid interface is close to thermodynamic equilibrium (i.e. with negligible interface kinetics and solute trapping). We present new results that shed light on the relative importance of thermodynamic noise, i.e. concentration fluctuations of microscopic origin, and the purely deterministic growth dynamics in the formation of secondary branches (sidebranching). The main new finding is the existence of a stable limit cycle for a range of large spacing of the array that can drive sidebranching even in the absence of noise. We also present a quantitative comparison of our results with available experimental data.

3:00 PM
Competitive Growth During Directional Solidification: A 3D Cellular Automaton Model: *Wei Wang*[1]; Peter D. Lee[1]; Malcolm McLean[1]; [1]Imperial College, Matls., Prince Consort Rd., London SW7 2BP UK

Grain selection plays an important role when producing directional solidified and single crystal Nickel-based superalloy turbine blades. Misorientated grains are normally overgrown by grains with their preferred growth direction, <100>, better aligned normal to the isotherms, producing a sharp texture in the final component. A 3D model that combines a Cellular Automaton description of grain growth with a Finite Difference solution of solute diffusion has been developed to simulate competitive growth. This model, which reproduces the full dendritic structure, including primary and secondary arms, was applied to simulate the competitive growth between both converging and diverging grains. The results show an excellent correlation to experiment, allowing an investigation of the overgrowth and branching mechanisms during both quasi-steady state and perturbed growth, leading to conditional overgrowth of misorientated grains.

3:20 PM
Effects of Multicomponent Diffusion and Thermodynamics on the Dendritic Structure in Ternary Fe-C-Mn: Hermann Josef Diepers[1]; Dexin Ma[1]; *Ingo Steinbach*[1]; [1]Access e.V., Intzestrasse 5, Aachen D-52072 Germany

An experimental and numerical study is performed to investigate the effect of multicomponent diffusion and thermodynamics on the dendritic structure of an Fe-Mn-C alloy. The directionally solidified dendritic structures are uncovered using the new technique of introducing artificial pores into the sample. This allows for the direct observation of the feritic structure even after peritectic transformation. The numerical approach uses a multicomponent phase-field model. The simulations are compared to the experimental results. Focus of the research is the importance of kinetics, thermodynamics and their interaction. In order to separate both effects ternary simulations with realistic diffusion and phase diagram data are compared to simulations with idealized quasibinary data. It is shown, that in general there is no quasibinary model available to describe both effects of diffusion and thermodynamics in multicomponent systems. The differences in tip selection, microsegregation and primary spacing are quantified.

3:40 PM Break

3:55 PM Invited
The Three Dimensional Evolution of Topologically Complex Structures During Coarsening: I. Savin[1]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Eng., Cook Hall, 2225 N. Campus Dr., Evanston, IL 60208 USA

The coarsening process in systems consisting of spherical particles in a matrix has been studied extensively. In contrast, coarsening in systems that possess both positive and negative curvatures, such as those present following dendritic solidification, have received less study. A challenge in understanding the evolution of such microstructures is that the evolution must be studied in three dimensions. We have employed three dimensional phase field calculations to follow the evolution of a topologically complex dendritic microstructure during coarsening. We compare the results of experiments on the coarsening of dendritic microstructures with the calculations by using the experimentally measured microstructures as initial conditions in our calculations. The calculations show the important role of topological singularities in the evolution of dendritic structures during coarsening, particularly the pinching of liquid channels via a Rayleigh instability.

4:25 PM Invited
Phase Field Theory of Multi-Domain Solidification in Alloys: *L·szlÛ Gr·n·sy*[1]; Tam·s Pusztai[1]; Tam·s B^rzs^nyi[1]; [1]Research Institute for Solid State Physics & Optics, PO Box 49, Budapest H-1525 Hungary

We present a phase field theory for describing the nucleation and growth of one and two-phase crystals solidifying with different crystallographic orientations in binary liquid alloys. The regular solution model is used to describe the thermodynamic properties. We investigate the kinetics of crystallization as a function of model parameters (anisotropy, symmetry, initial liquid composition) for primary, eutectic, and peritectic solidification under equiaxial conditions.

4:55 PM Invited
Prediction of Solidification Microstructures in Multi-Component Alloys Using Fixed Grid Methods and Thermodynamic Calculations: *Alain Jacot*[1]; Michel Rappaz[1]; [1]EPFL, Matls. Inst., MX-G, Lausanne CH-1015 Switzerland

The pseudo-front tracking method and the phase-field model are fixed grid numerical techniques which permit to describe diffusive phase transformations without having to explicitly track the interface. These methods have proved to provide accurate solutions to moving boundary problems. However, important issues are still to be addressed in order to apply such models to practical problems: the high computation time and the need to couple with thermodynamic databases to describe multi-component systems. The first part of this contribution will focus on the pseudo-front tracking method and its application to solidification in multi-component systems. Examples of calculations for various thermal and grain size conditions will be shown. Comparisons with phase field calculations will be presented with emphasis on the advantages and limitations of the two methods. In a second part, different strategies to couple microstructure models with thermodynamic databases will be presented. The issue of computation time will be discussed using simple estimators to show the influence of the solidification conditions and numerical parameters.

5:25 PM
Phase Field Modeling of Solidification with Liquid Convection and Solid Motion: *Adam C. Powell*[1]; Jorge Alberto Vieyra Salas[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139-4301 USA

A novel mixed-stress model permits coupling of phase field modeling of solidification with Navier-Stokes fluid dynamics in the liquid and linear elastic deformation in the solid. Stress is transitioned between Newtonian viscosity in the liquid and elasticity in the solid across the diffuse interface using an interpolation function of the phase variable. Solid stress is related to local solid strain, which is an additional tensor field in the model. The strain tensor and crystalline orientation are both rotated according to the local vorticity. Results are given for an incompressible fluid and solid with Poisson ratio of 1/2, using both Cahn-Hilliard and anisotropic Allen-Cahn phase field formulations in two dimensions, and extension to three dimensions is discussed.

Defects and Deformation of Crystalline Solids - in Honor of Dr. Man H. Yoo - VI

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Physical Metallurgy Committee
Program Organizers: Jong K. Lee, Michigan Technological University, Metallurgical & Materials Engineering Department, Houghton, MI 49931-1200 USA; Sean R. Agnew, University of Virginia, Materials Science and Engineering Department, Charlottesville, VA 22904-4745 USA; K. N. Subramanian, Michigan State University, Department of Material Science & Mechanics, East Lansing, MI 48824-1226 USA

Wednesday PM — Room: 17A
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Craig Hartley, Air Force Office of Scientific Research, Arlington, VA 22203-1977 USA; Vijay Vasudevan, University of Cincinnati, Dept. of Cheml. & Matls. Eng., Cincinnati, OH 45221 USA

2:00 PM Invited
High Temperature Mechanical Behavior of Cryomilled Al-Al_3Ti: *J. R. Weertman*[1]; B. Dehiya[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

Cryomilled Al-Al_3Ti has good high temperature strength and microstructural stability. Stress-strain curves over a range of temperatures show little or no strain-hardening. At high temperatures and fast strain rates a decided yield point is observed. The yield point disappears as the strain rate drops and serrated curves become evident. Holding a sample at elevated temperature before testing, with or without load, enhances the yield point. The yield point and serrations are simply modeled on the basis of initial low dislocation density, rapid generation and concurrent annihilation.

2:25 PM Invited
Effects of Dislocation-Crack Interactions on Fatigue Crack Growth: *K. Sadananda*[1]; Gregory Glinka[2]; Jerry Hsieh[3]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 20375 USA; [2]University of Waterloo, Dept. of Mechl. Eng., Waterloo, Ontario N2L 3G1 Canada; [3]Thomas Jefferson High School for Science & Technology, Fairfax Co. Board of Edu., 6560 Braddock Rd., Springfield, VA USA

Fatigue crack growth occurs due to irreversibility of plastic flow. Plastic flow occurs by the nucleation and movement of dislocations. Hence dislocation-crack interactions play a significant role in the mechanics of fatigue crack growth. It is well known that crack-tip plasticity contributes to fatigue crack growth via plastic-blunting process. However, there are several questions regarding the role of plasticity in the wake of a growing crack. Many theories and models have been developed based on the notion that plasticity behind the crack tip contributes to premature crack closure thereby retarding crack growth. This concept of plasticity induced crack closure is reexamined using dislocation-crack interactions. The results and their implications will be discussed in relation to fatigue crack growth kinetics.

2:50 PM
On the Mechanism of Mechanical Deformation Twin Thickening in Titanium: *Steven Celotto*[1]; Gareth G.E. Seward[2]; Robert C. Pond[1]; [1]University of Liverpool, Dept. of Eng., Matls. Sci. & Eng., Brownlow Hill, Liverpool L69 3BX UK; [2]University of Liverpool, Dept. of Earth Scis., Liverpool L69 3GP UK

In this study we have investigated the mechanism of deformation twinning in commercially pure titanium by experimental and theoretical means. Scanning electron microscopy has been used to determine the morphology of twins in lightly deformed specimens. Lateral boundaries of the twins often exhibit irregular shapes rather than lenticular form. The crystallography of individual twins has been established using electron back-scattered diffraction. In addition, the magnitude and direction of the shear have been found by measuring the distortions induced on a fiducial grid scribed on the initial specimen surface using a focussed laser beam. It was observed that in the vicinity of irregular twin boundaries, reorientation of the fiducial lines only occurred where they intersected the interface. These observations confirm that deformation has only occurred within the twins, and no emissary slip process as described by Sleeswyk has taken place near irregular lateral boundaries. Using atomic scale modelling, such irregular deformation can be explained in terms of localised twinning dislocation dipoles that are activated by the effect of applied stresses on stress concentrations near sessile interfacial defects.

3:10 PM

DHC Velocity and KIH of Zr-2.5Nb Tubes with Hydrogen Concentration: *Young Suk Kim*[1]; [1]Korea Atomic Energy Research Institute, Zirconium Grp., 150, Dukjin-dong, Yusong, Daejon 305-353 Korea

This study focuses on the elucidation of delayed hydride cracking (DHC) of zirconium alloys, which has not been clearly understood up to date. DHC tests were conducted on the compact tension (CT) specimens of a CANDU Zr-2.5Nb pressure tube with hydrogen concentration varying from 12 to 100 ppm. Hydrogen was charged electrolytically into the CT specimens followed by a homogenization treatment. The DHC velocity of the Zr-2.5Nb pressure tube had a temperature dependence with an activation energy of 49 KJ/mol in a temperature range from 100 to 300°C, and increased linearly to a constant with increasing hydrogen concentration. In contrast, the threshold stress intensity factor, KIH in the axial direction of Zr-2.5Nb tube had a drastic decrease of KIH to a constant with increasing hydrogen concentration. Thus, DHCV and KIH were nicely described as a function of the supersaturated hydrogen concentration over the terminal solid solubility for dissolution (TSSD) independent of temperatures. Based on these results , we propose that KIH is a critical stress intensity factor to initiate the nucleation of hydrides at the crack tip region, driving the crack tip with the supersaturated hydrogen in solution to reach the TSSD at any temperatures. Therefore, we conclude that the gradient of the equilibrium hydrogen concentration or TSSD at the crack tip region and the supersaturated one at the matrix region is a governing factor to initiate DHC. Normalization of the striation spacing by hydrogen diffusivity and hydrogen solubility demonstrates that the striation spacing decreases with increasing temperature. Therefore, the striation spacing, inversely proportional to DHCV, is a good parameter representing the DHC velocity of the Zr-2.5Nb tubes.

3:30 PM Break

3:50 PM Invited

Creep Anisotropy and Radiation Effects in Zr-Alloy Tubing: Application to In-Reactor Dimensional Prediction: *K. L. Murty*[1]; [1]North Carolina State University, 2500 Stinson Dr., PO Box 7909, Raleigh, NC 27695-7909 USA

Zirconium alloyed mainly with Sn and Fe known as Zircaloys commonly used in reactors are highly textured and exhibit anisotropic physical and mechanical properties. We summarize here the crystallographic textures commonly noted in Zircaloy cladding that lead to mechanical anisotropy and the effects of stress-relief and recrystallization anneals following tube reduction processes. Anisotropic creep behaviors were characterized through biaxial creep tests and creep loci. Quantitative description of the crystal texture through CODF combined with prism slip dominance predicted the creep locus of the recrystallized tubing. Deviations exhibited by the stress-relieved cladding were seen to stem from grain shape anisotropy. Neutron radiation exposure of the stress-relieved tubing resulted in radiation creep and stress-free radiation growth. The texture-creep model combined with radiation effects enabled us to predict the axial position dependence of the diametral decrease. The paper concludes with challenges posed by the new advanced cladding alloys with composition gradients as well as Nb-additions and surface modifications.

4:15 PM

Oxide Formation on the Creep Cavities of Type 316L Stainless Steel: *Yongbok Lee*[1]; Jinsung Jang[2]; Woo-Seog Ryu[2]; Dokyol Lee[1]; [1]Korea University, Dept. of Matl. Sci. Eng., Seoul 136-353 Korea; [2]KAERI, Nucl. Matls. Tech. Rsrch. Team, Daejeon 305-600 Korea

Creep failure of austenitic stainless steels is usually occurred through the nucleation and growth of cavities. Solution treated 316 L stainless steel was creep tested at 620 C and 220, 240 and 260 MPa in vacuum as well as in atmosphere. Creep rupture life, creep elongation and steady state creep rate in vacuum were better than those in atmosphere. Creep constants from the normalized creep curves were estimated as 2.55 and 2.89 in air and in vacuum, respectively. Oxide film, $(Fe_{0.8},Cr_{0.2})$ O, was found on the inner surface of creep cavities of type 316 L stainless steel and the change of surface energy of the cavity wall is expected to affect the nucleation or growth process. Crystal structure and the lattice parameter of the oxide film were investigated using TEM (JEM 2000FX) with EDS. Oxide film was FCC structured polycrystal and the lattice parameter of the oxide was about 4.056 angstrom.

4:35 PM Invited

Improvement of Creep-Fatigue Resistance Modifying the Carbide Interfacial Energy in Austenitic Stainless Steels: *Soo Woo Nam*[1]; Kyung Seon Min[1]; Ki Jae Kim[1]; Hyun Uk Hong[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701 Korea

Creep-fatigue life is reduced by cavitation at grain boundary carbides at high temperature in austenitic stainless steels. Because the interfacial energy between carbides and neighboring matrix can affect creep-fatigue life, it is suggested that interfacial energy can be decreased by modification of carbide and grain boundary characteristics. In AISI 304 and 316 stainless steels, it is found that the interfacial energy can be remarkably reduced through grain boundary serration to extend the creep-fatigue life. Therefore, the mechanism of the grain boundary serration is investigated. It is expected that planar carbides on serrated grain boundaries have a lower interfacial energy than that of triangular carbides on straight grain boundaries. The different influence of TiC and $Cr_{23}C_6$ on creep-fatigue life in AISI 321 stainless steel with the same carbide density at the grain boundary is based on the stronger cavitation resistance of TiC. It is verified that formation and growth of grain boundary cavities in TiC aged alloy are more difficult than those in $Cr_{23}C_6$ aged alloy.

5:00 PM

Defect Structures in the Massive γ_m Phase in a Quenched Ti-46.5 At.% Al Alloy: Ping Wang[2]; Mukul Kumar[3]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA; [3]Lawrence Livermore National Laboratory, 7000 East Ave., L-356, Livermore, CA 94550 USA

The defect structures in the massively formed gamma (γ_M) grains in a Ti-46.5 at.% Al alloy, rapidly quenched from the high-temperature α-phase field, have been studied by conventional and high-resolution TEM. Defect structures composed of dislocations, stacking faults (SFs) and antiphase boundaries (APBs) intimately associated with dislocations or stacking faults were observed. Analysis indicates that both 1/2<110] and 1/2<101] unit dislocations were present in the gm phase, with the latter linked by highly curved APBs. Comparison of experimental and computer simulated TEM images established that wide SFs, which are created by the dissociations of1/2<101> unit dislocations, lie on {111} planes and are bound by b = 1/6<121> Shockley partial dislocations of all possible types. In addition, APBs are found to commence or terminate on the SFs at the partial dislocations with b = 1/6<121], but not those with b = 1/6<112]. Confirmation for both the latter, as well as the intrinsic nature of the stacking faults was obtained using atomic models and analysis of the HREM images. Based on the observations and subsequent analyses, a model for the formation of these defects and defect configurations is proposed. The authors are grateful for support of this research by the National Science Foundation under grants DMR-9224473 and 9731349, Dr. Bruce MacDonald, Program Monitor.

Dynamic Deformation: Constitutive Modeling, Grain Size, and Other Effects: Symposium in Honor of Professor Ronald W. Armstrong: Other Effects

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Marc Andre Meyers, University of California-San Diego, Department of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA; George T. Gray III, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Naresh Thadhani, Georgia Institute of Technology, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA; Kenneth S. Vecchio, UC San Diego, Dept of Mechanical and Aerospace Engineering, La Jolla, CA 92093-0411 USA

Wednesday PM	Room: 16B
March 5, 2003	Location: San Diego Convention Center

Session Chair: Hans Conrad, North Carolina State University, Matls. Sci. & Eng., Raleigh, NC 27695-7907 USA

2:00 PM
Plastic Deformation Behavior of A356/357 Aluminum Cast Alloys: *Qigui Wang*[1]; [1]General Motors Corporation, CDVC-Powertrain, 1629 N. Washington Ave., Saginaw, MI 48605 USA

Strain hardening rate of A356/357 aluminum cast alloys during plastic deformation depends on eutectic particle size and morphology, secondary aluminum dendrite arm spacing (SDAS), and Mg contents. At low plastic strain, the eutectic particle aspect ratio and matrix strength dominate the work hardening, while at larger plastic strains the hardening rate depends on the secondary dendrite arm spacing (SDAS). For all materials, the average internal stresses increase very rapidly at small plastic strains gradually saturating at large plastic strains. Elongated eutectic particles, small SDAS or higher Mg content result in a higher saturation value. The difference in the internal stresses, due to different microstructural features, determines the rate of eutectic particle cracking and in turn the tensile instability of the materials. The fracture strain of alloys A356/357 corresponds to the critical fraction of cracked eutectic particles locally or globally, irrespective of the fineness of the microstructure.

2:20 PM
NEMD Simulations of Metallic Friction: *James E. Hammerberg*[1]; Brad L. Holian[2]; Timothy C. Germann[1]; Ramon J. Ravelo[1]; [1]Los Alamos National Laboratory, Appl. Physics Div., MS D413, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Theoretl. Div., MS B268, Los Alamos, NM 87545 USA

With the advent of large-scale parallel computers in the past decade it has become possible to study the microscopic physics of complex nonlinear many-body systems at scales approaching the mesoscale. The increase in computational power has made possible the study of very large systems, of order 10^7-10^8 atoms. This has, in turn, made possible the study of dislocations, nano-scale grain dynamics and nucleation, and non-equilibrium plastic flow in unprecedented detail. One of the areas where non-equilibrium deformation arises is that of dry sliding friction between ductile metals. We discuss the results of atomistic simulation studies of sliding friction in the velocity range 10-10^3 m/sec under a variety of loading conditions from 10^{-1}-10^3 GPa for a variety of tribo-pairs,including Cu/Cu, Cu/Ag, and Ta/Al,as well as model systems such as Lennard-Jones. The effective tangential force as a function of sliding velocity exhibits generic behavior at high velocities determined by nano-scale structural transformation, and in certain cases material mixing, at high strain and strain rates. The Non-Equilibrium Molecular Dynamics (NEMD) techniques used in these large-scale simulations will be discussed and their connections to the multi length- and time-scale theory of sliding friction will be presented.

2:40 PM
Three-Dimensional Dislocation Dynamics Simulations of the Interaction Between Dislocations and Low-Angle Grain Boundaries: *Tariq A. Khraishi*[1]; Yu-Lin Shen[1]; [1]University of New Mexico, Dept. of Mechl. Eng., Albuquerque, NM 87131 USA

Parametric simulation studies of dislocations interacting with low-angle grain boundaries were carried out using a discrete 3D Dislocation Dynamics code. The tilt boundaries, consisting of dislocation arrays and assumed to be fixed in space, are mainly characterized by the tilt angle which in turn controls the separation distance between dislocations in the wall (i.e., grain boundary). Dislocation sources were placed in between two walls representing the boundaries of a single grain. The simulations revealed an interesting sequence of events which depended primarily on the length scales of the problem: the separation distance between the dislocations in the wall, and the separation distance between the walls themselves, representing the grain size. In addition, the simulations illustrated the arrest of propagating dislocations into the strain field of the stationary wall dislocations. Implications on the strengthening effect and relevant micromechanical issues are also discussed.

3:00 PM
Dynamic Recrystallization: Plasticity Enhancing Structural Development Induced by Strain: *H. J. McQueen*[1]; [1]Concordia University, Mech. Industl. Eng., Montreal H3G 1M8 Canada

Dislocation mobility upsurge usually causes significant improvement in the plasticity of crystalline solids, partly through its ability to deter crack formation. Dynamic recrystallization DRX that proceeds during deformation reduces the flow stress and markedly raises the ductility, which characterize the hot working range. While it facilitates straining, its nucleation rate and equilibrium grain size are defined by the temperature and strain rate. Its operation is thermally activated like other deformation assisting mechanisms such as source unpinning, cross slip and climb; these last two reduce the strain hardening in combination with annihilation and polygonization of substructure that constitute dynamic recovery DRV. The flow stress in steady state is not directly defined by the grain size but rather by the average dislocation density which is tied to both DRV and DRX in so far as the migration of GB reduce the dislocation density below that attained by DRV alone. In hexagonal metals, stress concentrations at grain boundaries lead to a mantle of fine recrystallized grains in which the strain becomes concentrated while the old grain cores contribute little. Dynamic recrystallization does not in its operation produce any strain. It is a dynamic restoration process which does facilitate straining with respect to ease and extent. In distinction from its static conterpart its progress and its effect are intimately associated with the plastic deformations.

3:20 PM
Mechanisms of Deformation in Nanostructured Metals: *Robert John Asaro*[1]; [1]University of California-San Diego, Structl. Eng., R0085, La Jolla, CA 92093 USA

This talk will be concerned with establishing the mechanisms of deformation and strengthening in metals and alloys with nano-sized grains. Specifically, the role of dislocation motion both within and normal to slip planes will be developed within the context of a full constitutive theory and used, via numerical simulation, to describe the formation of grain boundary, and interface, ìboundary layersî that impart high strength and strain hardening. The role of grain boundary sliding and grain rotations will also be explored. Experimental evidence will also be provided to verify the concepts developed.

3:40 PM Break

3:50 PM
Quantitative Extraction of High Explosive Drive and Material Constitutive Parameters from Velocity Histories: *Wayne E. King*[1]; I. Harold Zimmerman[2]; Desmond Pilkington[1]; Oliver T. Strand[1]; Michael W. Danforth[3]; [1]University of California, Lawrence Livermore Natl. Lab., Chem. & Matls. Sci. Direct., L-356, Livermore, CA 94551 USA; [2]University of California, Lawrence Livermore Natl. Lab., Defense & Nucl. Tech. Direct., L-095, Livermore, CA 94551 USA; [3]University of California, Lawrence Livermore Natl. Lab., Defense Technologies Eng. Div., Livermore, CA 94551 USA

Fabry-Perot velocimetry is widely used, often in combination with hydrocode simulations, to study the physical processes underlying shock effects in materials. However, matching experiment with simulation is to some extent a qualitative undertaking. In this work, we employ non-linear least squares methods to develop a quantitative match between the velocity histories of two high-explosive-driven flat-plate experiments and predictions of 1 and 2-dimensional hydrocode simulations. Results extracted from the optimization include the Jones-Wilkins-Lee (JWL) equation of state parameters for the high-explosive drive and constitutive parameters describing the behavior of the flat-plates. This work performed under the auspices of US Department of Energy by University of California, Lawrence Livermore National Laboratory under contract no. W-7405-Eng.-48.

4:10 PM
Comparative Grain Size Influences in Dynamic Deformation and Creep: *Frank R.N. Nabarro*[1]; [1]University of the Witswatersrand, Condensed Matter Physics Rsrch. Unit, Private Bag 3, Johannesburg WITS 2050 S. Africa

Further consideration is given, as far as the polycrystal grain size may influence the material deformation rate, to whether the applied stress: (i) is first greater than the material Peierls stress; (ii) then to whether the the grain size is larger than the Taylor dislocation lattice spacing corresponding to the Peierls stress; (iii)followed by whether the product of stress and grain size exceeds the value for operation of modeled dislocation sources; and, lastly, (iv) to whether the product of stress and square root of grain size exceeds the Hall-Petch value for propagation of slip across grain boundaries {see Soviet Physics - Solid State Physics, Vol. 42, No. 8, pp. 1417-1419 (2000)}.†

4:30 PM
Effect of Low-Temperature Shock Compression on the Microstructure and Strength of Copper: *David H. Lassila*[1]; Tien Shen[1]; Marc A. Meyers[2]; [1]Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; [2]University of California-San Diego, La Jolla, CA 92093 USA

Copper with two purities (99.8 and 99.995%) was subjected to shock compression from an initial temperature of 90 K. Shock compression was carried out by an explosively accelerated flyer plates at velocities generating pressures between 27 and 77 GPa. The residual microstructure evolved from dislocation cells to mechanical twins and, at the 57 and 77 GPa pressures, to complete recrystallization with

WEDNESDAY PM

a grain size larger than the initial one. The shock-compressed copper was mechanically tested in compression at 10(superscript: -3) s(superscript: -1) at 300 K; the conditions subjected to lower pressures (27 and 30 GPa) exhibited work softening, in contrast to the conventional work hardening response. This work softening is due to the breakdown of the unstable shock deformation microstructure consisting high density of uniformly distributed dislocations and the formation of larger cells, with a size of approximately 1m. The 99.995% copper subjected to the higher shock compression pressures (57 and 77 GPa) exhibited a stress-strain response almost identical to the unshocked condition. This indicates that the residual temperature rise was sufficient to completely recrystallize the structure and eliminate the hardening due to shock compression. Thermodynamic calculations using the Hugoniot-Rankine conservation equations predict residual temperatures of 357 and 560 K for the 57 and 77 GPa peak pressures, respectively. The complete recrystallization is evidence for a higher residual temperature and suggests that a plastic deformation term should be added to the release isentrope.

4:50 PM

TiC-NiTi Cermets: Structure and Ballistic Performance: *Eugene A. Olevsky*[1]; Elizabeth Strutt[2]; Marc A. Meyers[1]; Werner Goldsmith[3]; David J. Benson[2]; [1]San Diego State University, 5500 Campanile Dr., San Diego, CA 92182-1323 USA; [2]University of California-San Diego, Dept. of MAE, La Jolla, CA 92093-0411 USA; [3]University of California-Berkeley, Dept. of Mechl. Eng., Berkeley, CA 94705 USA

TiC-NiTi cermets were produced by a new process consisting of two stages: combustion synthesis followed by quasi-isostatic pressing (QIP) using a granular pressure transmitting medium. The structure of the cermet consists of spheroidal TiC particles surrounded by a NiTi matrix, that has the thermoelastic properties. The material was charcterized by transmission electron microscopy. Ballistic testing was carried out in the DOP(depth of pentration) configuration. Steel specimens were impacted at the same velocities as cermet specimens and the ballistic efficiency was estimted; it was found to vary between 6 and 8, in comparison to hot rolled AISI 1018 steel. The experimental ballistic results are successfully compared with computed predictions. Research funded by the US Army Research Office MURI Program.

5:10 PM

Chaotic and Power Law States in the Portevin-Le Chatelier Effect: Madurai Srinivasan Bharathi[1]; Garani Ananthakrishna[1]; [1]Indian Institute of Science, Matls. Rsrch. Ctr., Bangalore, Karnataka 560012 India

Recent studies on the PLC effect report an intriguing crossover phenomenon from a low dimensional chaotic to an infinite dimensional power law regime in experiments on Cu-Al single crystals and Al-Mg polycrystals, as a function of strain rate. We devise a fully dynamical model which reproduces these results. At low and medium strain rates, the model is chaotic with the structure of the attractor resembling the reconstructed experimental attractor. At high strain rates, power law statistics for the magnitudes and durations of the stress drops emerge as in experiments. The spectrum of Lyapunov exponents changes from a set of positive, few zero and negative exponents in the chaotic regime to a power law distribution of null exponents in the power law state. These results can be made more transparent by investigating the spatial configuration of the dislocations on the slow manifold. This shows that while a large proportion of dislocations are pinned in the chaotic regime, most of them are pushed to the threshold of unpinning in the scaling regime, thus providing insight into the mechanism of crossover.

5:30 PM

Nonuniform Motion of Supersonic Dislocations: *Xanthippi Markenscoff*[1]; Luqun Ni[1]; [1]University of California, Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92093 USA

A nonuniform supersonic motion of a dislocation, i.e. with variable speed, is analyzed. In this case,the Mach wave fronts are not straight lines as in the uniform case, but curved. Comparisons will be made with the numerical simulation results of Gumbsch and Gao(1999). The dislocation core will be assumed to be either a step function discontinuity (Volterra dislocation), or ramp-like. The energetics will be also discussed.

Friction Stir Welding and Processing II: Friction Stir Joining: Mechanical Properties

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee

Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Thomas J. Lienert, University of South Carolina, Mechanical Engineering Department, Columbia, SC 29208 USA; Rajiv S. Mishra, University of Missouri-Rolla, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Wednesday PM — Room: 7B
March 5, 2003 — Location: San Diego Convention Center

Session Chair: Maria Posada, Naval Surface Warfare Center, Carderock Div., W. Bethesda, MD 20817 USA

2:00 PM

Process Parameters-Mechanical Properties Relationship in Self-Reacting Friction Stir Welds of Aluminum Alloys: *Z. X. Li*[1]; G. P. Adams[1]; [1]Lockheed Martin Space Systems Company, Michoud Ops., PO Box 29304, New Orleans, LA 70189 USA

Self-reacting friction stir welding process (SR-FSW) is a relative new technology that is derived from conventional friction stir welding (FSW) process. A number of advantages including elimination of backing anvil and significant lower process loads have made the self-reacting FSW process a competitive technology as compared with the conventional FSW process. In present work, butt joint configurations for joining plates in thickness ranges from 0.320" to 1.000" were friction stir welded using a self-reacting pin tool system. Various pin tool designs and process parameters were applied to understand the effect on the mechanical properties, microstructure, and defect formation of the self-reacting welds. Mechanical properties and microstructure of the resulting welds as a function of pin tool designs and process parameters will be highlighted.

2:25 PM

Welding Tool and Process Parameter Effects in Friction Stir Welding of Aluminum: *Kevin J. Colligan*[1]; James J. Fisher[2]; Joseph R. Pickens[3]; [1]Concurrent Technologies Corporation, 27980 Kim Dr., Harvest, AL 35749 USA; [2]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA; [3]Concurrent Technologies Corporation, 13815 Kennard Dr., Glenelg, MD 21737 USA

A unique style of welding tool, based on concepts originated by The Welding Institute (TWI, Cambridge, U.K.), was used in this study to make welds in 25.4-mm 5083-H131 aluminum. A variation of the TWI tool was used, employing a number of flats cut into the pin surface and much finer threads. It was hypothesized that the material flow produced by this pin design was based upon the capture of material by the flats, followed by deposition behind the pin. If this were the case, an important parameter governing the behavior of the pin would be the travel per flat per revolution, a measure of the amount of material captured by each flat on each revolution. Prelminary welding trials were performed to study the characteristics of this pin design in terms of power required, specific energy, torque, weld forces and macrostructure produced.

2:50 PM

Texture Development in Friction Stir Welded 2519 Aluminum: *John F. Bingert*[1]; Richard W. Fonda[2]; [1]Los Alamos National Laboratory, on temporary assignment to Naval Research Laboratory, Matls. Sci. & Tech., Code 6352, 4555 Overlook Ave. SW, Washington, DC 20375 USA; [2]Naval Research Laboratory, Matls. Sci. & Tech., Code 6324, 4555 Overlook Ave. SW, Washington, DC 20375 USA

Friction stir welding produces severe thermomechanical transients that generate crystallographic texture evolution throughout the weld-affected microstructure. In this study, a friction stir weld in a coarse-grained 2519 aluminum plate was investigated in order to resolve the influence of these thermal and deformation effects on texture development. Automated electron backscatter diffraction (EBSD) was applied to spatially resolve orientations in the base metal, weld nugget, and thermomechanical and heat-affected zones. Results show a gradient demarcated by an alteration in boundary character, texture, and precipitate distribution. EBSD scans and microstructural characterizations reveal substructure evolution from the base plate to the nugget indicative of dynamic recovery and recrystallization processes. Comparison of experimental results to those from polycrystal plasticity modeling suggests that texture evolution did not follow that predicted from simple-shear processing assuming restricted glide. Consequences of rigid-body spin and elevated-temperature shear deformation will be explored in relation to their influence on the observed texture.

3:15 PM Break

3:25 PM
Property Characterization of 2024Al/7075Al Bi-Alloy Friction Stir Welded Joints: *John A. Baumann*[1]; Richard J. Lederich[1]; David R. Bolser[1]; Rajesh Talwar[1]; [1]The Boeing Company, Adv. Mfg. R&D, PO Box 516, MC S245-1003, St. Louis, MO 63166-0516 USA

Direct joining of damage tolerant 2XXX alloys with high strength 7XXX alloys offers the promise of fabricating tailor-made structural components. Butt joints in 1" thick plates of 7075-T7351 and 2024-T351 were produced by friction stir welding. Joints were sound and free of voids and root surface disbonds. The joints have a joint efficiency (Fty) that is 80% of the parent metal 2024-T351. Properties are consistent as functions of depth and position along the length of the weld. Tensile failures are always ductile; elongations are typically 5%, which is a consequence of the deformation being localized to the lower strength heat affected zones. The fatigue response (Kt=1.5 and R=0.05) of these weldments was determined in the weld nugget, both heat affected zones, and parent material. The lifetimes were shortest in the heat affected zones, but were at least 1/3 of the parent metal lifetimes.

3:50 PM
Local Properties and Strain Distribution in Conventional and Self-Reacting Friction Stir Welds: *Anthony Peter Reynolds*[1]; Wei Tang[1]; [1]University of South Carolina, Dept. of Mechl. Eng., 300 Main St., Rm. A224, Columbia, SC 29208 USA

In previous work, the authors and others have demonstrated the ability to obtain the local constitutive properties from various regions of friction stir welded joints (e.g the nugget, TMAZ, and HAZ) by combination of a full-field strain measurement technique, a single transverse tensile test, and an assumption of iso-stress loading through the weld. The authors have applied this technique primarily to conventional FSW joints. In this paper, the local strain and property distributions in aluminum alloy friction stir welds made by conventional FSW and by use of a self-reacting or bobbin tool will be compared. The effects of the differing joint geometries will be examined for welds made by the two techniques, but with otherwise equivalent (in so far as is possible) welding conditions. Also, the local properties and strain distributions in transversely loaded joints made using welding conditions enabled by the self-reacting technology will be examined.

4:15 PM
Effect of FSW Parameters on the Structure and Superplastic Formability of AA 2095 SPF Sheet: *Hanadi G. Salem*[1]; Anthony P. Reynolds[2]; Jed S. Lyons[2]; [1]American University in Cairo, Dept. of Mechl. Eng., 113 kasr El Aini St., PO Box 2511, Cairo 11511 Egypt; [2]University of South Carolina, Dept. of Mechl. Eng., Columbia, SC 29208 USA

Joining of aluminum alloys with initial fine grain structures using conventional welding processes results in the deterioration of the properties due to grain growth. Friction stir welding (FSW) has proven its capability of producing sound welds almost defect free; in addition to refinement of the grain structure within the weld zone. FSW was employed to join dynamically recrystallized sheets of Al-Cu-Li base alloy with initial grain size of 2 mm. Microstructural, grain boundary misorientations and texture evolution of the welded sheets were investigated using optical, transmission and orientation image microcopy. The superplastic behavior of the weld nugget in the longitudinal direction was characterized. FSW of the dynamically recrystallized structure was successfully conducted with almost no grain growth throughout the weld. Hardness profiles indicated that hardness variation primarily depend on the particle size and distribution within the weld nugget. Uniform superplastic deformation greater than 400% was retained after FSW.

4:40 PM
Microstructural and Mechanical Evaluation of HSLA-65 and DH-36 FSW: *Maria Posada*[1]; John J. DeLoach[1]; Anthony P. Reynolds[2]; [1]Naval Surface Warfare Center, Carderock Div., Welding & NDE Branch, 9500 MacArthur Blvd., W. Bethesda, MD 20817 USA; [2]University of South Carolina, Mechl. Eng. Dept., 300 Main St., Columbia, SC 29208 USA

Double-pass friction stir welds were produced from Ω-inch thick HSLA-65 and DH-36. The microstructure of the weld nugget was comprised of four regions that include the stir zone, the swirl region within the stir zone, the thermal-mechanically affected zone (TMAZ), and the heat-affected zone (HAZ). Typical hardness plots of DH-36 showed an increase in hardness from the base metal through the HAZ /TMAZ into the stir-zone. The peak hardness corresponded to the swirl region located on the advancing side of the nugget. The hardness profile for HSLA-65 differed dramatically from typical steels evaluated to date. In fact, the hardness profile of HSLA-65 softened in the HAZ/TMAZ region and the peak hardness within the stir zone region was only slightly higher than its base hardness. The peak hardness was 3 times greater in DH-36 than in HSLA-65. HSLA-65 demonstrated greater impact toughness than DH-36. Longitudinal tensile tests were performed on both materials.

5:05 PM
Microstructural Characterization of FSW 7050 Al Alloys: S. R. Sharma[1]; *R. S. Mishra*[1]; J. A. Baumann[2]; R. J. Lederich[2]; R. Talwar[2]; [1]University of Missouri, Dept. of Metallurgl. Eng., 218 McNutt Hall, Rolla, MO 65409 USA; [2]The Boeing Company, Adv. Mfg. R&D, PO Box 516, MC S245-1003, St. Louis, MO 63166-0516 USA

Microstructure of a thick plate FSW 7050Al was characterized to address the issues of strength and ductility variations. The variation of microstructure at various locations in as-FSW and after heat treatment conditions will be presented. The post-FSW heat treatment leads to recovery of strength, but results in ductility problems. The variation of strength and ductility is linked with microstructural changes during heat treatment. The FSW nugget region shows abnormal grain growth. The origin of abnormal grain growth will be discussed.

General Abstracts: Fatigue, Fracture, and Plastic Deformation

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Wednesday PM — Room: 19
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Ellen Cerreta, Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA; Brian Cockeram, Bechtel Bettis Laboratory, ZAP 08D/MT, W. Mifflin, PA 15122-0079 USA

2:00 PM
Correlating Microporosity with Mechanical Properties in Die-Cast AM50 and AM60 Magnesium Alloys: *Gurjeev Chadha*[1]; John E. Allison[2]; J. Wayne Jones[1]; [1]University of Michigan, Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; [2]Ford Motor Company, Matls. Sci. Dept., Dearborn, MI 48124 USA

The role of porosity on the mechanical behavior of die-cast AM50 and AM60 magnesium alloy components has been investigated with special emphasis on ductility. Metallographic and fractographic studies have been conducted on samples with varying volume fractions of porosity to determine the nature of damage initiation during tensile deformation. Surface strain mapping has been performed to determine strain concentrations and subsequent failure in these alloys as a function of porosity size, volume fraction and pore spatial distribution. The results of these studies will be described in this presentation.

2:20 PM
Room Temperature Fatigue and 1000∞C-1400∞C Compression Response of a Mo-2Si-1B Alloy: *Amruthavalli P. Alur*[1]; Sharvan Kumar[1]; [1]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA

The ambient temperature fatigue response of a Mo-2wt%Si-1wt.%B alloy is compared with that of a powder-processed TZM alloy (MT-104). Both, S-N curves and da/dN-DK behavior were assessed; the results demonstrate that the Mo-Si-B alloy is superior in fatigue to TZM. Fracture in the TZM alloy primarily initiates at ZrO2 particles that often result from powder processing. In both materials, fracture is a mixture of transgranular cleavage and intergranular failure. Compression tests were conducted at elevated temperatures (1000∞C-1400∞C) over a range of strain rates (10-4 to 10-7 s-1) and the variation of flow stress with strain rate was determined. Isothermal oxidation studies were conducted in the temperature range 300∞C to 1200∞C and weight gain/loss was measured. Microstructural evolution following high temperature deformation was studied in a transmission electron microscope. Mechanical property results and microstructural observations will be presented and implications will be discussed.

2:40 PM
The Fracture Toughness, Flexural Strength and Shear Strength of Monolithic Silicon Carbide (SiC) Produced by Chemical Vapor Deposition and SiC/SiC Joints Fabricated by a Molybdenum Diffusion Bonding Technique: *Brian V. Cockeram*[1]; [1]Bechtel Bettis Laboratory, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122 USA

The production of robust SiC to SiC joints is required to enable the fabrication of larger and more complicated SiC-based structures. One new technique involves the use of molybdenum foils to develop a diffusion bond with SiC during a simple thermal treatment. In this work fracture strength data for SiC joined using a molybdenum foil diffusion bond is compared with results for monolithic Chemical Vapor Deposited (CVD) SiC. Flexure results are obtained using a standard 4-point flexural test method (ASTM C1161) from room-temperature to 1500C. Shear strength results obtained using a double-notched specimen are also compared for the joined and monolithic specimens. As has been observed for other ceramic materials, the shear strength values for the monolithic and joined specimens were lower than the flexural strength data. Fracture toughness values for monolithic CVD SiC are found to be independent of the CVD growth direction and test temperature from ambient to 1100C. Differences in elastic properties and coefficient of thermal expansion between SiC and phases that are produced in the molybdenum foil bond region result in the formation of slightly larger flaws in the SiC near the joint region. These flaws are shown to produce the fracture initiation sites in both the flexure and shear testing of molybdenum joined SiC. Given the fracture toughness of monolithic CVD SiC is inherently low (about 3.4 MPa m^1/2), the slight increase in flaw size can explain the slightly lower flexural strength values observed for molybdenum bonded SiC. However, the shear strength values for the molybdenum-joined SiC are found to be within the data scatter of the monolithic material.

3:00 PM
Structure and Mechanical Properties of FeAl-WC Prepared by Combustion Synthesis: *Akihiro Matsumoto*[1]; Keizo Kobayashi[1]; Toshiyuki Nishio[1]; Kimihiro Ozaki[1]; [1]National Institute of Advanced Industrial Science and Technology, Inst. for Structl. & Eng. Matls., 2266-98 Anagahora, Shimoshidami, Moriyama-ku, Nagoya, Aichi 463-8560 Japan

FeAl-WC composites have been synthesized by combustion synthesis. Effects of particle size on the structure and mechanical properties of the products were investigated. Pure iron, aluminum and tungsten carbide powders were blended using an automatic agitator and consolidated using pulse current sintering process. As the particle size of iron becomes finer, the structure of products becomes homogeneous. The obtained products consisted of FeAl intermetallic compound and WC. FeAl-50vol%WC has a hardness 800Hv and a transverse rupture strength 2.0GPa. Moreover, wear resistance of FeAl-WC were investigated.

3:20 PM Break

3:30 PM
Microbands and Microtwins Associated with Impact Craters in Copper and Brass Targets: Role of Stacking-Fault Energy (SFE): *Benjamin Gonzalez*[1]; Lawrence Eugene Murr[1]; Olga L. Valerio[1]; Hugo Lopez[1]; Erika Esquivel[1]; [1]University of Texas-El Paso, Dept of Metallurgl. & Matls. Eng., 500 W. University Ave., El Paso, TX 79968 USA

Deformation microbands coincident with {111} slip planes are observed to be associated with impact craters in large-grain (0.8mm) copper (sfe~80 mJ/m2) while microtwins coincident with{111} slip planes are observed in large-grain (~1.1 mm) brass (70 Cu-30 Zn; SFE~10 mJ/ m2). Taken together with other impact crater studies the evidence for a strong influence of SFE in the microband-microtwin transition seems compelling.

3:50 PM
Evaluation of Interfacial Adhesion of Hygrothermally Conditioned Carbon Fibre/Epoxy Composites by Tensile Tests: *B. C. Ray*[1]; [1]National Institute of Technology, Dept. of Metallurgl. & Matls. Eng., Rourkela-769008 India

The mechanical behaviour of interface is an important consideration in the assessment of polymeric composites. The magnitude of the stress transfer from the matrix to the fibre depends on the state of this interface and, in particular, the level of the fibre/resin adhesion. The standard deviations of matrix-dominated properties of such materials are relatively large. The important reason for the sensitive behaviour is due to the hygrothermal ageing of FRP composites. The present study aims to investigate the effects of hygrothermal exposure on transverse and ± 45' tensile properties of carbon/epoxy composites. The test results indicate that it could be the sensitive technique for the assessment of the interfacial adhesion strength in the composite. The degree of degradation of transverse tensile strength with the amount of absorbed moisture is incomparably higher in compare to the variation of ± 45' tensile strength. The quantitative correlation among the various techniques of interfacial adhesion in FRP composites, especially under moist and thermal environments, has yet to be concluded. Many studies demonstrated the importance of interfacial bond in controlling the transverse strength and stiffness of a composite.

4:10 PM
On Another Mechanism for Fatigue Strength Improvement of Metal Parts by Shot Peening: *Mei Yao*[1]; [1]Yanshan University, Col. of Matls. Sci. & Eng., 438 W. Hebei Ave., Qinhuangdao, Hebei 066004 China

Un-peened and shot-peened specimens made of quenched and tempered 300M steel were fatigue tested. The fatigue sources in specimens being tested at stress a little higher than their fatigue limits are located at the surface for un-peened specimens while, for shot-peened ones, in the interior. Obvious change of X-ray diffraction effect of both kinds of specimen being fatigue tested at stress equal to their fatigue limit has taken place in the surface layer for un-peened specimen, while, for shot-peened specimen, in the sub-surface layer. The calculated actual critical stress at the fatigue source position (the ìinternal fatigue limitî) for shot-peened specimen is about 138% of the (surface) fatigue limit of un-peened specimen. Then, the improvement of apparent fatigue limit of shot peened specimen with internal fatigue source is due to the fact that the internal fatigue limit of metal is higher than its surface fatigue limit.

4:30 PM
The Crack Propagation in Generalised Space and the Solid State Deformation Description: *Miklashevich A. Ihar*[1]; [1]Belarusian National Technical University, Theoretl. Mech., Fr. Skaryny Ave. 65, Minsk 220027 Belarus

The crack trajectory is described as a geodesic line of energy of fracture production in the Finsler space associated with the solid state deformation. The geometrical structure (metric properties) of material is determined by the distribution and kinds of defects through the structure of the connection coefficients definition. Three Cartanís tensors of curvature can be introduced independently. This help to describe all sorts of defects correctly. The symmetry group of deformation could be obtained from the fibering of manifolds into horizontal and vertical bundle. The gauge fields could be introduced basing on fibering. Method of determination of the fracture surface fractal dimension is proposed. Fractal dimension of the crack is determined as a ratio of Riemann crack trajectory length to Finsler crack trajectory. It is shown that fractal dimension is independent on the dislocation distribution in materials. Method of the fractal dimension control is proposed.

High Temperature Alloys: Processing for Properties: Cast Alloys and Machining

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee
Program Organizers: Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA; Jacqui B. Wahl, Cannon-Muskegon Corporation, Muskegon, MI 49443-0506 USA

Wednesday PM March 5, 2003 — Room: 13 — Location: San Diego Convention Center

Session Chair: Gerhard E. Fuchs, University of Florida, Dept. of Matls. Sci. & Eng., Gainesville, FL 32611-6400 USA

2:00 PM
Effect of Carbon Additions to Single Crystal Ni-Base Superalloys: *Gerhard E. Fuchs*[1]; *Khalid Al-Jarba*[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

The effect of carbon additions to a model third generation single crystal superalloy was examined. Alloys were processed with 5 different carbon levels, including a baseline without an intentional additions. The addition of carbon to the model alloy resulted in the formation of MC-type carbides and a significant reduction in the number of solidfication defects observed. The effect of these carbon additions on the microstructure, properties, microstructural stability and castability was examined. The microstructure and tensile and creep properties of the samples were examined after conventional solution and aging heat treatments and after long-term thermal exposures, and correlated back to the carbon additions.

2:20 PM
Effect of Solidification Processing Parameters on Microstructures and Properties of CMSX-10: *Gerhard E. Fuchs*[1]; *Elyssa Cutler*[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

A single master heat of CMSX-10, a third generation, single crystal Ni-base superalloy was processed with withdrawal rates ranging from 5cm/hr to 35cm/hr. The as-cast microstructure and solidification partitioning was characterized as a function of withdrawal rate and used to determine the gradient. After heat treatment, the microstructure and tensile and creep properties were determined. The microstructure and properties were correlated back to the withdrawal rate.

2:40 PM
Comparison of Directionally Solidified Castings Using Radiation and Liquid Metal Cooling (LMC): *Andrew J. Elliott*[1]; Tresa M. Pollock[1]; Michael F.X. Gigliotti[2]; Warren T. King[3]; [1]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]GE Corporate Research & Development, Niskayuna, NY 12309 USA; [3]GE Power Systems, Greenville, SC 29602 USA

A columnar grain variant of single crystal RenÈ N4 has been directionally solidified using a simplified geometry with cross-sections similar to an industrial gas turbine blade. Castings were solidified at rate of 2.5mm/min using conventional radiation cooling and at rates between 2.5 and 8.5mm/min using liquid metal cooling (LMC) with tin as a cooling medium. Thermal gradients were directly measured by thermocouples in the casting. The LMC process exhibited higher thermal gradients at all withdrawal rates. Combining high thermal gradients with the capability for faster withdrawal rates produces significantly increased cooling rates. The higher cooling rates result in refined structure measurable by the finer dendrite arm spacing. Additionally the conventionally cast material exhibited several freckles while none where observed in the LMC castings.

3:00 PM
Solution Heat Treatment Reponse of CMSX-4: *Gerhard E. Fuchs*[1]; *Brandon Wilson*[1]; Jennifer Hickman[1]; Ryan Scott[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 11640, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

The standard solution heat treatment of the second generation single crystal Ni-base superalloy, CMSX-4, requires several holds at high temperature. Cost reductions may be possible if the duration and/or temperatures of the holds in solution heat treatment could be reduced. Therefore, this study examines the effects of each step of the solution heat treatment on the microstructure, segregation and phase transformation temperatures of CMSX-4 single crsytal samples. The results of this study were then evaluated with respect to the potential to reduce the solution heat treatment temperatures and hold times.

3:20 PM Break

3:40 PM
The Effect of Solution Heat Treatment on the Microstructure and Properties of CMSX-4: *Gerhard E. Fuchs*[1]; *Brandon Wilson*[1]; Jennifer Hickman[1]; Ryan Scott[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

The mechanical properties of CMSX-4 are critical for its application to gas turbine engines. In order to achieve the optimal balance of properties, the materials must given the appropriate solution heat treatment. Without a solution heat treatment, superalloys would not be able to withstand the stresses or the environment to which they are subjected. Therefore, if the effect solution heat treatments have on mechanical properties could be determined, then the heat treatment step could possibly be shortened. Because of these concerns, research was conducted to observe the effect of solution heat treatments on the mechanical properties. Three sample groups were used for the testing. One group has no solution heat treatment, another has the full solution heat treatment, and the third group has the PWA 1480 solution heat treatment. The PWA 1480 heat treatment consists of lower temperatures and a shorter duration than the standard heat treatment. These results help determine the feasibility of shortening the solution heat treatment step; therefore, reducing production costs.

4:00 PM
Hydrogen Annealing Superalloys for Improved Oxidation Resistance: *James L. Smialek*[1]; [1]NASA Glenn Research Center, Matls. Div., 106-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Hydrogen annealing was used to desulfurize superalloys from nominally 10 ppmw to less than 0.1 ppmw to eliminate sulfur segregation and to improve scale adhesion and cyclic oxidation resistance. A range of sulfur contents was produced for PWA1480 samples by annealing at various temperatures (1000-1300°C), times (8-100 hr), and sample thickness (10-200 mils), as determined by the diffusion parameter, Dt/L2. Performance in 1100°C, 1000 hr cyclic oxidation tests show a distinct dependence of scale adhesion on low sulfur content. Similarly, hydrogen annealing ReneíN5 alloys produced 1150°C cyclic oxidation behavior superior to that of Y-doped alloys, due to desulfurization and decarburization. Finally, H2-annealing PWA1484 samples improved no-bondcoat PS-8YSZ-TBC 1100°C cyclic furnace lives to 1000-2000 hr, compared to 200 hr without annealing. A rule of thumb for maximum scale adherence is obtained whereby the total amount of sulfur available for segregation should be less than about 1 monolayer.

4:20 PM
Thermally Assisted Machining Processes: *Aparna Shilpa Durbhakula*[1]; *Sharada Gollapudi*[1]; Manasa Manga[1]; [1]Vasavi College of Engineering, Dept. of Mechl. Eng., Ibrahimbagh, Hyderabad, AP 500031 India

Advanced materials like ceramics and high-temperature alloys are widely used due to their superior mechanical properties like such as high hardness, thermal stability and wear resistance. They can be fabricated into products by a broad range of manufacturing processes but forming process can produce various shapes with a high production rate, though one has to compromise on dimensional accuracy and surface finish. A finishing operation is hence required. However, many advanced materials are known to be very difficult to machine. Hence, there exists a need for a more efficient machining techniques, particularly for such brittle materials. A promising technique is a thermally assisted machining process, an innovative and economic technique for machining ceramic materials by first softening them with heat from a laser.

Hot Deformation of Aluminum Alloys: Processing, Structure and Property - II

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA; Armand J. Beaudoin, University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; Balasubramaniam Radhakrishnan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6359 USA

Wednesday PM — Room: 6E
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Mike E. Kassner, Oregon State University, Mechl. Eng. Matls. Prog., Corvallis, OR 97331-6001 USA; Zhe Jin, Alcoa Technical Center, Thermomechl. Procg. & Alloy Dvlp., Alcoa Ctr., PA 15069 USA

2:00 PM Invited
Optimization of Sound Castings Aluminum Alloys for Automotive Structural Applications by L.H.I.P.: Marcello Cabibbo[1]; *Enrico Evangelista*[1]; Sergio Gallo[2]; Valentina Latini[1]; [1]University of Ancona, Mech., Via Brecce Bianche, Ancona I-60131 Italy; [2]Teksid SpA, Borgaretto 10040 Italy

The need for weight reduction in the automotive applications is bound in the increasing fuel consuming request due to a reduction need for CO2 pollution. Sand cast aluminum alloys often suffer from voids due to gas entrapped (H2, O2, N2) by the liquid alloy during turbulent filling of the die and from shrinkage during solidification. Both shrinkage voids and gas inclusion pores impair strength and fatigue resistance, as they can easily coalesce to premature fracture. Liquid Hot Isostatic Pressing (L.H.I.P.) process, by the combination of pressure and temperature, is able to close the voids due to shrinkage and drastically reduce the pores due to gases entrapment (H2, N2) through the dissolution of gaseous inclusions within the lattice. This process represents the evolution of H.I.P. (Hot Isostatic Pressing) in which a gas, argon, is used as the pressuring medium. L.H.I.P., presently, constitutes the best valuable solution for obtaining shrinkage-free and pores-minimum castings involving low-cost automotive co nstituents. These production advantages of L.H.I.P. have lead to strong interest in aluminum castings for applications where stringent mechanical requirements must be met. Recent tests on aluminum alloys, processed by L.H.I.P., showed good mechanical response: tensile and yield strength increased of up to 30 pct after L.H.I.P., while ductility tripled. Distribution pores have been investigated and characterized by means of light (LM) and electron microscopy (SEM) techniques. Statistical evaluations on L.H.I.P. processed aluminum alloys for automotive applications (such A356, A357, A319) revealed a pores mean size reduction of up 1/3 through L.H.I.P., the volume fraction being lowered down to one order of magnitude; yet, shrinkage voids were minimized in number and volume.

2:25 PM

The Optimized Tensile and Fatigue Properties of Experimental Semi-Solid Aluminum Alloys: S. C. Bergsma[2]; *M. E. Kassner*[1]; M. Z. Wang[1]; [1]Oregon State University, Mechl. Eng., Rogers Hall, Corvallis, OR 97331-6001 USA; [2]Northwest Aluminum Company, The Dalles, OR 97058 USA

The mechanical properties of various semi-solid formed (SSM) aluminum alloys (experimental designations DF-44, 51 and 95) are reported. These were Al-Si alloys that had various additions of principally Cu and Mg. The SSM parts were formed from ingot that was not stirred during solidification but had undergone a semi-solid thermal transformation (SSTT) during heating to produce a spherical microstructure suitable for semi-solid forming. The T5 and T6 tensile properties were also optimized for these alloys using various solutionizing and aging studies of formed parts. The fatigue properties of some of these alloys were also investigated.

2:45 PM Invited

Changes of Textures, Microstructures and Particles of a Continuous Cast Al-Mg Alloy During Hot Rolling: *Xiyu Wen*[1]; Tongguang Zhai[2]; Zhendong Long[1]; J. G. Morris[2]; [1]University of Kentucky, Ctr. for Al. Tech., 1505 Bull Lea Rd., Lexington, KY 40511 USA; [2]University of Kentucky, Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA

This paper reports an investigation of textures, microstructures and particles of a continuous cast Al-Mg alloy during hot rolling using X-ray diffraction, transmission electron microscopy, scanning electron microscopy and optical microscopy. It was found that the changes in texture during hot rolling reduction were followed by changes in the orientation distribution function. When the rolling reduction was below 38%, the development of the Brass and S texture components were inhibited, while above 38% in rolling reduction they increased sharply. In comparison, the copper component was continuously increased with increase in rolling reduction. Microstructure changes were studied with optical microscopy. The particle size, particle distribution and particle chemical composition, including both intermetallic and dispersion particles, were determined by TEM and SEM. The reasons for the changes in the character of intermetallic particles and dispersoids during hot rolling are discussed and are related to the particular determined texture.

3:10 PM

VIR[FAB], A Joint European Approach Towards Through Process Modelling of Hot Deformation of Aluminium Alloys: *Menno van der Winden*[1]; [1]Corus Research, Development & Technology, IJmuiden Techl. Ctr., PO Box 10.000, IJmuiden 1970 CA The Netherlands

In March 2002, the VIR[FAB] project has started. In this project, all major European Aluminium Producers collaborate with leading universities and research institutes. The objective of this project is to create a so-called Through Process Model (TPM) of the rolling and annealing operations that are part of the fabrication of aluminium sheet. This model predicts the influence of the (upstream) processing parameters on the (final) microstructure. Inevitably, a TPM can only function if the sub-models which constitute it, are linked to each other. The way in which VIR[FAB] has handled this is discussed. Furthermore, the complexity of working on one TPM with 15 different partners brings its own challenges, which will also be discussed. Another aspect that needs attention is the relative balance of efforts between the different sub-models. Typically, the focus has been determined by the specific interests of individuals. Linking the models creates the opportunity to do a comparative sensitivity analysis of the individual sub-models. Additionally, the involvement of so many industrial partners in the project created the opportunity to validate the TPM against full-scale trials on different alloys and different plants. The results of this exercise will be discussed.

3:30 PM

Spray Rolling Aluminum Strip: Process Modeling and Parametric Studies: *J.-P. Delplanque*[1]; S. B. Johnson[1]; E. J. Lavernia[2]; Y. Zhou[2]; Y. Lin[2]; K. M. McHugh[3]; [1]Colorado School of Mines, Eng. Div., Golden, CO 80401-1887 USA; [2]University of California, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616-5294 USA; [3]Idaho National Engineering and Environmental Laboratory, Industl. & Matl. Tech. Dept., Idaho Falls, ID 83415-2050 USA

A joint project, between the Idaho National Engineering and Environmental Laboratory, the University of California-Davis, and the Colorado School of Mines, has been undertaken to investigate the feasibility of the spray rolling process and to evaluate the material properties of spray-rolled aluminum alloys. This investigation is being accomplished in part through modeling based characterization of the spray-rolling process. Models describing the behavior of individual droplets may be integrated and a statistical approach taken to relate process parameters to the state of the spray prior to impact on the rolls. Knowing the condition of the deposited material, the rolling process may then be modeled. Simulations are conducted to explore the process parameter space.

3:50 PM

Spray Rolling Aluminum StripñProcess Development and Strip Properties: *Kevin M. McHugh*[1]; E. J. Lavernia[2]; Y. Zhou[2]; Y. Lin[2]; J.-P. Delplanque[3]; S. B. Johnson[3]; [1]Idaho National Engineering and Environmental Laboratory, Industl. & Matl. Tech. Dept., PO Box 1625, MS 2050, Idaho Falls, ID 83415-2050 USA; [2]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., CA 95616-5294 USA; [3]Colorado School of Mines, Eng. Div., CO USA

Spray rolling is a new manufacturing technology that is under development for producing aluminum strip and other flat products. It consists of atomizing molten metal with a high velocity inert gas, quenching the resultant droplets in-flight, and directing the spray between mill rolls. Hot deformation of the semi-solid material results in the formation of a fully consolidated, rapidly solidified product. Results of a collaborative project between the Idaho National Engineering and Environmental Laboratory, The University of California-Davis, and the Colorado School of Mines to develop spray rolling technology are presented. This paper describes process development issues, and compares the microstructure and material properties of spray-rolled 3003 and 5083 aluminum alloys with commercial material made by conventional ingot/metallurgical processing.

4:10 PM

Rolling Process Optimizations by Sensor Fusion Control Architectures: *George A. Fodor*[1]; [1]ABB Automation Technology Products, Force Measurement, Vasteras SE-72159 Sweden

Process optimization using real-time gray and white physical models is a favorite approach to assess material microstructure and surface properties in aluminum thermo-mechanical processes. Employing a set of models achieve the balance between the process optimization performance goals vs. estimating a large number of physical parameters. Additionally, a large set of sensors ensures that a vector space of appropriate size is available for the estimation of the model parameters. Industrial-grade flexible model-based systems that allow rich sensory equipment for non-laboratory conditions have been recently introduced in industry. The key technological advance that allows this introduction is the use of sensor fusion control architectures. The paper presents a modeling and control approach in hot / cold rolling of aluminum using ABB integrated modeling and geometry sensor equipment. Results show that parameter estimation has high accuracy, however a better model integration could increase substantially the estimated data available from measurements.

4:30 PM

The Effect of Homogenization Practice on the Microstructure of AA6063 Alloys: *Y¸cel B˝rol*[1]; Metin Usta[2]; Gˆkhan Kara[1]; Selda Ucuncuoglu[1]; Osman Cakir[1]; Ayhan N. Bal[3]; Timur Ulucak[3]; [1]Materials and Chemical Technologies Research Institute, Marmara Rsrch. Ctr., TUBITAK, Gebze, Kocaeli 41470 Turkey; [2]Gebze Institute of Advanced Technology, Gebze, Kocaeli Turkey; [3]SARAY Aluminum, G¸neʃli, Istanbul Turkey

Homogenization is an essential step in the preparation of aluminum billets for extrusion and is performed to produce a homogeneous solid solution and to transform the beta-AlFeSi particles to the finer and more equiaxed and thus more acceptable cubic alpha variety. Homogenized billets require lower extrusion pressures and give extrusions with better surface finish and higher strength than as-cast billets. The extrudability of the billet is maximized once the dissolutionizing of the

Mg2Si particles and the bÆa transformation are both complete. The latter takes higher temperatures and longer times and often requires help through revision of the alloy chemistry, i.e. addition of some Mn. The cooling practice in a homogenization treatment is just as significant as soaking and a variety of microstructures ranging from a fully solutionized homogeneous matrix to a heterogeneous one with a coarse dispersion of Mg2Si particles can be produced by simply adjusting the cooling rate. The optimum rate is that which gives a Mg2Si precipitation readily redissolvable during subsequent processing and is dictated by the preheating practice. The present work was undertaken to identify the optimum homogenization soaking and cooling practice for a semicontinous DC-cast AA6063 billet which, due to its composition (very low Mn), relies solely on the soaking practice for the bÆa transformation.

International Symposium on Gamma Titanium Aluminides: High Temperature Properties

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee

Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

Wednesday PM	Room: 6F
March 5, 2003	Location: San Diego Convention Center

Session Chairs: Thomas R. Bieler, Michigan State University, Metall. & Matls. Eng., E. Lansing, MI 48824-1226 USA; Patrick L. Martin, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA

2:00 PM Invited

Elementary Deformation Mechanisms Occuring During Creep of TiAl Alloys at 750∞C: *Couret Alain*[1]; Malaplate Joel[1]; [1]CEMES/CNRS, 29 Rue J. Marvig, BP 4347, Toulouse 31055 France

This presentation is aimed to study the elementary deformation mechanisms occurring during primary and secondary creep stages of TiAl alloys at 750∞C. Two Ti48Al48Cr2Nb2 alloys have been processed by powder metallurgy and casting, leading to the formation of duplex and lamellar microstructures, respectively. They have been subsequently crept at 750∞C under different applied stress. Stress jumps have been performed at different deformation levels in order to measure the activation volumes and the stress exponents. Results indicate that these two activation parameters are constant all over the curve. It is then concluded that primary and secondary stages are controlled by the same dislocation mechanism. The microstructures of crept samples have been investigated by transmission electron microscopy. Up to 3% of deformation, ordinary dislocations dominate the microstructure; they have been found to move by glide and climb. Respective contributions of these glide and climb processes are then discussed.

2:30 PM

Investigation of α_2/γ Phase Transformation Controlling the Creep Deformation Rate in Lamellar TiAl Intermetallics: *Seung Jin Yang*[1]; Hye Jin Jung[1]; Soo Woo Nam[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701 Korea

A lamellar TiAl alloy has been regarded as one of the strongest candidate for structural materials in high temperature use. In recent years, it has been known that the lamellar interface of TiAl alloy has an important role to control the creep deformation rate. In this study, the creep deformation mechanism and the phase transformation process occurred at the alpha2/gamma interfaces are discussed for the investigation of creep mechanism. At the early stage of primary creep α_2-ledges at the alpha2/gamma interface are transformed to gamma phase with the absorption of dislocations and the irregular shape of the interface is changed to be flat. After the early stage, continual α_2 to gamma phase transformation is occurred at the flat interface with the emission of dislocations. This phase transformation involving the emission and absorption of dislocations may control the creep deformation rate and atomistic models of this phase transformation are suggested.

2:50 PM

Influence of Different Texture Aspects on Creep Properties of a Fully Lamellar Ti-47Al-2Cr-2Nb Alloy: *Thomas Marc*[1]; Zghal Slim[2]; Sanchez Claire[1]; Couret Alain[2]; [1]ONERA, DMMP, 29 Ave. de la Division Leclerc, Chatillon, Cedex 92322 France; [2]CEMES-CNRS, 29 Rue J. Marvig, BP 4347, Toulouse, Cedex 4 31055 France

The texture-stress-creep relationships have been evaluated in this two-phase alloy of composition Ti-47Al-2Cr-2Nb (at%) with a fully lamellar microstructure. We have performed a number of mechanical tests on appropriately oriented specimens that have confirmed the texture behavior already assessed in PST-single crystals. This mechanical data base has been used successfully for the interpretation of deformation modes in a fully lamellar microstructure. Additionally, varying the cooling rate was found to significantly alter the decomposition modes of the high-temperature alpha phase, yielding different gamma/gamma interface type distributions. We will review TEM observations highlighting three different mechanisms occurring at different temperature ranges. The related microstructural development made relevant statistical studies of the distribution of individual lamellae to be carried out for the interpretation of lamellar phase transformations. This work aimed at determining if the different sequences of lamellar interfaces would play a significant role in measuring the creep properties in lamellar TiAl-based alloys.

3:10 PM

The Effect of Mo and Al Content on the Creep Deformation of Ti-48Al-2Cr-2Nb-xMo Titanium Aluminides: *Eric A. Ott*[1]; Tresa M. Pollock[2]; Patrick L. Martin[3]; [1]GE Aircraft Engines, Matl. & Process Eng., 1 Neumann Way, MD M89, Cincinnati, OH 45215 USA; [2]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., H. H. Dow 2042, Ann Arbor, MI 48109 USA; [3]Wright Patterson AFB, Matls. & Mfg. Direct., Air Force Rsrch. Lab., Dayton, OH 45433 USA

The creep testing of Ti-48Al-2Cr-2Nb-xMo alloys for x between 0 and 1 atom % has shown that the minimum creep rate is significantly affected by Al and Mo content. Increased Al levels in alloys on the Al-lean side of stoichiometry resulted in decreased creep rates for equiaxed type microstructures. Mo present in solid solution in the gamma phase also resulted in decreased creep rates for these structures. For alloys containing equal to or greater than 0.5 atom % Mo, small amounts of the B2 phase were present in the microstructure, however, the presence and amount of B2 between 0 and 5 volume % did not result in significant creep strengthening. Creep testing of alloys having microstructures which contain other alpha2 morphologies including combinations of lamellar and Widmanstatten alpha2 plates confirmed that the presence of Mo in solid solution also results in decreases in the minimum creep rate.

3:30 PM

Strength Properties of Niobium Containing TiAl Alloys: *Jonathan D.H. Paul*[1]; Uwe Lorenz[1]; Stefan Eggert[1]; Michael Oehring[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str., Geesthacht D-21502 Germany

Titanium aluminides are nearing use in aerospace applications and are already being used in automobile applications. Alloys of industrial relevance must not only show high strength and ductility combined with oxidation and creep resistance but must also exhibit predictable mechanical behaviour. A novel class of alloys that seems to meet the above demands contain large amounts of niobium. However, according to the Griffith criteria, the high strengths obtainable in these alloys require that the minimum defect size be reduced. Therefore good microstructural and chemical homogeneity are required, combined with relatively small colony sizes in fully lamellar materials. Thus, the aim of this paper is to investigate the room temperature mechanical properties of a high niobium-containing alloy in terms of Weibull statistics and thus obtain information regarding material reliability.

3:50 PM Invited

Creep of Directionally Solidified TiAl-Base Alloys and their Tensile Properties: Yuji Omiya[1]; Haruyuki Inui[1]; *Masaharu Yamaguchi*[1]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan

TiAl-base alloys with a lamellar microstructure aligned parallel to the growth direction possess a good combination of mechanical properties. The lamellar orientation of a wide variety of TiAl-base alloys can be controlled through seeding and directional solidification. Mechanical properties of directionally solidified ingots of binary TiAl and TiAl-Si-X alloys have been investigated and, indeed, found that they exhibit an excellent combination of room temperature tensile properties and high temperature strength, in particular, creep strength. In this paper, the results of investigations on the creep and tensile properties of directionally solidified ingots of the binary and multi-component alloys using relatively large-sized specimens are presented.

4:20 PM

Multi-Axial Creep Strength in Gamma-Ti Base Alloys: *Kamran Nikbin*[1]; [1]Imperial College, MED, Exhibition Rd., London SW7 2BX UK

Gamma-Titanium Aluminide were received in four batches, three of which consisted of rectangular blocks and one batch consisted of a section of a cast blade. The batches were of the same nominal chemical composition bu had a wide range of grain sizes, in particular the batch from blade section. Notched bar specimens, with sharp and medium notch acuities were machined and tested at 750°C under static load. In addition compact tension specimens were also tested in order to ascertain the multi-axial failure behaviour of this material. The results are compared with uniaxial results on the same batches. The mode of failure in this material shows that notch strengthening exists for short term tests. However for longer term tests the multiaxial failure shows insensitivity to variation in batch and grain size whereas the uniaxial failure shows a marked reduction in failure lives between their different blocks and blade material. A metallographic examination is undertaken and an effective stress criteria is used to identify the mode of failure in the notched bar specimens.

4:40 PM

Characterization of Gamma TiAl Castings for Engine Blade Component: *Sadao Nishikiori*[1]; [1]Ishikawajima-Harima, IHI, Matls. Dept., Rsrch. Lab., 1 Shin-nakaharachou, Isogo-Ku, Yokohama 235-8501 Japan

In this study, castabilities, weld repairabilities and mechanical properties have been investigated for four engineering TiAl alloys which include 47XD alloy, K5C alloy, and two IHI alloys (TiAl-Mo-V-Si and TiAl-Mo-V-Si-C). Through casting experience of the engine blade component on sub-production scale, castability of each alloy has been evaluated. TiAl-Mo-V-Si alloy shows good component filling. For other alloys, counter gravity casting process is discussed to improve component filling. In order to reduce the cost of castings, development of weld repair technology is investigated. Proper welding of casting cracks should yield increased productivity. The weldability of these alloys is compared along with pertinent welding processes. The mechanical properties of these cast alloys were evaluated in fully lamellar microstructure forms, with particular emphasis of carbon effect on creep resistance. The effect of the carbon addition on the lamellar colony size is also discussed.

5:00 PM

High Temperature Fatigue Evaluation of a Cast TiAl Intermetallic Compound: *Massimo Marchionni*[1]; Giovanni Onofrio[1]; [1]CNR-IENI, Tempe, Via Cozzi, 53, Milano 20125 Italy

Gamma Titanium aluminide alloys are of great interest in high temperature application, due to their low density, high stiffness and satisfactory elevated temperature mechanical properties. The material performance in low cycle fatigue and fatigue crack propagation regimes is very important from a design point of view, together with the evaluation of the thermomechanical fatigue (TMF) behaviour that can describe the material service condition with great accuracy. The paper describes the low cycle fatigue (LCF) and fatigue crack propagation (FCP) behaviour of a duplex gamma TiAl aluminide alloy in the temperature range of 600∞C-800∞C and the aspect of fatigue damage and material degradation. Some tests have been performed in the thermomechanical (TMF) fatigue regimes, assuming testing conditions similar to those of the component in service (diamond cycle, temperature range 300-800∞C, etc.) The fatigue results have been analysed according to Coffin-Manson and Paris relationships and the analysis of fracture surfaces has also been performed in order to evaluate the fatigue damage mechanisms.

5:20 PM

Investigations of the Creep Behavior and the Onset of Microstructural Instability During Short Term Creep in a Gamma TiAl Based Alloy with Fully Lamellar Microstructure: *Anita Chatterjee*[1]; Helmut Clemens[2]; Gerhard Dehm[3]; Christina Scheu[3]; Heinrich Mecking[4]; Eduard Arzt[3]; [1]GfE Metalle und Materialien, Nuremberg 90431 Germany; [2]GKSS Research Center, Geesthacht 21502 Germany; [3]Max-Planck Institut f¸r Metallforschung, Stuttgart 70569 Germany; [4]TU Hamburg-Harburg, Hamburg 21073 Germany

It is well established that the interface spacing in fully lamellar microstructures has a major influence on creep behavior of gamma-TiAl base alloys. In this paper, the dependence of the creep properties on interface spacing in fully lamellar Ti-46.5at.%Al-4at.%(Cr,Nb,Ta,B) sheet material as well as the onset of microstructural instability during short term creep at 800∞C was studied. Creep tests were conducted over a temperature range of 700 to 900∞C and at stresses between 100 and 260 MPa. The results indicate that the primary creep strain as well as the minimum creep rate decrease with decreasing interface spacing. A model based on the work hardening theory was applied to explain the role of the interface spacing on both the primary creep strain and secondary creep rate. The observed microstructural instability during creep is correlated with an Ti_3Al content exceeding the thermodynamical equilibrium due to the formation process of the fully lamellar microstructure.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C. T. Liu: Intermetallics VI–High Temperature Intermetallics I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Seetharama C. Deevi, Philip Morris USA, Research Center, Richmond, VA 23234 USA; Fritz Appel, GKSS Research Centre, Geesthacht D-21502 Germany; Robert W. Cahn, University of Cambridge, Materials Science and Metallurgy, Cambridge CB2 3QZ UK; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Guo Liang Chen, University of Science and Technology-Beijing, State Key Laboratory for Advanced Metals and Materials, Beijing 100083 China; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208-3108 USA; Shuji Hanada, Tohoku University, Sendai 980-8577 Japan; Linda Horton, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6132 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Dong-Liang Lin, Shangai Jiao-Tong University, Shangai 200030 China; T. G. Nieh, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; Masaharu Yamaguchi, Kyoto University, Department of Materials Science & Engineering, Kyoto 606-8501 Japan

Wednesday PM Room: 8
March 5, 2003 Location: San Diego Convention Center

Session Chairs: David G. Morris, CENIM CSIC, Dept. of Physl. Metall., Madrid E-28040 Spain; Guoliang Chen, University of Science & Technology-Beijing, State Key Lab. for Adv. Metals & Matls., Beijing 100083 China

2:00 PM Invited

Laves Phases: Structure, Structural Stability and Polytypic Transformations: *Sharvan Kumar*[1]; [1]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA

Laves phase structures have been examined for their potential as high temperature structural alloys as well as for hydrogen storage. These compounds occur with cubic (3C) or hexagonal (2H, 4H) structures, and their compositional range of existence is apparently dictated by the e/a ratio. According to published phase diagrams of the Cr-X binary systems (X = Ti, Zr, Hf, Nb or Ta), the Laves phase is present in its hexagonal form at high temperatures and in the cubic form at low temperatures. Experimental observations of two-phase alloys in several of these systems demonstrate that the hexagonal structure is often metastably retained at room temperature and subsequent annealing at elevated temperatures transforms the hexagonal structure through a series of polytypes to the stable cubic structure. In two-phase alloys, this transformation is extremely sluggish and is accompanied by a compositional change. Current understanding and issues related to this transformation will be discussed.

2:20 PM Invited

Progress of Advanced Material Science and Technology in China: *Baiyun Huang*[1]; [1]Central South University, Changsha 410083 China

In this paper, the status quo of material science and technology in China has been discussed firstly. In some high-tech research field, for example, nano-materials, artificial crystals, magnetic materials, China has made significant progress. But, in some research field, for example, high performance steel, processing techniques for high quality Al, much work should be done. The affiliation to WTO has provided many opportunities and privileges to Chinaís material industry, but brought many challenges at the same time. In recent years, China has paid much effort to accelerate its progress in material science and technology. It has launched an important programs, National High-Tech Research and Development Programme (863), focusing on high technique research. Material science and technology has been paid much attention in this programme, and is a subdivision of this programme, called National Advanced Materials Program. The organization, priority research field and the progress of some important projects in the National Advanced Materials Program have been described in this paper. As information industry grows very fast, its demands for high performance materials are more and more imminent. An important part of this program is allotted to photoelectronic materials and electronic materials. In order to enhance the competitiveness of Chinaís material industries and material-related industries, much efforts have been paid to R&D of high performance structural materials and functional materials. Considering the technical reserve for future development, nanometer materials and superconductive materials are also under investigation in this program.

2:40 PM Invited

Phase Equilibria and Structure Change in Quasibinary Ni_3Nb-Ni_3M Systems at Elevated Temperatures: *Masao Takeyama*[1]; Mitsuhisa Yamanaka[1]; Akane Suzuki[1]; Yukinori Yamamoto[1]; Takashi Matsuo[1]; [1]Tokyo Institute of Technology, Dept. of Metall. & Ceram. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Ni base A_3B compounds with different crystal structures can be classified into two types in terms of B atom arrangement on the close-packed planes; one is R-type that B atoms set in rectangular array, and the other T-type in triangular array. Ni_3Nb-δ ($D0_a$) belongs to R-type with its superlattice stacking sequence of abab, and others are Ni_3Ti-η ($D0_{24}$:T-type, abac), Ni_3Al-γí ($L1_2$:T-type, abc), Ni_3V-γî ($D0_{22}$:R-type, abcaíbící). Among them, $D0_a$ has the lowest crystal symmetry whereas $L1_2$ phase is the highest. In quasibinary systems of these compounds, some of them has been well established, i.e. Ni_3Al(T)-Ni_3Ti(T). However, as far as R-T combinations are concerned, a very limited number of studies has been reported. In this study, we have mainly studied the phase equilibria between Ni_3Nb(R)-Ni_3M(T or R) systems. The structure change and stability of these compounds including new intermediate phases will be discussed in terms of tetragonality.

3:00 PM Invited

A Study on the Directionally Solidified Nb/Nb5Si3 Composites: *Yafang Han*[1]; Shiyu Qu[1]; Rongming Wang[2]; [1]Beijing Institute of Aeronautical Materials, PO Box 81-4, Beijing 100095 China; [2]Peking University, Physics Dept., Beijing 100871 China

The directionally solidified Nb/Nb5Si3 composites have been fabricated using optical floating zone (OFZ) technology and heat-treated at 1550∞C for 100 hours in Ar atmosphere. The microstructure of the composites has been investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM) equipped with X-ray energy dispersive spectrum (EDS) The results show that the microstructure of the composites consists of consists of dispersed Nb particles and two kinds of niobium silicides matrix, i.e., Nb3Si and Nb5Si3 phases. After heat-treated at 1550∞C for 100 hours, the equilibrium Nb+Nb5Si3 dual-phase microstructure is acquired via a eutectoid reaction, Nb3Si->Nb+Nb5Si3. The room temperature compression property of the directionally solidified Nb-18.7Si (at.%) alloy have been investigated, and compared to those of the Nb-10Si (at.%) and Nb-18.7Si (at.%) alloys fabricated by arc melting. The results indicated that the fracture stress of the directionally solidified Nb-18.7Si (at.%) alloy is as high as 1.9 GPa, which is about 20% higher than those of composites fabricated by arc-melting and no yield has been found. The fracture surface of composites consists of a great amount of like-cleavage facets.

3:20 PM

Interstitial Chemistry and Oxidation Resistance of Ti_5Si_3 and $Ti_5Si_3Z_x$ (Z = C, N, O): *Andrew J. Thom*[1]; Jason J. Williams[2]; Mufit Akinc[3]; [1]Iowa State University, Ames Lab., 142 Spedding Hall, Ames, IA 50011 USA; [2]Arizona State University, Tempe, AZ 85287 USA; [3]Iowa State University, 3053 Gilman Hall, Ames, IA 50011 USA

Changes in the structure of Ti_5Si_3 were measured by x-ray and neutron diffraction as carbon, nitrogen or oxygen atoms were systematically incorporated into the lattice. The measured trends in lattice parameters of Ti_5Si_3 show that most of the previous studies on supposedly pure Ti_5Si_3 were actually contaminated by these pervasive light elements. The oxidation behavior of Ti_5Si_3 was then carefully studied to reconcile inconsistencies observed in the recent literature. Ti_5Si_3 has poor oxidation resistance in air due to formation of a rutile-rich scale and sub-scale phases. In contrast, $Ti_5Si_{3.2}$ has excellent oxidation resistance because of the formation a silica scale. Samples with interstitial oxygen or nitrogen show only slight improvements in the early stages of oxidation compared to Ti_5Si_3, which is in stark contrast to previous research. However, samples with interstitial carbon displayed excellent oxidation resistance, consistent with previous research.

3:35 PM Break

3:50 PM Invited

Anomalous Grain Size Effects During Creep of Some Intermetallics: *K. Sadananda*[1]; C. (Jerry) R. Feng[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 20375 USA

Grain size normally affects creep rates in metals and alloys in the Newtonian Viscous regime. The grain size exponent, p, is normally around 3 for Coble creep involving grain boundary diffusion. Its value is around 2 for Naborro-Herring creep. Intermetallics such as MoSi2 and stoichiometric TiAl show grain size exponents greater than 4. Furthermore, the grain size effects persists even into power-law creep regime. In addition, the governing mechanism depends on the loading history. These anomalies will be discussed in light of the existing theories of creep.

4:10 PM

ìFe Effectî on Environmental Embrittlement of NiTi Alloys: *Jiahong Zhu*[1]; Chain T. Liu[2]; [1]Tennessee Technological University, Dept. of Mechl. Eng., 115 W. 10th St., Box 5014, Cookeville, TN 38505 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831 USA

Environmental effect on mechanical behavior of intermetallic alloys has been an ongoing research topic for over a decade. Even though significant progress has been made in this area, there are still many challenges to be answered. Recently, a unique ìFe effectî on environmental embrittlement in NiTi alloys was discovered in our laboratory. It was shown that there exists a critical Fe content in NiTi, i.e., about 9 at.%, below which no environmental embrittlement was observed and above which hydrogen from test environments severely embrittled NiTi. The mechanism responsible for the observed ìFe effectî was addressed in this presentation. Hydrogen diffusivity measurements from hydrogen-charging experiment show that Fe addition reduces the hydrogen diffusivity in NiTi, implying that the observed ìFe effectî is not due to reduction of hydrogen diffusion in the material. Also, laser desorption mass spectrometric results indicate the kinetics of surface reaction to generate atomic hydrogen does not change very much with Fe additions. Based on the experimental evidences, it is proposed that the existence of a critical Fe content is closely related to the effect of Fe on the critical hydrogen concentration to induce hydrogen embrittlement in NiTi. This research was sponsored by the Division of Materials Sciences, US Department of Energy, under contract DE-AC05-96OR22464 with UT-Battelle, LLC.

4:25 PM

Effects of Microstructure on the Oxidation Behavior of Mo-Rich Mo-Si-B Intermetallics: *Voramon Supatarawanich*[1]; David R. Johnson[1]; Chain T. Liu[2]; [1]Purdue University, Matls. Sci. & Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907-1289 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS6115, Oak Ridge, TN 37831-6115 USA

The effects of volume fractions and morphologies of the Mo, Mo3Si, and Mo5SiB2 (T2) phases on the oxidation behavior of the multiphase alloys at 600-1300C were examined. The alloys were cast and heat treated at 1600C before the cyclic oxidation tests. The multiphase alloys showed poor oxidation resistance at 800C while a protective glass scale formed at 1300C. At the oxide/matrix interface, a Mo layer was found adjacent to the T2 phase while a Mo5Si3 interfacial layer was found adjacent to the Mo3Si phase. The alloy with 50 vol.% bcc-Mo solid solution formed a protective scale at 1300C. However, the specimens with the highest B/Si ratio (the lowest volume fraction of Mo3Si) did not form a protective glass scale at 1300C. The results indicate that, the Mo3Si phase is an important source of silicon for controlling the composition of the protective glass scale.

4:40 PM

Effects of Environment and Temperature on the Mechanical Behavior of the Ni-19Si-3Nb Intermetallic Alloy Doped with Boron and Carbon: *Shian-Ching Jang*[1]; [1]I-Shou University, Dept. of Matls. Sci. & Eng., 1, Sec. 1, Hsueh-Cheng Rd., Ta-Hsu Hsiang, Kaohsiung Co. 84008 Taiwan

The effect of the small additions of boron and carbon on the environment embrittlement at different temperature of the Ni-19Si-3Nb based alloy was investigated by atmosphere-controlled tensile test in various environments. The results revealed that the Ni-19Si-3Nb base alloy performs very ductile mechanical behavior (UTS ~1250 MPa, e ~14%) at room temperature in vacuum (2 x 10®C4 torr) as well as in pure oxygen atmosphere. This indicates that the environmental embrittlement of water vapor is the major factor to deteriorate the ductility of Ni-19Si-3Nb base alloy. In parallel, the effect of 300 ppm boron addition improves the ultimate tensile strength and the ductility (UTS ®R 1200 MPa, e ®R 12%) in the Ni-19Si-3Nb base alloy significantly at room temperature in various atmosphere, such as vacuum, pure oxygen, air (contains 14000 ppm water vapor), and pure water vapor (contains 850 ppm water vapor). In addition, the yield strength of the Ni®C19Si-3Nb-0.15B-0.1C alloy was revealed to exhibit an increasing trend of temperature dependence, and the maximum yield strength occurs at 600°Ê. However, the ductility of the Ni®C19Si-3Nb-0.15B-0.1C alloy drops obviously when the temperature increases to 700°Ê. In addition, the observation of fracture surface for the specimen tested at different temperature revealed a transition temperature of fracture mode between 600 °Ê to 700 °Ê. The fracture mode changes from ductile dimple fracture mode into the brittle cleavage fracture mode.

4:55 PM

Synthesis of Titanium Nitride Nano-Particles by Reduction of TiCl4 in Liquid Ammonia: *Hongmin Zhu*[1]; Boyan Yuan[1]; Gensheng Sun[1]; Zhangmin Cao[1]; [1]University of Science & Technology Beijing, Dept. of Physl. Chem., 30 Xueyuan Rd., Beijing 100083 China

In the media of liquid ammonia, titanium tetrachloride was reduced by sodium dissolved in liquid ammonia. The product of the reaction was nano-sized fine powder and sodium chloride dissolved in the solvent. The nono-powder was heated under different pressure, and crystalline titanium nitride were obtained. The particle size was in the range of some ten non-meters varying with the heat temperature. The product powder was analyzed by different method, and the component ratio of titanium to nitrogen in the powder was around 1.

5:10 PM

Mechanical and Structural Properties of Aluminum Doped Mo5Si3: *Pedro D. Peralta*[1]; W. Hernandez[1]; Thomas Groy[2]; Raul Coltters[3]; [1]Arizona State University, Dept. of Mech. Eng., Main Campus, MC 6106, Tempe, AZ 85287 USA; [2]Arizona State University, Dept. of Chem. & Biochem., Main Campus, MC 1604, Tempe, AZ 85287 USA; [3]Universidad Simon Bolivar, Dept. de Ciencia de Materiales, Caracas, DF Venezuela

Mo5(Si,Al)3 alloys with 3, 5, 7 and 10 at% Al (nominal) were prepared using arc-melting. Microstructural characterization was carried out using Scanning Electron Microscopy, and the lattice structure was studied using X-ray power diffraction. The results indicate that a second phase appears after a nominal composition of 7 at% Al and that the lattice parameters in the solid solutions increase with Al additions. Microhardness was used to characterize the mechanical properties as a function of composition. The hardness was found to decrease with increasing Al additions; however, the presence of extensive micro-cracking is still observed for all compositions. This suggests that Al does not contribute substantially to reduce the thermal expansion anisotropy in Mo5Si3, which is the main cause of post-processing cracks in this material. An alloy with 3 at% Nb and 5 at% Al presented drastically reduced micro-cracking, confirming previous literature reports on the beneficial effects of Nb additions.

5:25 PM

Glass Transition and Crystallization of $Mg_{75}Ni_{10}Nd_{15}$ Metallic Glass Studied by Temperature-Modulated DSC: *Z. P. Lu*[1]; C. H. Kam[2]; Y. Li[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6115 USA; [2]National University of Singapore, Dept. of Matls. Sci. 119260 Singapore

Glass transition and crystallization behavior of $Mg_{75}Ni_{10}Nd_{15}$ metallic glass has been systematically studied by a temperature-modulated differential scanning calorimeter (TMDSC). The truly vitreous state of as-spun $Mg_{75}Ni_{10}Nd_{15}$ alloy was confirmed by the direct observation of glass transition behavior for as-spun and isothermally annealed ribbons through TMDSC measurements. It was concluded that the glass transition on normal differential scanning calorimeter (DSC) heating curves was hidden due to the relatively strong heat release of the primary nanocrystallization of intermetallic Mg_3Nd phase. This was further verified by the fact that the comparable glass transition behavior was observed by both TMDSC and DSC for final residual amorphous matrix in which the primary nanocrystallization has finished. The $Mg_{75}Ni_{10}Nd_{15}$ metallic glass was confirmed to crystallize in a primary crystallization m ode, e.g. Amó>Am[i] + Mg_3Ndó>an unknown phase + Mg_3Nd. Clear observation of structural glass transition and full understanding of crystallization micromechanism in such alloys can offer us new alloying and processing strategies for the preparation of bulk glassy alloys (e.g. consolidation route) and development of new nanocrystalline/amorphous composite materials.

International Symposium on Structures and Properties of Nanocrystalline Materials: Phase Transformation

Sponsored by: Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee

Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Wednesday PM Room: 14B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Michael Atzmon, University of Michigan, Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; Vijay K. Vasudevan, University of Cincinnati, Matls. Sci. Dept., Cincinnati, OH 45221-0012 USA

2:00 PM

Transformation Behavior in Nanoscale Powders of Binary Aluminum-Copper Alloys: *Jixiong Han*[1]; Jai A. Sekhar[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Matls. Sci. Dept., PO Box 210012, Cincinnati, OH 45221-0012 USA

A study was made of the synthesis, microstructure and transformation behavior of nanoscale Al-Cu alloy particles. Nanoparticles were synthesized by plasma ablation from Al-4.4 wt.% Cu ingots. These particles were exposed to air and formed a 3-5 nm thick oxide scale. The particles were found to be in the supersaturated state displaying a variation in the individual particle composition. Several of the particles were faceted and bound by (111) planes. The nanoparticles were heat treated to examine the precipitation sequence. At the beginning of transformation, precipitates that were considerably enriched in copper were observed; these reduced in copper content as the heat treatment progressed. These precipitates were observed to form along the aluminum oxide-particle interface. Details of the precipitation sequence, nature and structure of second phase precipitates and interphase interfaces and formation mechanisms will be reported. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

2:20 PM

Size-Dependent Characterization of Gas Atomized Amorphous AlñGd-Ni-Fe Powders: *Baolong Zheng*[1]; Yizhang Zhou[1]; Enrique J. Lavernia[1]; [1]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616 USA

Great interest in the development of Al alloy systems is generated after the finding of aluminum based amorphous alloys with an Al content above 80 at. %, which exhibited tensile strengths of over 800 MPa as well as good ductility. This research is aimed at characterizing the amorphous formation ability of the atomized Al-7at.%Gd-2at.%Ni-1at.%Fe alloy powder. Based on experiment, an Al-7at.%Gd-2at.%Ni-1at.%Fe alloy was gas atomized with different gas atomization pressure and different atomization gas mixture, and its microstructure, thermal stability, transformation sequence and Micro-hardness are investigated with SEM, DSC and XRD as a function of powder particle size. With decreasing powder particle size, the volume fraction of amorphous materials increase, and micro hardness also increase. Powder with <25 m particle size can be considered fully amorphous, and 25-38 consisted of FCC-Al nano-crystals embedded in an amorphous matrix. The high thermal stability of the amorphous + fcc-Al nanostructured state and the high hardness of this fine powder make it a good candidate for consolidate bulk material for technological applications.

2:40 PM

Production and Characterization of Metal-C Composites (Where Metal=Al or Fe and C=Graphite or Fullerene) Obtained from Mechanically Alloyed Powders: *F. C. Robles-Hern·ndez*[1]; H. A. Calderon[2]; V. Garibay-Febles[3]; M. Umemoto[4]; [1]University of Windsor, Formerly at the Dept. de Ing. Met. ESIQIE-IPN, MÈxico, Windsor, Ontario Canada; [2]ESFM-IPN, Dept. de Ciencia de Materiales, MÈxico, D.F. MÈxico; [3]Istituto Mexicano del Petroleo, Formerly of the Dept. de Ing. Met. ESIQIE-IPN, Mexico, MÈxico, D.F. MÈxico; [4]Toyohashi University of Technology, Dept. Production Sys., Toyohashi 441-8580 Japan

This paper summarizes the results obtained with regards to the production and characterization of composites (Al or Fe) 15%at.C (where C is either graphite or fullerene (C_{60}+C_{70})). These composites were produced by using mechanical alloying techniques (horizontal and SPEX) under inert atmosphere. As-milled powders were compacted by using spark plasma sintering (SPS) at 773K, for 10min. and under 50MPa. SPS allowed a moderate grain growth from an average size of ~50nm to ~100nm. After sintering all systems reacted to produce carbides except for Fe-$C_{fullerene}$ that was rolled at 1273K. After rolling, fullerene showed a novel crystalline structure (tetragonal-I4/mmm). Several techniques like X-ray diffraction, scanning and transmission electron microscopy, microhardness and porosity-density measurements were performed in order to characterize microstructure and physical properties of the composites. Microhardness results from the Al-$C_{fullerene}$, and Fe-$C_{fullerene}$ before and after sintering increased from 50 to 300 HV and from 100 to 722 HV, respectively.

3:00 PM

Thermal Plasma Processing of SiC Powders: *Leo V.M. Antony*[1]; Jonkion M. Font[1]; Ramana G. Reddy[1]; [1]University of Alabama, Metallurgl. Eng., Box No 870202, Tuscaloosa, AL 35487 USA

Thermal plasma processing of fine powders has been the subject increasing interest over the past years. Its potential over the production of advanced materials is highly recognized. In this study, synthesis of ultra fine powder of SiC using non-transferred arc plasma has been studied. The raw silicon oxide powder and methane gas were used as the starting materials. The experiments were carried out at different power levels of the plasma reactor. Based upon the Gibbs energy minimization method the molar ratio of SiO2/CH4 was fixed for the system. The effect of the particle size on the vaporization time of the particle was studied. Scanning electron microscope (SEM), energy-dispersive X-ray spectrometer (EDS) and X-ray diffraction analysis were used for products characterization. The results showed that ultra pure fine spherical shaped SiC particles are produced.

3:20 PM

Experimental and Modeling Study of Cooling Rates for Atomizing Amorphous Al-Gd-Ni-Fe Powder: *Baolong Zheng*[1]; Yaojun Lin[1]; Yizhang Zhou[1]; Enrique J. Lavernia[1]; [1]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616 USA

In order to obtain amorphous alloy, the melt is rapidly cooled into a solid state without crystallizing. Cooling rate is one of the most important parameters for making metallic glass. Based on experiment, analyses of atomized 2024 Al powders are presented. Efforts were focused on determination of secondary dendrite arm spacing (SDAS), cooling rate, and their correlation with powder size. Characterization of powders includes surface and interior morphology, particle size distribution, dendrite arm spacing and cooling rate. The work is aimed at simulation of cooling rate in atomizing amorphous Al-7at.%Gd-2at.%Ni-1at.%Fe alloy powder. The calculation for cooling rate modeling is based on Newtonian cooling with forced convection. The energy balance, gas dynamics, droplet dynamics, properties of the model alloy and atomization gas, and heat transfer between gas and droplet are considered in modeling calculation. The modeling results are helpful for insight understanding the process of atomizing amorphous Al-7at.%Gd-2at.%Ni-1at.%Fe alloy powder.

3:40 PM

Transformation Behavior in Nanoscale Powders of Binary Aluminum-Zinc Alloys: *Martin J. Pluth*[1]; Jai A. Sekhar[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Matls. Sci. Dept., PO Box 210012, Cincinnati, OH 45221-0012 USA

A study was made of the synthesis, microstructure and transformation behavior of nanoscale Al-Zn alloy particles. Nanoparticles were synthesized by plasma ablation from Al-15 wt.% Zn ingots. The particles were found to be in the supersaturated state, but displayed variation in composition compared with the bulk alloys. These particles were exposed to air and formed a 3-5 nm thick oxide scale. Several of the particles were faceted and bound by (111) planes. The alloy nanoparticles were heat treated to examine spinodal decomposition and/or precipitation. A spinodal structure was observed, as were precipitates of pure zinc with an f.c.c. structure. The precipitates were seen to form along the aluminum oxide-particle interface. Details of the precipitation sequence, nature and structure of second phase precipitates and interphase interfaces and formation mechanisms will be reported. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

4:00 PM

Processing Bulk Structures from Nanoscale Powders of Aluminum: *Martin J. Pluth*[1]; Jixiong Han[1]; Jai A. Sekhar[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Matls. Sci. Dept., PO Box 210012, Cincinnati, OH 45221-0012 USA

A study was made of the compaction, sintering behavior and workability of nanoscale aluminum powders into bulk structures. The nanoparticles of pure Al ranged in diameter from ~30 to 150 nm and contained a 2-5 nm outer oxide layer. The powders were cold-compacted in air, then sintered between 400 and 600°C. Both hot and cold rolling of the pressed pellets was utilized to assess workability and hardness after processing. Density was measured, and thin foils were prepared for TEM. In some cases, the oxide films surrounding the nanopowders were breached, resulting in grain growth and texturing after rolling. However, many of the nanoscale oxides did not break during processing and a very fine grain structure resulted. Similar studies on nanoscale powders of binary Al-Cu and Al-Zn were also performed. Results of the sintering and consolidation behavior of pure Al and alloy nanopowders will be presented. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

4:20 PM

Threading Dislocation Deduction by Nanometer Scale Lateral Epitaxy Overgrowth of GaN Over SiNx Nanomask Employing Block Copolymer Lithography: *Wei Zhou*[1]; [1]University of Southern California, Dept. of Matls. Sci., Compound Semiconductor Lab., Los Angeles, CA 90089 USA

Hexagonally ordered arrays of nanoscale holes were defined on thin SiNx film covered GaN buffer surface employing block copolymer lithography. Selective nucleation of GaN inside the predefined nanoscale holes was carried out followed by nanoscale lateral epitaxy overgrowth. Coalescence of surface was achieved within a thickness of 200nm, resulting in a smooth GaN surface. We demonstrated here that lateral epitaxy overgrowth of GaN in nanometer scale distinguishes itself from that in much larger scale in the sense that the migration has to be subdued instead of enhanced at the initial stage of lateral overgrowth. TEM observation shows that this approach may provide a promising path for GaN threading dislocation deduction uniformly across the sample surface.

4:40 PM

Electrodeposited Magnetic Nanostructures: *Vladimir Grigoríevich Shadrow*[1]; Lyudmila Vasilíevna Nemtsevich[1]; Anatolyi Vasilíevich Boltushkin[1]; [1]Academy of Sciences, P. Brovki, 17, Minsk 220072 Belarus

Growth processes,structure peculiarities and magnetic properties of electrodeposited magnetic nanostructures have been investigated by means of EM, XRD, AFM, VSM and AGFM. Regularities of content modulated FeCu and CoCu nanophase particles formation in the pores of aluminium anodic oxide and a mechanism of nanocrystalline structure formation in continuous Co based films are discussed as well as crystallized film structure formation process peculiarities. Intergranular magnetic interaction and magnetization reversal processes in the above structures are reported through remanence and delta M curves analysis and rotational hysteresis loss and angular variations of hysteresis parameters measurements.

5:00 PM

Nanostructured Titanium Doped Iron Oxides Prepared by Laser Pyrolysis: F. Dumitrache[1]; *Rodica Alexandrescu*[1]; I. Morjan[1]; I. Sandu[1]; I. Soare[1]; I. Voicu[1]; E. Vasile[2]; R. Piticescu[3]; [1]National Institute for Lasers, Plasma and Radiation Physics, PO Box MG 36, R-76 900, Bucharest Romania; [2]S.C. METAV S.A, Str. Zapada Mieilor 16-18, Bucharest Romania; [3]S.C. IMNR S.A., Bd. Biruintei 102, Bucharest Romania

The chemical, magnetical and electrical properties of nanostructured oxides are extremely sensitive both to the huge surface/volume ratio of nanoparticles and to the synthesis path employed. Titania doped iron oxide nanostructures are expected to promote increased sensitivity when acting as sensing or catalyst devices. This work aims to investigate the production and characterization of titania doped iron oxides nanopowders using the IR laser pyrolysis from a gas phase mixture containing several basic components: vapors of Fe(CO)5/TiCl4, an oxidizing agent (usually air) and a sensitizer (either Sf6 or C2H4). The pyrolysis is based on the resonance between the emission line of a CO2 laser and the infrared absorption band of the sensitizer component (acting like an energy transfer agent. Several analytical methods (TEM, XRD, IR and Raman spectroscopy, chemical determinations, thermal analysis) were used for the characterization of particle composition and morphology. Titanium iron oxide doping was achieved in the range 0.5-1.5 wt%. It was found that the initially mainly amorphous g-Fe2O3 transforms at about 245C in crystalline g-Fe2O3 and further, at about 595C, in crystalline a-Fe2O3.

Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging: Creep and History-Influenced Deformation Processes

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee

Program Organizers: J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Srini Chada, Motorola, Department APTC, Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Jud Ready, MicroCoating Technologies, Atlanta, GA 30341 USA; Jin Yu, KAIST, Center for Electronic Packaging Materials 305-701 Korea

Wednesday PM	Room: 15B
March 5, 2003	Location: San Diego Convention Center

Session Chairs: Jin Yu, KAIST, Ctr. for Elec. Pkgg. Matls., Daejon 305-701 Korea; Jud Ready, MicroCoating Technology, Atlanta, GA 30341 USA

2:00 PM Invited

The Anomalous Creep Properties of High-Sn Solders: *John William Morris*[1]; Ho Geon Song[1]; [1]University of California, Dept. Matls. Sci., 555 Evans Hall, Berkeley, CA 94720 USA

Creep tests on solder joints of Sn-rich, Pb-free solders show anomalies in creep behavior at temperatures near room temperature. The anomalies include a strong temperature dependence of both the stress exponent and the apparent activation energy. The anomalies appear to have their source in the behavior of the Sn consitutent itself. The microstructures of these solders are, primarily, Sn, and test joint of pure Sn show the same anomalies. The present paper discusses this anomalous behavior in terms of the creep behavior of pure Sn and the microstructures of the solder joints.

2:25 PM

The Constitutive Creep Equation of Eutectic Sn-Ag Alloy Using the Modified Theta Projection Concept: *Yoshiharu Kariya*[1]; Masahisa Otsuka[2]; William J. Plumbridge[3]; [1]National Institute for Materials Science, Eco-Matls. Ctr., Namiki 1-1, Tsukuba, Ibaraki 3050044 Japan; [2]Shibaura Institute of Technology, Matls. Eng. & Sci. Dept., Shibaura 3-9-14, Minato-Ku, Tokyo 1088548 Japan; [3]The Open University, Matls. Eng. Dept., Walton Hall, Milton Keynes MK7 6AA UK

Creep of solder alloys is generally analyzed by relating the steady state strain rate to the stress through a power law relation such as the Norton equation. However, the power law relations may not be suitable to describe the strain-time relationship of solders, as the creep of most of solder alloys consists of only primary and accelerating stages. In this study, creep data of eutectic tin-silver alloy at temperatures between 298K and 398K has been analyzed using the modified theta projection concept instead of the steady state creep constitutive equation. The constitutive equation is composed of one rate constant representing the reciprocal of relaxation time and two different strain factors representing work hardening and weakening. The equation well describes the creep curves of the eutectic tin-silver alloy up to tertiary stage. The advantages of the method, and the stress and temperature dependency of the materials constants will be presented.

2:45 PM

Creep Behavior of Sn-Ag/Cu Solder Joints at Small Length Scale: *Matthew Kerr*[1]; Jason J. Williams[1]; Nikilesh Chawla[1]; [1]Arizona State University, Cheml. & Matls. Eng., Mechl. Behaviors of Matls. Fac., Tempe, AZ 85287-6006 USA

In order to adequately characterize the behavior of solder balls in electronic devices, the mechanical behavior of solder joints needs to be studied at small-length scales. This study examined the creep behavior of Sn-Ag/Cu solder joints at room temperature and elevated temperatures. Lap shear testing of single solder ball joints was conducted using a microforce testing system. Microstructure characterization and fractography were conducted to elucidate the governing creep mechanisms. A change in the creep exponent with increasing stress was observed and will also be discussed. Research supported by the National Science Foundation (Program Manager: Dr. K. L. Murty, contract# DMR-0092530).

3:05 PM

Effects of Surface Finishes on the Creep and Low Cycle Fatigue Characteristics of Sn Based Solder Joints: S. W. Shin[1]; *K. O. Lee*[1]; Jin Yu[1]; [1]Korea Advanced Institute of Science and Technology, Ctr. of Elect. Pkgg. Matls., 373-1 Gusong-dong, Yusong-gu, Daejon 305-701 S. Korea

According to Kang et.al.[1], different surface finish layers on PCB laminates gave different dissolution rate of the surface finish layers and varying thickness of intermetallic compounds at the soldering interface, which ultimately affect the microhardness in the BGA solder joints and possibly their reliability. In this study, effects of surface finish layers on the creep and low cycle fatigue resistance of Sn-based solder joints have been investigated. Lead-free Sn-3.5Ag-XCu(X=0 and 0.75), Sn-3.5Ag-2.5Bi and Sn-0.7Cu alloys were prepared as solder balls, and two types of surface finishes including combinations of Cu and Au/Ni/Cu on opposite sides of the BGA balls were investigated. Firstly, single lap shear creep tests were conducted at 373K under 5-11Mpa, and then lap shear fatigue tests were done under controlled total displacement strokes, 10, 12, 15 and 20 μm at room temperature. Microstructures of reflowed solder balls showed dense dendritic structures similar to those of solder balls with the quenching rate of 145°C/sec. Shear strain rates of lap shear specimens were typically 2-3 orders lower than those of bulky specimens and affected by the surface finish conditions of the PCB laminates. Usually, Sn-3.5Ag and Sn-3.5Ag-0.75Cu alloys showed best creep resistance, while Sn-3.5Ag-0.75Cu, Sn-3.5Ag and Sn-0.7Cu alloys showed longest fatigue life. Bi containing alloys showed the worst fatigue resistance due to the fatigue propagation along the intermetallic/solder interfaces. Another points addressed in the paper include the effects of solder ball size on the microstructure of solder balls and mechanical reliability. [1]S.K. Kang, W.K. Choi, D.Y. Shih, P. Lauro, D.W. Henderson, T. Gosslin and D.N. Leonard, 52nd ECTC Proc. P.146(2002).

3:25 PM

Microstructural Effect on the Creep Behaviors of a Lead Free Sn-3.5%Ag Alloy and its Joint to Cu Wire: *Kazuya Miyahara*[1]; [1]Nagoya University, Dept. of Molecular Design & Eng., Furoh-cho, Chikusaku-Ku, Nagoya, Aichi-Plef. 464-8603 Japan

Three kinds of microstructure of a lead free Sn-3.5%Ag alloy are obtained by altering the cooling rate of its ingot cast into iron mold and the effect of these microstructure on the creep behaviors of the alloy have been investigated. Rapidly cooled material (cooling rate: 8.0 C/sec) showed the finest microstructure, which is composed of the particles of Ag3Sn and Sn matrix, and also had the largest creep strength. Microstructure of the material cooled at the lowest rate (0.016 C/sec) is composed of the plate like phase of Ag3Sn and Sn matrix and this indicated the smallest creep strength which is smaller by about two orders of magnitude than that of the rapidly cooled material. The material cooled at the medium rate (2.0 C/sec) indicated a medium creep strength among these three materials. The effect of microstructure on the creep behaviors of the solder joint part of Sn-3.5%Ag alloy and Cu wire were also investigated.

3:45 PM Break

3:55 PM Invited

Resonant Vibration Behavior of Lead-Free Solders: *Jenn Ming Song*[1]; Truan Sheng Lui[1]; Li Hui Chen[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Eng., Tainan 701 Taiwan

Failure of solder joints may occur due to vibration, particularly when local or general resonance happens. Therefore, the vibration fracture resistance should be taken into consideration in the development of lead-free solders. This study investigated the resonant-vibration characteristics of some potential lead-free solders, including Sn-Zn, Sn-Ag and Sn-Bi alloys. The effects of the third element were also examined. Results show that Sn-Ag eutectic alloy exhibits a higher vibration life than Sn-Zn and Sn-Bi under a fixed vibration force. This is closely related to the damping capacity and crack propagation resistance of the materials. Also, the striated deformation in Sn-rich phase plays an important role in absorbing vibration energy. The morphology and distribution of the compounds also affect the vibration behavior significantly. Moreover, microstructural modification can be achieved through doping with the third element and thus the damping capacity and vibration fracture resistance can be improved.

4:20 PM
Effects of Copper Content on Vibration Fracture Behavior of Sn-Ag-Cu Solders: Fang I. Li[1]; *Jenn Ming Song*[1]; Truan Sheng Lui[1]; Li Hui Chen[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Eng., Tainan 701 Taiwan

Considering applications under vibrating conditions, the vibration fracture characteristics of solder materials are worthy of notice to improve reliability. This study investigated the resonant vibration fatigue of Sn-3.5Ag-Cu alloys, which have been developed as a potential lead-free solder, especially in the copper content effect. The copper content of the samples ranges from 0wt% to 1.5wt%. Results show that the vibration life of the test samples decreases in turn from the 0Cu, 0.5Cu to 1.0Cu sample. This can be attributed to refined Sn-rich dendrites which lead to a lower damping capacity and higher deflection amplitude thus produced. Itís significant to note that the 1.5Cu sample possesses the highest damping capacity and vibration fracture resistance. The presence of numerous massive Cu6Sn5 dispersoids may account for this phenomenon.

4:40 PM
Effects of Cooling Rate on Tensile and Creep Behavior of a Sn-3.5Ag Lead-Free Solder: *Felipe A. Ochoa*[1]; Jason J. Williams[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA

The mechanical behavior and microstructure of bulk, pure Sn-3.5 Ag solders as a function of cooling rate was studied. Creep tests were performed at constant stress at ambient temperature, 60°, 90°C and 120°C. Cooling rate was found to significantly affect the secondary dendrite arm spacing of the Sn-rich phase and the aspect ratio of Ag_3Sn. The changes in the microstructure induced by the cooling rate significantly affected the mechanical behavior of the solder. Scanning and transmission electron microscopy were used to characterize the effects of cooling rate on solder microstructure, as well as to understand creep deformation mechanisms in the solder. Creep mechanisms will be presented and discussed in terms of the creep stress exponent and the solder microstructure. Research supported by the National Science Foundation (Program Manager: Dr. K.L. Murty, contact# DMR-0092530).

5:00 PM
Reliability Investigation on Lead-Free Chip Scale Packages: *Shyh-Rong Tzan*[1]; Chien-Yu Hsu[1]; [1]Materials Research Laboratories, ITRI, Metallic Devices for Elect. Lab., 195-5 Chung-Hsing Rd., Sec. 4, Chutung, Hsinchu 310 Taiwan

Environmental friendly production has become worldwide trend recently. The importance of the lead-free soldering technologies has gathered the attention of the organizations, industries, and regulators. The alternative materials and the possible risk of the reliability problems for the lead-free products have been a major concern for the industries. In this paper, one of the major considerations, the reliability issue has been investigated by evaluating the material properties and the soldering performance. A set of chip scale packages will be focus and discussed. The thermal cycle and mechanical cycling tests have been used to examine the reliability performance of a few types of lead-free solder paste as well as the tin-lead solder paste. The thermal aging and the intermetallic effects are studied to characterize the properties of the lead-free solder pastes and packages. The weibull distribution model is also used to evaluate the lead-free soldering technologies.

5:20 PM
Electromigration Studies on SnCu Flip Chip Solder Joint: *C. Y. Liu*[1]; H. T. Chiew[1]; C. J. Lu[1]; [1]National Central University, Cheml. & Matls. Eng., No. 300, Jung-da Rd., Jung-li City, Taoyuan 320 Taiwan

Electromigration is one of most urgent issues for flip chip technologies. The phenomenon of electromigration in SnPb and SnAg Pb-free solder has been reported previously by K. N. Tu et al.. Besides SnAg3.5, SnCu0.7 is another potential Pb-free candidate for C4 flip chip solder bump. Alloying effect of Cu in Al interconnect line is a well-known phenomenon. Small Cu content greatly enhanced the lifetime of Al conductive line. To understand the Cu alloying effect on Sn-based solders, we investigated electromigration behaviors on four SnCu solder alloys, which are pure Sn, SnCu0.2, SnCu0.7, and SnCu1. The test structure is in the form of C4 flip chip bump. The stressing current density is 10-5 A/cm2. In this paper, void and hillock formation will be presented and possible mechanism of Cu alloying effect will be proposed during this talk.

Magnesium Technology 2003: Magnesium Alloy Development-Mechanical Properties - II and Magnesium Wrought Alloys

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, LMD-Magnesium Committee, International Magnesium Association, MPMD-Solidification Committee
Program Organizers: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

Wednesday PM   Room: 2
March 5, 2003   Location: San Diego Convention Center

Session Chairs: Jiang Feng Nie, Monash University, Sch. of Physics & Matls. Eng., Victoria 3800 Australia; Darryl L. Albright, Hydro Magnesium, Mg. Market Dvpt., Livonia, MI 48152 USA

2:00 PM
AMC-SC1: An Elevated Temperature Magnesium Alloy Suitable for Precision Sand Casting of Powertrain Components: Colleen Joyce Bettles[1]; Christopher Forwood[1]; David StJohn[2]; Malcolm Frost[3]; Darryl Jones[1]; Ma Qian[2]; Guangling Song[2]; John Griffiths[1]; Jian Feng Nie[4]; [1]CAST (CSIRO Manufacturing and Infrastructure Technology), PB 33, Clayton S. MDC, Victoria 3169 Australia; [2]University of Queensland, CRC Cast Metals Mfg., UDP No. 55, St. Lucia, Queensland 4072 Australia; [3]Australian Magnesium Corporation, PO Box 1364BC, Milton, Queensland 4064 Australia; [4]Monash University, CAST (Sch. of Physics & Matls. Eng.), Clayton, Victoria 3169 Australia

AMC and the Australian Cooperative Research Centre CAST (Centre of Alloy solidification Technology), with the support of VAW and AVL, have developed a new sand cast alloy suitable for engine block applications. The creep and bolt load retention properties of this alloy are comparable to those of Al 319. The alloy requires similar casting temperatures to Al 319 and a T6 heat treatment, and therefore no significant new process technology is required to cast this alloy. Parallel development work on production costs has been successful indicating that AMC-SC1 is an economically viable magnesium alloy. The mechanical properties of the new alloy are presented, and discussed in relation to the properties required for a specific engine block design.

2:20 PM
Magnesium for Crashworthy Components: *Trevor B. Abbott*[1]; M. Easton[1]; R. Schmidt[1]; [1]Monash University, CRC for Cast Metals Mfg., Sch. of Physics & Matls. Eng., Wellington Rd., Clayton, Victoria 3168 Australia

With the emergence of new materials in automotive applications there is a tendency among design engineers to be cautious until the properties of the new materials are fully characterised. Often the properties which receive the greatest attention are those where the current materials have some deficiencies. These deficiencies do not necessarily carry over to the new material. A case in point is strain rate sensitivity for crash worthiness of steel and magnesium. Low carbon steels exhibit a distinct yield point during deformation, usually followed by a drop in load followed by work hardening. As the strain rate is increased, the yield point tends to increase such that it exceeds the post yielding ultimate tensile strength. In this situation there is no effective work hardening, resulting in the localisation of deformation such that only a small part of the component participates in energy absorption. From their past experiences in steel, design engineers are keen to understand the high strain rate properties when deciding whether to design a component in magnesium. The work described in this paper shows that magnesium does indeed show a strain rate dependence, and that the degree of sensitivity is highly dependant on the aluminium content of the alloy. However, the nature of the strain rate sensitivity is quite different to steel and appears to be beneficial to energy absorption. This paper discusses how these beneficial properties can be applied to the design of efficient energy absorbing components.

2:40 PM
Magnesium Wrought Alloys for Automotive Applications: *R. Gradinger*[1]; P. Stolfig[2]; [1]ARC Leichtmetallkompetenzzentrum, Ranshofen GmbH Austria; [2]Stolfig GmbH, Geisenfeld Germany

For about ten years magnesium alloys have gathered strong interest by the automotive industry resulting from new legal and market requirements. While pressure die casting soon found its applications, wrought alloys suffered from high costs and limited supply. However, parallel to activities to improve the production of sheets and extrusions, Stolfig GmbH started feasibility studies on new applications: material tests in the range of room temperature to 250∞C, extrusion of thin walled hollow profiles and deep drawing of sheets as well as Nd:YAG laser welding were performed in collaboration with the Light Metal Competence Center Ranshofen (LKR). The aim of the authors was to develop and test components for automotive usage. This efforts finally resulted in the delivery of prototype parts to Volkswagen. Their record-breaking 1-litre-car was equipped with space frame parts and interior paneling made of wrought magnesium alloys by Stolfig GmbH.

3:00 PM

The Effect of Temperature and Strain Rate on the Tensile Properties of Textured Magnesuim Alloy AZ31B Sheet: Ozgur Duygulu[1]; *Sean R. Agnew*[1]; [1]University of Virginia, Matls. Sci. & Eng., 116 Engineerís Way, Charlottesville, VA 22904-4745 USA

One problem facing the application of wrought magnesium alloys is their poor cold-forming properties. Historically, this has been linked to an inadequate number of independent slip systems, however, our recent research has suggested that 5 independent slip systems do participate in plastic deformation albeit with very different critical resolved shear stresses and hardening behavior. This understanding has been obtained by measuring stress-strain behavior and deformation texture evolution under various straining paths, as well as modeling of the same using a polycrystal plasticity simulation code known as the viscoplastic self-consistent (VPSC) model. The objective of the current research is to develop a fundamental understanding of the formability problem with a view towards improvement. It is well known that magnesium alloys can exhibit excellent forming characteristics at elevated temperatures (>180∫C). Thus, the temperature dependence of strain hardening, strain rate sensitivity, and plastic anisotropy of commercial AZ31B sheet will be presented.

3:20 PM Break

3:35 PM

Plasma Arc Lamp Processing of Magnesium Alloy Sheet: *Joe A. Horton*[1]; Craig A. Blue[2]; Sean R. Agnew[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, MS6115, PO Box 2008, Oak Ridge, TN 37831-6115 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg 4508-MS6083, PO Box 2008, Oak Ridge, TN 37831-6083 USA; [3]University of Virginia, Dept. of Matls. Sci., 116 Engineerís Way, PO Box 400745, Charlottesville, VA 22904-4745 USA

Plasma arc lamp processing of magnesium alloy sheet has the potential of reducing the number of processing steps involved in sheet production especially when coupled with a direct casting method. The high density infrared plasma arc lamp has continuously variable power densities up to 3.5 kW/cm2. Preliminary experiments on wrought commercial sheet, AZ31b, resulted in complete recrystallization with a uniform grain size through the thickness of the sheet. Incorporation of a pinch roller attached to and following the lamp will add to the versatility of this process by allowing for further cooling rate control and improved surface quality. Mechanical properties and microstructures will be presented as a function of the process variables on both wrought sheet and on arc lamp treated as-cast sheets.

3:55 PM

Indirect Extrusion of AZ31 and AZ61: *Klaus Mueller*[1]; [1]Technical University of Berlin, Extrusion R&D Ctr., Gustav-Meyer-Allee 25, Berlin 13355 Germany

The paper describes the results of indirect extrusion of the magnesium alloys AZ31 and AZ61. The extrusion trials were carried out on the 8 MN horizontal extrusion press of the Extrusion R&D Center (ERDC) of the Technical University Berlin, Germany. Different shape of product cross sections with nearly the same extrusion ratio were extruded at a billet temperature of about 300°C with different extrusion speeds and cooling rates. Since the extrusion press is equipped with load cells comparisons were made in necessary extrusion forces. The extrudates were tested for their mechanical properties such as compression/tensile strength, yield strength and fracture elongation. Texture analysis, hardness measurements and optical microscopy for grain size measurement were done at the Institute for Material Science and Technology, Technical University Berlin. The purpose is to determine the extrudability of AZ alloys and the application of the extrudates in space frame structure for automobile constructions.

4:15 PM

Influence of the Thermomechanical Treatment on the Microstructure of Magnesium Alloy AZ31: *Jan Bohlen*[1]; A. Hortsmann[1]; F. Kaiser[1]; A. Styczynski[2]; Dietmar Letzig[1]; Karl Ulrich Kainer[1]; [1]GKSS Research Centre, Ctr. for Mg Tech., Max-Planck-Strasse 1, Geesthacht 21502 Germany; [2]Technical University Hamburg-Harburg TUHH, Eiflendorfer Str. 42, Hamburg 21073 Germany

Magnesium wrought alloys offer a large potential for structural applications due to improved mechanical properties and microstructural homogeneity compared to thin-walled casted magnesium parts. Generally, the process parameters during rolling or extrusion are dependent on the formability of the used material as well as on the dynamic and static recrystallisation. The treatment itself leads to a microstructure that has a significant influence on mechanical properties. However, in the case of magnesium and its alloys the basic mechanisms of this influence are not well understood today. Two basic approaches are derived for the analysis of magnesium wrought alloys. One is the direct analysis of the recrystallisation behaviour during the wrought process. The other one is the determination of mechanical property dependences on the microstructure of rolled magnesium or extruded profiles. Results are shown using the grain size and their homogeneity as well as the precipitation distribution as a measure for the micro structure and mechanical tests for the determination of material properties. The dependences for the process parameters of magnesium wrought is shown in order to optimise mechanical properties.

4:35 PM

Low Temperature Super-Plasticity Response of Severe Plastic Deformed Az31b Magnesium Alloy: *Adi Ben-Artzy*[1]; Avigdor Shtechman[1]; Arie Bussiba[2]; Jacob Salah[1]; Sigalit Ifergan[2]; Moshe Kupiec[2]; Ronen Grinfeld[3]; [1]Rotem Industries, Ltd., Metal Forming Grp., PO Box 842, Metar 85025 Israel; [2]Rotem Industries, Ltd., Mechl. Characteriz. Grp., PO Box 9001, Beer-Sheva 84190 Israel; [3]Ben-Gurion University, Matl. Sci. Dept., PO Box 653, Beer-Sheva 84105 Israel

Commonly, super-plasticity phenomena occurred at low strain rates that applied at elevated temperature in materials with fine grain microstructure. Recently, several efforts have been devoted to study of the unusual super-plastic behavior of sever plastic deformation (SPD) at different parameters, motivated by practical and economical considerations. With this context, super-plasticity deformation in this metallurgical state (SPD) may be achieved at low temperatures with relatively high strain rates. Among other materials, Magnesium alloys exhibits this unique deformation mode mainly due to their tendency to dynamic recovery and recrystallization processes. Alloying elements as Zr stabilizes fine grain size of ZK60, which shows super-plastic behavior at wide strain rates and at relatively low temperature range. In the current research, (SPD) has been applied to as cast AZ31B alloy (200 µm average grain size) using Equal Channel Angular Extrusion (ECAE) technique resulted in 5-10 µm average grain size. The super-plastic response of this alloy was characterized by means of elevated temperature tensile test, using round specimens, at low temperature range with various strain rates. The grain refinement microstructure obtained by ECAE was analyzed using modern image analysis method. The results as reflected by the super-plastic curves (elongation, stress versus strain rate), were found sensitive to the number of passes but less sensitive to the successive rotations by 90° of the sample between the passes. Comparison between the super-plastic response of ECAE fine grain size AZ31B to the same alloy which was grain refined by conventionally hot extruded process, indicated that the maximum elongation of the former specimens was shifted to higher strain rate by magnitude order of two. This dramatic finding, point out the great influence of the ECAE in changing conditions of super-plastic behavior.

Materials Lifetime Science and Engineering - IV

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; D. Gary Harlow, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015-3085 USA; Dwaine L. Klarstrom, Haynes International, Inc., Kokomo, IN 46904-9013 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Robert P. Wei, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015 USA

Wednesday PM	Room: 18
March 5, 2003	Location: San Diego Convention Center

Session Chairs: D. Gary Harlow, Lehigh University, Mechl. Eng. & Mech., Bethlehem, PA 18015-3085 USA; Kwai S. Chan, Southwest Research Institute, Matls. Eng. Dept., San Antonio, TX 78228-0510 USA

2:00 PM Invited

Life Prediction Strategies for Land-Based Gas Turbine Blades: *Kwai S. Chan*[1]; N. Sastry Cheruvu[1]; Gerald R. Leverant[1]; R. Viswanathan[2]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., PO Drawer 28510, San Antonio, TX 78228-0510 USA; [2]EPRI, 3412 Hillview Ave., PO Box 10412, Palo Alto, CA 94303 USA

A wide range of coatings including overlay, diffusion, duplex, and thermal barrier coatings, are used on the first-stage blades of land-based gas turbine machines. Life-limiting mechanisms of the first stage blades include cyclic oxidation, thermomechanical fatigue, and among others, hot corrosion. In this overview paper, the development of a science-based methodology for lifing coated first-stage blades is presented. The potential failure mechanisms in coated gas turbine blades are summarized. The current status of a science-based life prediction methodology, which is implemented into a computer software named COATLIFE, for treating cyclic oxidation and thermomechanical fatigue is then highlighted. The scientific bases of COATLIFE are described. The technical capabilities and accuracy of COATLIFE are illustrated by comparisons against laboratory data and field experience. Extension of COATLIFE to predict the service life of TBC-coated blades will also be discussed. Works supported by Electric Power Research Institute and Department of Energy through Contract No. DE-FC26-01NT41321, Mr. Norman T. Holcombe, Program Manager.

2:30 PM Invited

Analysis of Environmental Effects in the Unified Approach: *Asuri K. Vasudevan*[1]; Kuntimaddi Sadananda[2]; Roland L. Hotlz[2]; I. W. Kang[3]; [1]Office of Naval Research, 800N Quincy St., Arlington, VA 22217 USA; [2]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 20375-0001 USA; [3]Defense Quality Assurance Agency of Seoul, Korea and Naval Research Laboratory, Washington, DC 20375 USA

In the Unified Approach fatigue crack growth rates are analyzed using two-parameter approach involving amplitude *K and peak stress intensity, Kmax. By considering these two parameters it is shown that one can analyze most of the fatigue phenomena without the need of crack closure concept. Effects of environment naturally manifest through the Kmax driving force. These effects are dominant at low *K values or slow crack growth rates. Hence they play a significant role in determining the total fatigue life of a component. A systematic analysis of the effects of superposition of environmental effects on fatigue crack growth is made and the behavior is classified. A detailed discussion will be presented taking examples from the literature.

3:00 PM Invited

Fatigue Crack Growth of Discontinuously Reinforced Aluminum (DRA): *Vasudevan V. Ganesh*[1]; Jason Williams[1]; Nikhilesh Chawla[1]; Asuri Vasudevan[2]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA; [2]Office of Naval Research, Arlington, VA 22217-5660 USA

Discontinuously reinforced aluminum (DRA) is emerging as a lightweight, high performance alternative to monolithic aluminum alloys. Developing a framework for fatigue lifetime prediction of these heterogeneous materials requires a fundamental, microstructure-based understanding of fatigue crack growth. In situ fatigue crack growth studies were conducted using a QuestarTM traveling telescope. Fatigue crack growth studies were conducted at varying R-ratios and the behavior analyzed using the two-parameter approach that relates DKth to Kmax, proposed by Vasudevan et al.1 The interaction between the fatigue crack and reinforcement particles or particle clusters was quantified. Preliminary microstructure-based modeling to understand and predict the effect of particle clusters on fatigue crack rate and stress intensity at the crack tip will be discussed. 1A.K. Vasudevan, K. Sadananda, and N. Louat, Mater. Sci. Eng., (1994) A188 1. Research sponsored by the Office of Naval Research under contract #N00014-01-1-0694.

3:30 PM

Fatigue Fracture Mechanism and Fatigue Life Assessment of Aluminum Castings: *Qigui Wang*[1]; [1]General Motors Corporation, CDVC-Powertrain, 1629 N. Washington Ave., Saginaw, MI 48605 USA

The increasing application of shape-cast aluminum components in both automotive and aerospace industries has drawn great concern in fatigue properties of aluminum castings. In order to enhance the fatigue properties of aluminum castings and to increase the reliability and wider use of aluminum castings in cyclic loading applications, a better understanding of fatigue fracture mechanism that presents in aluminum castings and the factors affecting the fatigue crack initiation and propagation is absolutely necessary. For this propose, a critical review and case study has been carried out to address the fatigue fracture mechanism, the factors affecting the fatigue properties and fatigue life assessment methods and eventually to point out further challenges for the improvement and accurate prediction of aluminum fatigue.

3:50 PM

Fatigue and Fretting Fatigue of Biomaterial, Ti-29Nb-13Ta-4.6Zr, in Air and Simulated Body Environment: *Mitsuo Niinomi*[1]; Toshikazu Akahori[1]; Keita Ishimizu[1]; [1]Toyohashi University of Technology, Dept. of Production Sys. Eng., 1-1, Hibarigaoka, Tempaku-cho, Toyohashi 441-8580 Japan

The fatigue and fretting fatigue characteristics of the newly developed biomaterial, Ti-29Nb-13Ta-4.6Zr, were investigated in both air and simulated body environment, Ringerís solution. The fatigue strength of Ti-29Nb-13Ta-4.6Zr is not degraded in Ringerís solution. The fretting fatigue damage is well correlated to the Youngís modulus. The fretting fatigue strength of Ti-29Nb-13Ta-4.6Zr is greater in Ringerís solution than in air in the low cycle fretting fatigue life region, but smaller in Ringerís solution than in air in the high cycle fretting fatigue life region. The effect of lubrication due to Ringerís solution is greater in the low cycle fretting fatigue life region, but the effect of corrosion is greater in the high cycle fretting fatigue life region.

4:10 PM

Effect of Alpha Grain Size on Low-Temperature Fatigue Property of Ti-5%Al-2.5%Sn ELI Alloy: *Yoshinori Ono*[1]; Tetsumi Yuri[1]; Hideshi Sumiyoshi[1]; Saburo Matsuoka[1]; Toshio Ogata[1]; [1]National Institute for Materials Science, Matls. Info. Tech. Sta., 1-2-1, Sengen, Tsukuba, Ibakaki 305-0047 Japan

The effect of alpha grain size on low-temperature fatigue property has been investigated in Ti-5%Al-2.5%Sn ELI alloy used for fuel turbo pumps of Japanese domestic space rocket. Grain size of specimens was controlled to be about 30 micron or 80 micron. Tensile strengths of both specimens are almost same and are increased with a decrease of temperature. However, low-temperature fatigue property is quite different between both specimens. In the specimen with 30 micron grains, 106 cycles fatigue strength at 4K and 77K is 1.6 and 1.5 times higher than that at 293K, respectively. On the other hand, in the specimen with 80 micron grains, 106 cycles fatigue strength at 4K and 77K gets lower to the same level as that at 293K. Hence, it is concluded that refinement of alpha grains plays a very important role to obtain the good low-temperature fatigue property for Ti-5%Al-2.5%Sn ELI alloy.

4:30 PM

Re-Consideration of Fatigue Crack Closure Effect in the Lifetime Prediction of Structural Materials: *Daolun Chen*[1]; [1]Ryerson University, Dept. of Mechl., Aeros. & Industl. Eng., 350 Victoria St., Toronto, Ontario M5B 2K3 Canada

The integrity of some engineering structures, e.g., riveted and welded components, depends predominantly on the lifetime spent in the crack growth, resulting in the development of a defect-tolerant approach. This approach relies on an integration of the crack growth expression, based upon an accurate description of the effective driving force by considering fatigue crack closure effect. However, many researchers have questioned the validity of the conventional crack closure concept. In view of the discrepancies and confusions reported in the literature, we have re-modeled and re-defined the fatigue crack closure effect by taking into account the role of the lower portion of fatigue cycles below the opening point, so that this concept can be used to predict better the fatigue life of materials/components. The aim of this paper is to review the fatigue crack closure effect and to present further the experimental evidence on the modified crack closure definition.

4:50 PM

Characterization of Fatigue Crack Origins in 2024-T3 Aluminum Alloys: *Jonathan Tsang*[1]; Ali Merati[1]; [1]Institute for Aerospace Research Structures, Materials, and Propulsion Laboratories, Bldg. M-13, 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada

There is a high probability of interaction between corrosion and fatigue and the probability increases as aircraft age. New analytical tools and procedures for structural integrity analysis are based on a holistic life assessment that uses the Initial Discontinuity State (IDS) of the material, as the initial condition for crack growth analysis. NRC-IAR is participating in a collaborative research and development project with the objective of producing a reliable corrosion/fatigue life prediction methodology. The work is part of the USAF-Lockheed Martin Corrosion Fatigue Structural Demonstration (CSFD) project. This extensive study has been carried to physically measure the IDS for pristine 2024-T3 aluminum alloys. The type of discontinuity being characterized in this study is limited to constituent particles. The overall IDS data were measured by metallographic sectioning as well as fractography of fatigue coupons. The two distinctive controlling factors responsible for crack nucleation will be discussed. It was found that for bare 2024-T3 materials, the constituent particles were the main microstructural discontinuity origination the cracks. Comparatively, the fatigue crack origins in clad 2024-T3 samples were located at the surface of the clad. No constituent particles were found to be associated with crack nucleation. Multi-nucleation sites were observed in this material. This paper summarizes the result of tests for the clad and bare 2024-T3 aluminum alloys.

Materials Processing Fundamentals: Thermodynamics and Reaction Kinetics

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee, EPD-Process Fundamentals Committee

Program Organizers: Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA; Princewill N. Anyalebechi, Grand Valley State University, L. V. Eberhard Center, Grand Rapids, MI 49504-6495 USA

Wednesday PM March 5, 2003 — Room: 1A — Location: San Diego Convention Center

Session Chair: Seshadri Seetharaman, Royal Institute of Technology, Div. of Metall. Dept. of Matls. Sci. & Eng., Stockholm SE-100 44 Sweden

2:00 PM

Thermodynamic Studies of FeO-Containing Slag Systems: *Patrik Fredriksson*[1]; Seshadri Seetharaman[1]; [1]Royal Institute of Technology, Div. of Metall., Dept. of Matls. Sci. & Eng., Brinellvagen 23, Stockholm SE-100 44 Sweden

Thermodynamic data concerning FeO-containing slags is of importance in ladle refining of steel. With a view to generate a set of reliable and self-consistent thermodynamic data for these slags, experimental determination of the activities of FeO in binary and ternary slags was carried out using the gas equilibration method involving CO-CO2-Ar gas mixtures at steelmaking temperatures. The slag samples in Pt crucibles were quenched after the equilibration and subjected to chemical analysis. The thermodynamic activities of ìFeOî in the slags were calculated from the experimental data. The results are incorporated into a thermodynamic description of silicate melts in the present laboratory. The model is based on a Temkin-Lumsden approach and is able to compute the thermodynamics of higher order systems from the lower order ones.

2:30 PM

Kinetics of Chlorination and Oxychlorination of Hematite: *N. Kanari*[1]; D. Mishra[1]; I. Gaballah[1]; [1]Laboratoire Environnement et Mineralurgie, Rue du Doyen M. Roubault, BP 40, Vandoeuvre, Cedex 54501 France

Kinetics of Fe2O3 reactions with Cl2 and Cl2+O2 gas mixtures were investigated up to 1025 C using thermogravimetric technique. Effects of gas flow rate, temperature and partial pressure of Cl2, O2 and Cl2+O2 on the reaction rate were evaluated. Data were processed to predict the reaction rate mechanism. The values of the apparent activation energy ëEaí of Fe2O3 chlorination were respectively 180 and 74 kJ/mol for the temperatures lower and higher than 875 C. The oxychlorination of hematite proceded with an Ea of 148 kJ/mol between 600 and 1025 C. The increase of the oxygen partial pressure in the Fe-O-Cl system led to low reaction rate of hematite chlorination. The apparent reaction orders with respect to Cl2, O2 and Cl2+O2 at 750 C were respectively 1.44, -0.61 and 0.71.

2:50 PM

Hydrothermal Precipitation and Thermal Transformation of Boehmites: *D. Mishra*[1]; N. Kanari[1]; I. Gaballah[1]; S. Anand[2]; R. P. Das[2]; [1]Laboratoire Environnement et Mineralurgie, Mineral Processing and Environmental Engineering Team, Rue du Doyen M. Roubault BP 40, Vandoeuvre, Cedex 54501 France; [2]Regional Research Laboratory, CSIR, Bhubaneswar, Orissa 751 013 India

Boehmites (gamma-Al2O3.xH2O) are the oxide-hydroxides of aluminum and find extensive use for manufacture of high surface area catalysts, coatings, aluminum and alumina derived materials. The present paper describes a novel hydrothermal precipitation route to obtain high surface area boehmites and their thermal transformation sequences to alpha-Al2O3. Hydrothermal precipitations were carried out in autoclave with inorganic aluminum salt solutions [Al2(SO4)3, AlCl3, Al(NO3)2] and urea as the neutralizing agent. Effects of residence time and temperature on the crystallization extent of boehmites obtained either from Al2(SO4)3 had a lower water content and a higher surface area than the boehmites obtained either from AlCl3 or Al(NO3)2. Calcination of these boehmites between 450-550 C produced gamma-Al2O3 of high surface area (280-330 m2/g). This compound was converted into alpha-Al2O3 at about 1400 C.

3:10 PM

A Novel Method for Extraction of TiO2 from Ilmenite Ores: Vilas D. Tathavadkar[1]; *Animesh Jha*[1]; [1]University of Leeds, Inst. of Matls. Rsrch., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK

Three different types of processes, namely the sulfate process, the chloride process, and the chloride-ilmenite process, are used worldwide for production of titanium dioxide. Of these three methods of production, the chloride process is more popular and is therefore widely used. The sulfate process creates large amounts of dilute acid effluent, whereas the chloride route yields a hazardous waste than the sulphate process. However, the volume produced is much smaller. For example, one-ton production of titanium dioxide generates 12 tons of wastes material from the sulfate process while the chloride process generates only 4 tons wastes. Iron chloride, generated in the chloride route is acidic and hazardous. We have investigated a novel route for the separation of titanium dioxide from ilmenite ores. In this process, ilmenite ore is roasted with alkali carbonate in the temperature range of 800 to 1100°C to form alkali titanates. The roasted mass was leached with water and then with dilute acid solution to remove the impurities. The physical and chemical properties and phase equilibria of roasting reactions were investigated. The results of isothermal roasting, water and acid leaching experiments are discussed in details in this paper. Nearly 95% pure TiO2 was obtained via this method, which can be further improved by acid leaching treatment.

3:30 PM

The Polymorph Transformations of Antimony White in Hydro-Process: *Xiao Songwen*[1]; Yan Xiaohui[2]; Xiao Xiao[3]; Zhang Duomo[4]; [1]Changsha Research Institute of Mining & Metallurgy, Changsha, Hunan 410012 China; [2]Hunan Sunrise Nanometer Material Company Ltd., Changsha, Hunan 410014 China; [3]Central South University, Changsha, Hunan 410083 China; [4]Central South of University, Col. of Metallurgl. Sci. & Eng., Changsha, Hunan China

On grounded of the theoretical models of growth unites with coordination polyhedron structure of anion, the polymorph transformation mechanisms of antimony white Sb2O3 in hydro-process is presented. The new findings has been verified in the commercial-scale test, and it shows that adding a small amount of tratrate ions is effective for transformation of antimony white in hydro-process.

Materials Processing Under the Influence of Electrical and Magnetic Fields - VI

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee

Program Organizers: Joanna R. Groza, University of California-Davis, Chemical Engineering Material Science Department, Davis, CA 95616 USA; George S. Dulikravich, The University of Texas at Arlington, Multidisciplinary Analysis, Inverse Design, and Optimization (MAIDO) Program, Department of Mechanical and Aerospace Engineering, Arlington, TX 76019 USA; Nagy H. El-Kaddah, University of Alabama, Department of Metallurgical & Materials Engineering, Tuscaloosa, AL 35487-0202 USA; James W. Evans, University of California, Department of Materials Science and Mineral Engineering, Berkeley, CA 94720 USA; Zuhair Munir, University of California, College of Engineering, Davis, CA 95616-5294 USA; Srinath Viswanathan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Wednesday PM March 5, 2003 — Room: 14A — Location: San Diego Convention Center

Session Chairs: K. Linga Murti, National Science Foundation, Arlington, VA 22230 USA; Joanna R. Groza, University of California-Davis, Cheml. Eng. Matl. Sci. Dept., Davis, CA 95616 USA

2:00 PM Invited

Real Time Measurements of the Effect of Electric Fields on Grain Growth in Nanocrystalline Silver Thin Films: *E. A. Stach*[1]; T. Freeman[1]; X. Phung[2]; L. Stanciu[2]; J. R. Groza[2]; K. Hukari[3]; R. Dannenberg[4]; [1]National Center for Electron Microscopy, Lawrence Berkeley Natl. Lab., Berkeley, CA 94720-0001 USA; [2]University of California at Davis, Dept. of Matls. Sci. & Eng., Davis, CA 95616 USA; [3]AFG Development Corporation, Petaluma, CA USA; [4]Chahaya Optronics, Fremont, CA USA

One method for creating materials with nanometer grain sizes is by sintering of nanoparticles. However, a critical step in the nanopowder consolidation process concerns the ability to achieve full densification with minimal microstructural coarsening. In this work, we have chosen to study coarsening in a model system, that of nanocrystalline silver thin films, to determine the effects of temperature and electric field on the process. We have developed a methodology that allows us to determine in real time and at high spatial resolution the effects of initial grain size, temperature and applied electric field on the grain growth characteristics. To do this, we have constructed a combined heating/electrical bias stage for a transmission electron microscope. This allows us to heat samples to temperatures in excess of 1000°C as well as apply electric fields in excess of 30 V/m to the sample. This can all be done within the objective lens of the electron microscope, allowing quantitative, real time characterization of the activation energy of grain growth and development of texture in these silver thin films. In this presentation, we will emphasize the details of this new methodology, discuss its strengths and limitations, and present quantitative data concerning the effects of applied electric fields on grain growth.

2:30 PM

Unique Facility for Materials Processing in High Temperatures and High Magnetic Fields: *Sastry V. Pamidi*[1]; Ulf P. Trociewitz[1]; Hiroshi Maeda[1]; Saleh Hayek[2]; Askar D. Sheikh-Ali[2]; Cristiane B. Bacaltchuk[2]; Marwan Elkawni[2]; Shi Shen Yan[2]; Hamid Garmestani[3]; Justin Schwartz[1]; [1]Florida State University Center for Advanced Power Systems, Natl. High Magnetic Field Lab., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA; [2]Florida A&M University & Florida State University College of Engineering, Tallahassee, FL 32310 USA; [3]Florida A&M University & Florida State University College of Engineering, Natl. High Magnetic Field Lab., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA

Processing of materials under the influence high magnetic fields has been reported to cause significant enhancements in useful properties of many materials. The mechanisms of influence of magnetic field on texture development, grain growth, and recrystallization need to be understood in order to optimize the processes and to develop commercial applications of in-field heat treatment processes. We have designed, built, and tested several furnaces for the 20 T resistive magnet at the National High Magnetic Field Laboratory. The furnaces can be used for experiments in magnetic fields up to 19 T and temperatures up to 1600C. All the furnaces are equipped with temperature programmers and gas flow systems to control the atmosphere during heat treatments. The volume in the uniform magnetic field and temperature zone is up to 60 mm in diameter and 100 mm in height to accommodate large samples. Some of our experiments focused on enhancing the texture of Bi2212 high temperature superconducting tapes and understanding r ecrystallization, grain growth, phase transformations, and solidification from the melt in NdFeB, Fe-Si, Zn-Al and terfenol-D systems. The paper describes the unique features of our facilities and some of the experiments that have been performed with the facilities.

2:50 PM

Application of High Magnetic Field to Texture Development in Zn Alloy: *Askar D. Sheikh-Ali*[1]; *Dmitri A. Molodov*[2]; Hamid Garmestani[1]; [1]National High Magnetic Field Laboratory, Lab. for Micromech. of Matls., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA; [2]Institut f¸r Metallkunde und Metallphysik, RWTH Aachen, Aachen D-52056 Germany

Highly textured Zn-1.1%Al alloy with fine-grained microstructure are annealed in a high magnetic field of 32T. The texture of the samples is characterized by the two 0002 components tilted at 15-20° from the normal to the rolling direction of the sheet. The annealing of samples parallel to the field preserves the maximum intensity of texture components and redistributes the intensity closer to the normal direction. The annealing of samples parallel to the field preserves the maximum intensity of texture components and redistributes the intensity between original orientation of 0002 components and normal direction. The obtained results are interpreted in terms of selective grain growth due to the anisotropy of (dia)magnetic susceptibility in Zn.

3:10 PM

Grain Boundary Dynamics in High Magnetic Fields: *Dmitri A. Molodov*[1]; [1]Aachen University of Technology, Inst. of Physl. Metall. & Metal Physics, Kopernikusstr. 14, Aachen 52056 Germany

The movement of grain boundaries is one of the fundamental mechanisms of microstructure evolution during heat treatment of metallic materials. Therefore, control of grain boundary motion means control of microstructure evolution, which is a key for the design of advanced materials. Grain boundary motion can be affected by a magnetic field, if the anisotropy of the magnetic susceptibility generates a gradient of the magnetic free energy. In contrast to curvature driven boundary motion, a magnetic driving force also acts on flat boundaries so that the motion of crystallographically fully defined boundaries can be investigated, and the true grain boundary mobility can be determined. By appropriate positioning and repositioning of the specimen in the magnetic field the energy gradient can be changed and even inverted for the same boundary. This allows for the first time to study asymmetry effects of boundary dynamics. We will report in detail results obtained on Bi and Zn bicrystals. Selective grain growth in deformed Zn monocrystals and texture changes in Zn alloy sheet in high magnetic fields will be discussed.

3:30 PM Break

3:45 PM

Experiments on Dynamic Behavior of Molten Metal and Solid Particles Under Direct Current Passing: *O. I. Raychenko*[1]; V. P. Popov[1]; O. V. Deretíyanko[1]; T. I. Istomina[1]; A. I. Repenko[1]; [1]IPMS, NAS of Ukraine

At electrodischarge sintering of powder mixtures by electric direct current some essential displacements of molten metal and solid particles were revealed. Such displacements at conventional sintering are not known for us. The objects were powders (mixtures): Sn-Cu (dispersivity 100-500 mkm); Sn - high-temperature alloy on nickel base or electrocorundum; coper alloy - electrocorundum (dispersivity 63-500 mkm); Ni (dispersivity 10 mkm). The electric direct current was passed in vertical direction parallel with the axis of cylindrical mould or through the series circuit steel-powder nickel layer-hard alloy. If cylindrical countainer (mould) had not the upper punch (a load on top was absent) then sample after solidification had form of a ìbulletî. This sample became convex on top and concave on bottom. At presense of pre-pressing (start pressing) on top a movement of suspension (molten matrix and solid particles) occured in the similar way. Under action of electro-magnetic forces a redistribution of solid partic les in volume of suspension occurs. Particles with more high conductivity than liquid phase begin to cluster at center. In the case of lower conductivity the motion of the particles to outer side surface of mould prevails. At current passing through series circuit steel-nickel powder layer-hard alloy three named parts of circuit were sintered into single block. After destruction at testing the interlayer had the form of a ring. This shows non-uniformity of passing electric current through interlayer. The cause of displacements of solid (non-molten) particles may be simultaneous influence of thermic convection and squeezing out by the electromagnetic Lorentz-forces, which are directed radially. The phenomena described may be applied at production of objects with gradient (variable in volume) concentration of particles added specially. This refers to products of their interaction with molten matrix as well.

4:05 PM Invited

Theory of Motion of Inclusions in a Viscous Medium Under Joint Influence of Electromagnetism and Thermocapillarity: *O. I. Raychenko*[1]; [1]IPMS, NAS of Ukraine

The motion of a fine inclusion in a viscous medium is subjected to the theoretical examination. This motion can be qualified as an elementary act of the motion of fine phase particles in mixtures: suspensions, emulsions, airsols, foamed liquid materials. The erroneous character of the theory of thermocapillary motion of inclusions, which is universally recognized now, is revealed. It is proposed to take into account the ìenergy of locationî of the inclusion in a non-uniform temperature field, in order to formulate more correctly the Stokes equation describing the velocity field inside the fluid inclusion. The solution of the set of the Stokes equations referring to a liquid particle and a surrounding medium has allowed to obtain the formula for the motion velocity of a particle under joint influence of electromagnetism and thermocapillarity. This formula is suitable for theoretical calculations of transfer processes in suspensions, emulsions and gaseous media containing various inclusions. The calculation on inclusions in molten metals (for example, in Cu, Al, Fe, Ag) shows that the exertions caused by electromagnetism and thermocapillarity are comparable.

4:25 PM
Aspects of Magnetic Field Use for Treatment of Water and Water Systems: *V. V. Honcharuk*[1]; V. V. Malyarenko[1]; [1]Ukraine National Academy of Science, Dumansky Inst. of Colloid Chem. & Chem. of Water, 42, Vernadsky Ave., Kyiv-03142 03680 Ukraine

In aqueous solutions at magnetic field (MF) induction 0,108 T and liquid velocity in cell 0,51,5 m/s the electric driving force (EDF) is approximate to it calculated. As it is elicited, the EDF cause a current for which as electrodes and as external load double the conducting inner wall of the cell. MF action on the system is analogic to electric current energize. At MF-treatment some surface electrode processes (solution or precipitation metal, gassing) cause a running renovation of the surface, that decrease of scale. At the same time composition of the solution change under effect of the electrochemical processes. After choice of the MF-treatment regime these factors can use for water demineralizing, for act on microorganisms, for change of surface charge and colloids coagulable, for galvanic solution and/or precipitating of metals.

Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction: Microscale Modeling & Simulation

Sponsored by: Structural Materials Division,
Program Organizers: James M. Larsen, US Air Force, Air Force Research Laboratory, Materials and Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA; Leo Christodoulou, Defense Advanced Research Agency, Arlington, VA 22203-1714 USA; William J. Hardman, Naval Air Systems Command, Propulsion and Power Directorate (AIR 4.4.2), Patuxent River, MD 20670-1534 USA; Andrew J. Hess, Naval Air Systems Command, Propulsion and Power Directorate, Patuxent River, MD 20670-1534 USA; J. Wayne Jones, University of Michigan, College of Engineering, Department of Materials, Science and Engineering, Ann Arbor, MI 48109 USA; Stephan M. Russ, Air Force Research Laboratory, Materials and Manufacturing Directorate, AFRL/MLLN, Wright-Patterson Air Force Base, OH 45433-7817 USA

Wednesday PM — Room: 16A
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: J. Wayne Jones, University of Michigan, Col. of Eng. Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; David Alan Johnson, Air Force Research Laboratory, WPAFB, OH 45433-7817 USA

2:00 PM
Micron- and Nano-Scale Mechanical Testing for Model Parameterization: *Dennis M. Dimiduk*[1]; Michael Uchic[1]; Triplicane A. Parthasarathy[2]; Yoon-Suck Choi[2]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, WPAFB, OH 45432 USA; [2]UES, Inc., Dayton, OH 45432 USA

A necessary part of achieving a materials-based prognostics capability is the development, parameterization, and validation of continuum materials behavior models. Such models should be appropriately informed from microstructural information and knowledge of mechanistic behavior of the material. Continuum crystal plasticity methods are at the forefront of techniques that incorporate first-level microstructural information. However, a deficiency in parameterizing those models lies with the inability to obtain single crystal property information from individual grains, especially when such parameters must reflect the subtleties of material process history. Our effort has sought to measure single-crystal critical resolved shear stresses and strain hardening rates from micro and nano-scale samples extracted from relevantly processed structural alloys. In this presentation we present the experimental methods, challenges associated with those methods, and early results for this new approach toward structural property assessment.

2:30 PM Invited
Modeling the Physics of Microstructure Failure in a Nickel Alloy: *Donald A. Shockey*[1]; Jeffrey W. Simons[2]; Brian D. Peterson[2]; Takao Kobayashi[2]; [1]SRI International, Ctr. for Fracture Physics, 333 Ravenswood Ave., Menlo Park, CA 94025 USA

SRI International is constructing a failure prognostic algorithm for engineering materials that attempts to overcome limitations of current continuum approaches. The algorithm is based on the physics of material failure at the grain and subgrain level in a nickel-based superalloy. The evolution of microstructure deformation and failure, as observed and measured in interrupted fatigue tests is described mathematically, quantified with data generated in the tests, and implemented into a finite element code. The algorithm is demonstrated by simulating damage developing at the tip of a fatigue crack. The model is being extended to include microcrack coalescense and enable simulations of crack propagation in a turbine disk.

2:55 PM Invited
Enhanced Damage-State Awareness Through Full-Field Measurement Techniques: *David Alan Johnson*[1]; [1]Air Force Research Laboratory, AFRL/MLLMN, Bldg. 655, Ste. 1, 2230 Tenth St., WPAFB, OH 45433-7817 USA

A key to integrating mechanism-based prediction and damage-state awareness is better understanding of the fundamental physics of damage. In order to gain this physical understanding, much more efficient means of studying actual material behavior through experimentation must be developed. One very promising area for enabling this efficient experimentation is through full-field deformation mapping accomplished with machine vision. With full-field deformation mapping, it is possible to efficiently study damage development under a wide variety of conditions and at a wide variety of spatial scales. It is also possible to efficiently calibrate and evaluate probabilistic damage models through automated and extensive sampling. An example of the use of deformation mapping in the study of the failure physics of a large-colony, fully lamellar intermetallic will be presented, along with a discussion of the many possible future uses of full-field measurement techniques in studying damage progression, both quantitatively a nd quantitatively. The future real-time integration of modeling and experimentation made possible by full-field techniques will also be discussed.

3:20 PM Break

3:50 PM
Mechanisms of Elevated Temperature Fatigue Crack Growth of Ti-6Al-4V: Tarun Goswami[1]; David S. Gelles[2]; Russell H. Jones[2]; [1]Arkansas Tech University, Mechl. Eng., Russellville, AR 72801 USA; [2]Pacific Northwest National Laboratory, Matls. Sci., PO Box 999, Richland, WA 99352 USA

The Ti-6Al-4V alloy is used as a fan disk material because of its high strength to weight ratio, toughness and fatigue strength; however, very little is known about its elevated temperature fatigue crack growth mechanism. The fatigue crack growth mechanism of Ti-6Al-4V has been evaluated with transmission electron microscopy (TEM) of fatigue crack growth specimens tested at an R of 0.1, frequency of 10 Hz at 175, 230, 290 and 345 C. TEM samples were removed at various locations along the crack wall with special care to locate the foil within the crack tip plastic zone. A major proportion of the dislocations was concentrated in a-grains with only a small fraction present in b-grains. Within the a-grains a combination of both <c> and < a> dislocations formed with some twinning. The <c> type dislocations in planar arrays could be demonstrated with g=0002 near [1210]. Within these grains, dislocation density increased as the temperature was increased. The relevance of these observations to fatigue crack growth mechanisms will be described.

4:15 PM
Integrating Damage State: From Micro-Mechanism to Macroscopic Behavior: *Xijia Wu*[1]; [1]National Research Council of Canada, Inst. for Aeros. Rsrch., M-13, 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada

Fatigue and creep are the two most important failure modes in a high performance gas turbine. Integrated constitutive equations are presented for gas turbine metallic materials under fatigue and creep conditions. These equations are derived from the understanding of the physical mechanisms governing deformation and crack growth in metallic materials. For example, fatigue damage is known to accumulate by restricted slip reversal, which eventually leads to a mode of transgranular crack propagation. On the other hand, the accumulation of creep damage is more complex in a polycrystalline material: it occurs by competition of grain boundary sliding, volume diffusion and intragranular dislocation mechanisms. Recognizing each physical component of deformation would provide an improved life prediction methodology and accurate feedback to the design of the alloy for better performance. A universal equation has been derived that the rate of crack propagation (under either fatigue or creep conditions) is proportional to the accumulation of plastic strain, e_p (or strain range, Delta e_p) and the plastic zone size, l_p, at the crack-tip, as expressed by the formula: da/dt (or da/dN) = e_p (or Delta e_p) . l_p. Using this type of relationships, damage accumulation and the materialís life can be evaluated through integration of the understanding of the microphysical processes. Such physically based relationships are also useful to enhance the damage state awareness, in coupling with advanced NDI techniques.

4:40 PM Invited

Creep Life Prediction Using a Multiple-Damage State Variable Model: Application to Aluminium Alloy 2650: Alex Djakovic[1]; Hector Basoalto[1]; *Brian F. Dyson*[1]; Malcolm McLean[1]; [1]Imperial College of Science, Technology and Medicine, Matls., Prince Consort Rd., London SW7 2BP UK

A generic Continuum Damage Mechanics approach to creep of particle-strengthened alloys has been developed to account for the influence of the concurrent evolution of a number of types of damage. The approach identifies three broad categories of damage that are fully interactive through being coupled with the rate of creep deformation. The damage mechanisms considered are (a) dislocation accumulation, (b) thermal changes in particle size/distribution and (c) grain boundary cavitation. Primary creep is due to progressive load transfer from the deforming matrix to the particles as creep strain evolves and not to ìstrain hardeningî since dislocations weaken rather than strengthen the alloy in this model. The relative contributions of the different types of damage are material specific, but the formalism allows these differences to be accommodated parametrically within the model. These parameters are constrained within physically realistic limits determined theoretically or by independent experiment, rather than being fitted empirically to creep data. The model will be illustrated in relation to the creep behaviour of the Al-Cu-Mg precipitation strengthened alloy 2650-T8. The principal strengthening precipitate in this alloy is -phase. Account has been taken of its rod-like morphology in accounting for its obstacle strength; the extent of primary creep due to stress transfer from the matrix; and to the asymmetric coarsening of the rods in contributing to tertiary creep. In representing the effects of creep cavitation, it is shown that modelling the transition from unconstrained to constrained cavity growth captures the form of the minimum creep rate and rupture life as functions of stress over a range of temperatures. The multi-damage model also provides a good representation of shapes of creep curves and provides a basis for identifying the damages that have most influence on the creep behaviour under various loading conditions.

5:05 PM Invited

Crystal Rotations During Multiaxial Creep in Single Crystal Superalloys: Characterisation and Modelling: Mamoud G. Ardakani[1]; Hector Basoalto[1]; Barbara A. Shollock[1]; *Malcolm McLean*[1]; [1]Imperial College of Science, Technology and Medicine, Matls., Prince Consort Rd., London SW7 2BP UK

Life prediction procedures for single crystal superalloys, which are now state of the art for advanced gas turbine blades, are largely based on the interpretation of uniaxial creep and low cycle fatigue data. However, turbine blades in service experience significant multiaxial stresses at the blade roots and in the neighbourhood of cooling channels. It is appreciated that uniaxial stresses along non-symmetric orientations cause crystal rotations during creep. It is less well known that multiaxial stressing of symmetric single crystal superalloys can also lead to large crystal rotations that can be spatially heterogeneous. In the present study, the creep behaviour of the single crystal nickel-based superalloy CMSX-4 with <001>, <111> and <011> nominal orientations has been studied on cylindrical specimens with circumferential notches at 850∞C and net-section stresses between 600 and 850 MPa. The distribution in deformation across the specimen diameter has been characterised by mapping the changes in crystal or ientation using electron backscatter diffraction (EBSD) and comparing this with lattice rotations observed in uniaxial creep specimens. Photogrammetry experiments have also been carried out within the notch to study strain distribution. An anisotropic creep model based on a continuum damage mechanics (CDM) formalism that was developed and validated for uniaxial loading of nickel-base superalloys has been used to simulate creep in multiaxial loading in notched specimens. The predictions of crystal rotation resulting from creep deformation are compared with the experimental results.

Materials Research to Meet 21st Century Defense Needs

Sponsored by: TMS, National Materials Advisory Board,
Program Organizers: Arul Mozhi, National Materials Advisory Board (NA-966), National Research Council, Washington, DC 20001 USA; Harvey W. Schadler, Naskayuna, NY 12309 USA

Wednesday PM Room: 5B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Robert L. Rapson, Air Force Research Laboratory, Non-Metallic Matls. Div., WPAFB, OH 45433-7750 USA; Alan M. Lovelace, General Dynamics Corporation, Commercial Launch Services, Cape Canaveral, FL 32920 USA; Arul Mozhi, National Research Council, Natl. Matls. Advisory Board, Washington, DC 20001 USA

2:00 PM Opening Remarks Robert L. Rapson

2:10 PM Invited

Overview of Defense Materials Needs and Crosscutting Research Priorities: *Harvey W. Schadler*[1]; [1]General Electric, Corporate R&D Ctr., 4024 Windsor Dr., Niskayuna, NY 12309 USA

The Department of Defense (DoD) requested the National Materials Advisory Board to study critical materials and processing research and development (R&D) required to meet 21st-century defense needs. The study identified DoD materials needs and explored R&D priorities in five classes of materials: Structural and multifunctional materials, Energy and power materials, Electronic and photonic materials, Functional organic and hybrid materials, and Bioderived and bioinspired materials. This paper integrates the R&D priorities from all five materials areas and presents the studyís R&D recommendations. The study committee recognized that realizing the revolutionary new defense capabilities that materials science and engineering offer will depend on more than just R&D; innovative management will also be needed to reduce risks in translating fundamental research into practical materials, and to promote cross-fertilization of scientific fields. This paper also discusses these issues and presents the studyís recommendation for needed innovations in management.

2:40 PM Invited

Structural and Multifunctional Materials: *Harry A. Lipsitt*[1]; [1]Wright State University, Matls. Sci. & Eng., 1414 Birch St., Yellow Springs, OH 45387 USA

The Structural and Multifunctional Materials Panel focused on emerging materials and the processes used for their fabrication, with special attention to the types of multifunctionality that could be designed into a material. This paper discusses DoD structural materials development approaches and goals. It highlights the importance of lighter, stiffer, and stronger materials, and the need for materials to operate for long periods at high temperature with predictable degradation. These materials are necessary to improve vehicle mobility, maneuverability, transportability, and survivability. The panel identified four areas of R&D opportunity. These four opportunities are expanded upon, with emphasis on design of structural materials that are truly multifunctional. Investments in these research areas should result in advances that would yield many of the necessary new DoD materials. Such advances will: reduce development time and costs, modernize design criteria, predict and verify functionality, continuously monitor in-service health, and predict residual life.

3:10 PM Invited

Energy and Power Materials: *James Baskerville*[1]; [1]General Dynamics Company, Bath Iron Works, 700 Washington St., MS 6570, Bath, ME 04530 USA

The Energy and Power Materials Panel examined advanced materials and processes in this area. DoD needs for energy and power materials are many, among them, batteries for energy storage; capacitors for storage and release of pulsed power; fuel cells for efficient direct conversion of chemical to electrical energy; photovoltaics for harvesting energy; explosives for enhanced and tailorable lethality; and microturbines for powering unmanned aerial vehicles. The panel identified key materials aspects of each major application, and derived broad themes for materials research. Areas identified were those where DoD funding would be needed due to the lack of commercial interest. Successful pursuit of these themes will provide numerous benefits to the DoD, including: reduced development time and cost; increased energy density in storage devices; improvements in lethality of munitions; practical energy-harvesting devices; and reduced weight of energy and power systems, which will reduce soldier and system payload.

3:40 PM Break

3:55 PM Invited
Electronic and Photonic Materials: *Julia M. Phillips*[1]; [1]Sandia National Laboratories, Physl. & Cheml. Scis. Ctr., Organization 1100, MS 1427, PO Box 5800, Albuquerque, NM 87185-1427 USA

The Electronic and Photonic Materials Panel examined research needs for defense systems in electronics, optoelectronics, photonics, and microsystems (including sensors). The innovation of the private sector allowed the panel to consider which defense needs could be met by making use of commercial developments and which require DoD investment. The panel examined the following military needs that would benefit from electronic and photonic materials: detection, identification, and defense against or avoidance of threats; high fidelity imaging signals; communications systems; compact systems to transmit at very high power and high frequency; enemy identification and monitoring; dynamic camouflage/stealth; and health monitoring of equipment and personnel. While this panel considered a wide variety of military needs from several vantage points, ranging from individual devices or components to entire miniature systems, a number of common themes emerged that point to important areas for research. These research areas will be presented.

4:25 PM Invited
Functional Organic and Hybrid Materials: *Frank E. Karasz*[1]; [1]University of Massachusetts, Dept. of Polymer Sci. & Eng., Conte Rsrch. Ctr., Amherst, MA 01003 USA

The Panel on Functional Organic and Hybrid Materials addressed general concepts that will emerge in the next two decades. The panel predicts that organic materials of high and low molar mass will continue to increase their penetration of military materials applications because of the clear advantages they have in terms of functional flexibility, low weight, and facile processibilityóall leading to economic gain over the life cycle. The panel identified a number of research opportunities which will be presented. If these opportunities are pursued, the panel expects that: modeling will become a routine first step in organic materials development; synthesis and processing of organic materials will tend to converge; polymers of high purity with controlled microstructure will become available; aggregates of organic materials on the nanometer scale will yield new opportunities; combinations of low- and high-molar-mass organic molecules with inorganic materials will become widespread.

4:55 PM Invited
Bioderived and Bioinspired Materials: *Michael Jaffe*[1]; [1]NJ Center for Biomaterials and Medical Devices, 111 Lock St., Newark, NJ 07103 USA

The Panel on Bioinspired and Bioderived Materials focused on how the integration of biology and the physical sciences could result in greatly improved, lightweight, multifunctional materials for DoD. Materials derived from biology, for example, biological molecules as the active element in sensors, and materials inspired by biology were considered. The potential impact of applying biological paradigms to the development of materials to meet DoD requirements was reviewed indepth. This paper will present specific DoD opportunities in the areas of structural materials, functional materials, materials for chemical and biological warfare, wound healing, and human performance enhancement. The panel concluded that: biological toughening mechanisms offer a route to next generation lightweight, tough materials; preservation of biological function of biological molecules is a key driver for next generation of biologically-enabled devices; and in-vivo detection strategies to identify toxins and pathogens, including masked agents, may make it possible to detect a single agent molecule.

5:25 PM Closing Remarks Alan M. Lovelace

Measurement and Interpretation of Internal/ Residual Stresses: Composites

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Shaping and Forming Committee
Program Organizers: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Bimal K. Kad, University of California, Ames Laboratory, La Jolla, CA 92093-0085 USA

Wednesday PM — Room: 17B
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Don Brown, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; David C. Dunand, Northwestern University, Matls. Sci. & Eng. Dept., Evanston, IL 60208-3108 USA

2:00 PM Invited
Load-Sharing in Cemented Carbide Composites: *Aaron D. Krawitz*[1]; Jon W. Paggett[1]; [1]University of Missouri, Dept. of Mech. & Aeros. Eng., Eng. Bldg. E., Columbia, MO 65211 USA

Load-sharing characteristics of WC-Ni and WC-Co cemented carbides (particulate) composites were studied under uniaxial compressive loads using neutron diffraction on the SMARTS instrument at the Los Alamos Neutron Science Center. Cylindrical samples with volume fractions of 0.08, 0.16 and 0.30 Ni and 0.16 Co were employed. The strain response in each phase was monitored using compressive load cycles of 0-500-0, 0-1000-0, and 0-2000-0 MPa, some of which were sequentially repeated. The very substantial thermal residual stresses were measured before and after loading. In addition, the distribution of strain in each phase was monitored as a function of load. Measurements were made in the axial and transverse directions of the samples.

2:30 PM
Measurement and Modelling of the Strain Dependence of Phase Stresses During Plastic Straining of Aluminium/Nickel Composites: *Francis Delannay*[1]; FrÈdÈric Lani[1]; Laurence Ryelandt[1]; Catherine Salmon[1]; [1]UniversitÈ catholique de Louvain, PCIM, Place Sainte Barbe 2, Louvain-la-Neuve B-1348 Belgium

Composites consisting of a pure Al matrix reinforced with a network of sintered Ni-base fibres were prepared by squeeze casting. The composites inherit the in-plane transverse symmetry of the fibre orientations in the preform. The large ductility of these composites allows using them as model systems for assessing current models for predicting the plastic behaviour of ductile multiphase solids. The evolution of the phase stresses during tensile straining of two composites with 20 vol% and 30 vol% of Ni fibres was measured in the 3 principal directions by neutron diffraction. The results were compared to predictions of an elasto-plastic model of the composites based on the equivalent inclusion method and a mean field approach. The model aims at accounting for both the symmetry of the composite and for the connectivity of the reinforcing phase. These factors are shown to drastically affect the partition of plastic strains between the two phases.

2:50 PM
Processing Effects on Residual Stresses and Stress Partitioning in Al/AlCuFe Composites: *Fei Tang*[1]; Thomas Gnaupel-Herold[2]; Henry J. Prask[2]; Iver Eric Anderson[1]; [1]Iowa State University, Ames Lab., Metal & Ceram. Scis. Prog., Rm. 223, Metals Dvlp. Bldg., Ames, IA 50011 USA; [2]NIST Center for Neutron Research, 100 Bureau Dr., Stop 8562, Gaithersburg, MD 20899-8562 USA

A simple metal matrix composite (MMC) system was designed to investigate the effect of consolidation processing methods on residual stress strengthening. An unalloyed Al matrix was reinforced by Al-Cu-Fe quasicrystal (QXL) particulate, using either commercial purity (99.7%) or high purity (99.99%) powders (dia.<10µm) and consolidated by either vacuum hot pressing or quasi-isostatic forging. By neutron diffraction, the residual stresses were measured in composites reinforced by 15, 20, and 30vol.% QXL particles. Also, matrix/reinforcement load transfer was studied using an in-situ tensile frame. This tensile stress partitioning was studied at stress levels from 10 MPa to 150 MPa for the 30vol.% QXL composites. The ambient environment tensile properties of all MMC types were tested, while the elastic modulus was measured by an ultrasonic method. Microstructures and fractography of the composites were also characterized by SEM. Fund-

ing of this project is from DOE Basic Energy Sciences under contract number W-7405-Eng-82.

3:10 PM
Dynamic Response of Residual Stresses Due to Rapid Temperature Changes in Saffil Reinforced AA339 Aluminum: G. Langelaan[2]; *S. Saimoto*[1]; W. J. Baxter[3]; [1]Queenís University at Kingston, Matls. & Metallurgl. Eng., 60 Union St., Rm. 207, Nicol Hall, Kingston, Ontario K7L 3N6 Canada; [2]K. U. Leuven, Dept. MTM, Heverlee Belgium; [3]Formerly of General Motors, R&D Ctr., Warren, MI USA

The residual stresses in the matrix of an AA339 aluminum 15% (vol.) Saffil composite were measured by X-ray diffraction at 100∞C and 400∞C , after temperature cycling were performed. A special heating stage was developed with a very low thermal expansion to minimize systematic errors due to specimen alignment and a very low thermal mass to permit rapid temperature changes. A linear position sensitive detector enabled diffraction peak profiles to be measured in 5 seconds. The slow thermal cycling was at 10∞C/min for both heating and cooling whereas that for rapid cycling was 200∞C/min between 100∞C and 400∞C. These conditions nearly simulates the temperature responses for the piston head on changing between idling and acceleration. The residual stress responses to thermal cycling for the as-cast and samples in the T5 condition were compared by means of a stress-temperature plot. The greater stability of the T5 specimens is attributed to the associated microstructural reactions to the heating/cooling cycles in the distribution of precipitates.

3:30 PM
Strain Evolution After Fiber Failure in a Single Fiber Al/Al2O3 Composite: *Jay C. Hanan*[1]; Sivasambu Mahesh[2]; Ersan Ustundag[1]; Irene J. Beyerlein[2]; Donald W. Brown[3]; Mark A.M. Bourke[3]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Theoretl. Div., Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

The evolution of elastic strain with applied cyclic tensile loading was monitored for each phase of an Al-matrix/Al2O3-fiber composite using neutron diffraction. The Al2O3 fiber fractured during the first loading while the matrix deformed plastically. A finite element model was developed to interpret the diffraction strain data. The model was shown to account for micromechanical phenomena such as fiber breaking, matrix plasticity and interfacial slipping.

3:50 PM Break

4:05 PM Invited
Measurement and Modeling of Internal Stresses in a Ti-6Al-4V/SiC Composite: *Hahn Choo*[1]; Partha Rangaswamy[2]; Mark A.M. Bourke[3]; [1]The University of Tennessee, Matls. Sci. & Eng., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, ESA-DE, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

Internal stresses in a Ti-6Al-4V alloy reinforced with 35 volume % SiC continuous fibers were systematically studied using in-situ high temperature neutron diffraction. First, we present the thermal residual stresses (TRS) in the matrix and fibers within the composite measured from axial and transverse directions during heating from room temperature to 1170K. Second, the anisotropic thermal expansion behavior of the constituent phases in the composite as well as the bulk expansion behavior will be discussed in terms of the TRS and its inelastic relaxation at elevated temperatures. Finally, we will present results from a recent study on the load partitioning in the composite during uniaxial tensile loading at 700K. The neutron diffraction results, coupled with a finite element analysis, will provide insights to the micromechanics of the TRS evolution, thermal expansion behavior, and load-partitioning in a continuous fiber reinforced composite at elevated temperatures.

4:35 PM
Deformation of Fiber Reinforced Bulk Metallic Glass Matrix Composites: *Bjorn Clausen*[1]; Seung-Yub Lee[1]; Ersan Ustundag[1]; Mark A.M. Bourke[2]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, MST-8, PO Box 1663, MS H805, Los Alamos, NM 87545 USA

Bulk metallic glasses (BMG) have superb mechanical properties: yield strength of above 2 GPa, fracture toughness of up to 55 $MPa.m^{1/2}$ and elastic limit of 2%. However, monolithic BMGs fail catastrophically under unconstrained deformation. To overcome this problem, BMG matrix composites with fiber reinforcements were proposed. We have recently investigated the deformation behavior of composites with W, Mo, Ta and Fe fibers. Loading measurements were performed using in-situ neutron diffraction to determine the lattice strains in the fibers. The diffraction data were then combined with finite element modeling to determine behavior of the matrix. It was shown that usually the reinforcements yield first, then start transferring load to the matrix. The reinforcements possess different yield strengths, elastic constants, interface strengths and coefficient of thermal expansion (CTE) values. The effects of these parameters on the overall composite deformation will be described.

4:55 PM
Micro and Macro Residual Stresses in a Two-Phase Material: *Mohammed Belassel*[1]; [1]Proto Manufacturing, Ltd., 2175 Solar Crescent, Oldcastle, Ontario N0R 1L0 Canada

Residual stresses are generated during processing such as shaping, machining, rolling grinding, heat treatment, etc. These residual stresses can be defined as: Micro-stresses and Macro-stresses and be measured using X-ray diffraction technique, separately in each phase. In this study, the residual stresses are generated in a two-phase material, pearlite and measured after heat treatment and plastic deformation. The results obtained were compared to other experimental techniques and theoretical models such as Self-Consistent. The results also have showed that the second phase cementite can play an important role in the generation of both types of stresses. A methodology for stress measurement in a two phase material have been developed.

5:15 PM
High-Temperature Deformation of Silicon Nitride and its Composites: *Geoffrey A. Swift*[1]; Ersan Ustundag[1]; Bjorn Clausen[1]; Mark A.M. Bourke[2]; Hua Tay Lin[3]; Chien Wei Li[4]; [1]California Institute of Technology, Matls. Sci., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; [4]Honeywell Corporation, Morristown, NJ 07962 USA

The deep penetration of neutrons in most materials allows in-situ studies in extreme environments. This advantage of neutron diffraction was utilized in the investigation of strain and texture evolution during high-temperature deformation of monolithic Si3N4 and its composites with SiC particulates. Tension experiments were performed near 1400∞C using the new SMARTS diffractometer at the Los Alamos Neutron Science Center. The diffraction data provided information about thermal expansion coefficients and elastic constants at high temperature. In particular, the hkl-dependent strains were interpreted by employing self-consistent modeling.

Mercury Management: Mining Operations

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizer: Larry Twidwell, Montana Tech of the University of Montana, Metallurgical and Materials Engineering, Butte, MT 59701 USA

Wednesday PM Room: 1B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Courtney Young, Montana Tech of University of Montana, Sch. of Mines & Eng., Butte, MT 59701 USA; Kumar Ganesan, Montana Tech of University of Montana, Sch. of Mines & Eng., Butte, MT 59701 USA

2:00 PM Invited
Update: Environmental Assessment of Gold Processing by Mercury Amalgamation in a Developing Country: *Courtney A. Young*[1]; Christopher H. Gammons[1]; Rich L. McNearny[1]; Darell G. Slotton[2]; Fernando Quispe[3]; Estaben Aquino[3]; Alfredo Camac[3]; [1]Montana Tech of the University of Montana, Sch. of Mines & Eng., 1300 W. Park, Butte, MT 59701 USA; [2]University of California-Davis, Dept. of Environml. Sci. & Policy, 2132 Wickson Hall, One Shields Ave., Davis, CA 95616 USA; [3]Universidad Nacional del Altiplano, Mining & Geologl. Eng., Puno Peru

Gold ores mined in developing countries are being processed by mercury amalgamation, a technique long banned in the US due to mercury toxicity and its effects on the environment unless it includes a retort operation for mercury recovery. Amalgamation is a simple process that involves contacting a ground, slurried concentrate or ore with mercury in a slightly agitated vessel usually for several hours. The amalgamated mercury is separated by gravity and then filtered through a fine cloth to collect the gold amalgam as a sponge which is then fired to remove final remnants of mercury and produce high quality gold. However, in Peru and other developing countries in South America, tailings are discarded into uncontrolled drainages and ponds and the sponge is fired in open air. Clearly, in this case, amalgamation is not environmental friendly and, to make matters worse, is typically conducted by families without proper protection. Furthermore, the technique is inefficient. For the past two years, faculty members from Montana Tech and the Universidad Nacional del Altiplano in Puno, Peru have teamed to study the La Rinconada Mine Complex located in southern Peru. The mine is over 5,000 meters above sea level and exists at the foot of a receding glacier in the Andes Mountains. Sediment and water samples were taken for mercury analysis from (1) around the mine, (2) an old mercury furnace site, (3) one of approximately fifty amalgamation operations, (4) the main drainage and tailings pond, (5) various downstream water bodies including a small lake and river three kilometers away, and (6) a site in the river four kilometers north of Lake Titicaca. In addition, a representative suite of predator fish in Lake Titicaca were analyzed for Hg concentration. Results are presented and discussed with relevance to safety and health concerns for the miners as well as the people in the downstream communities.

2:30 PM Invited
Reduction of Mercury Emissions in Small Scale Mining Using Advanced Gravity Concentration: *Hermann Wotruba*[1]; Wolfram M¸ller[1]; [1]University of Technology-Aachen, Aachen Germany

Amalgamation is widely used to extract especially fine gold from primary and secondary deposits in small scale mining operations. To avoid the environmental impact by the use of mercury in gold processing pure gravity concentration can be used. If a final step to upgrade the gold concentrate with mercury is necessary, a retort can be used which leads to a minimization of mercury emissions up to 90% compared to traditional methods. The authors have experience in processing gold from deposits of different origin with high recoveries. Even ultrafine and interlocked gold of primary deposits could be treated successfully. The equipment used therefore can be adapted to the traditional processing units. One of the main advantages of the tested method is the full acceptance by the small scale miners. In the first part of the paper different methods applying mercury in gold processing in South American small scale operations and their impact to the environment are described. In the second part successful, mercury free, operations based on gravity concentration only that have been carried out in Venezuela, Bolivia and Brazil are presented. Recoveries up to 90% could be achieved and the method was accepted by the small scale miners.

2:55 PM Invited
Mobility of Mercury in Aged Gold Mine Tailings: *Kumar Ganesan*[1]; [1]Montana Tech of the University of Montana, Dept. of Environml. Eng., Montana Sch. of Mines, 1300 W. Park, Butte, MT 59701 USA

Elevated mercury levels are expected in aged gold mine tailings due to residual mercury from amalgamation process. The fate of mercury in tailings is not well understood. Its potential health risk depends mainly on the degree to which mercury can be mobile in soil. Mercury is removed from soil by evaporation, plant uptake, microbial activities, leaching, and other physico-chemical processes. In this research the amount of mercury lost by evaporation was evaluated. Several aged gold mine tailings in Montana were sampled for total mercury and its flux into the atmosphere. Mercury levels in tailings were higher than the general background levels in most sites. The mercury levels in some cases were as high as 10,000 ug/g. The field measurements using flux chambers indicated a wide range of mercury flux from aged gold mine tailings. The mercury flux ranged from 0-11,000 ng/m2/hr. The mercury loss by evaporation helps the soil to rid of mercury but the mercury vapor then will participate in the atmospheric mercury cycle. The low solubility of elemental mercury in water helps to minimize mercury leaching from tailings. However, conversion of elemental mercury to more soluble forms by bio-geo-chemical processes can enhance its mobility posing increased health risk.

3:20 PM Invited
Mercury Extraction from Precious Metals Ores: *Charles Washburn*[1]; Charles Gale[1]; [1]Summit Valley Engineering and Equipment, 450 E. 1000 N., Salt Lake City, UT 84054 USA

The authors review mercury scources, and contaminent streams, with particular emphasis on mercury that is extracted from precious metal ores. The design and operation of both large and small mercury distillation facilities are detailed. The relevent properties and characteristics of mercury and mercury vapor are presented. Facility engineering with respect to industrial hygene, area ventilation, and material handling is presented.

3:45 PM Break

4:00 PM Invited
Alkaline Sulfide Treatment of Mercury: *Suzzanne Nordwick*[1]; [1]MSE Technology Applications, 2000 Tech. Way, Butte, MT 59701 USA

The metallurgical treatment of mercury-containing materials for precious or base metals recovery imposes many processing and environmental challenges. Mercury is usually present as elemental or cinnabar. Historically, mercury has been removed from mined ores pyrometallurgically by roasting; but, hydrometallurgical methods have also been employed. For example, alkaline sodium sulfide solution acts as an universal solvent for most mercury compounds. While, most other metals are highly insoluble in this solution. This allows for a high degree of selective separation of mercury from other metals with the exception of tin, antimony and arsenic. Mercury leached with a solution of sodium hydroxide and sodium sulfide or sulfur can be recovered by cementation or stabilized through sulfide precipitation. Hydrometallurgical technology for the pretreatment of mercury removal from gold, silver, and base metal ores using alkaline sulfide leaching will be discussed with a focus on alkaline sulfide leaching of mercury sulfide-containing materials.

4:25 PM Invited
The Treatment of Mercury Bearing Tetrahedrites from Solvakia: *Corby G. Anderson*[1]; [1]Montana Tech of the University of Montana, Ctr. for Adv. Minl. & Metallurgl. Procg., 1300 W. Park, Butte, MT 59701 USA

In the Rozonava deposit in Slovakia tetrahedrite mineralization has historically been processed pyrometallurgically for its silver and copper values. These tetrahedrites contain large amounts of mercury which pose both a processing and environmental challenge. This paper outlines the processing of these materials by industrial alkaline sulfide hydrometallurgical methods. Relevent economics will be discussed.

4:55 PM Invited
Small Scale Artisanal Gold Operation in the Nambija District (Ecuador): *David F.J. Y. Bastin*[1]; Vicente Mata Balseca[1]; Jean Frenay[1]; [1]University of Liege, Metall. & Minl. Procg., Chemin des Chevreuils 1, Bldg. 52, Liege 4000 Belgium

The Nambija District is one of the gold district of Southeastern Ecuador mainly operated by artisanal and small scale mining. This paper describes a typical small scale (50 to 80 t/d) artisanal operation encountered in this region. The flowsheet of operations is described with special emphasis for the Hg participation. A metallurgical balance of the gravimetric circuit was calculated. Mineralogical observations of some of the amalgamation tails were realized. The different tails were subjected to orientative gravimetric tests to recover Au and Hg. Preliminary results are discussed.

Microstructural Processes in Irradiated Materials: Stainless Steels, Radiation Induced Segregation, Ion Irradiation and Other Materials II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

Wednesday PM — Room: 11A
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Jeremy Busby, University of Michigan, Nucl. Eng. & Rad. Scis., Ann Arbor, MI 48108 USA; Lance Snead, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37830-6138 USA

2:00 PM
Role of Irradiated Microstructure and Microchemistry in Irradiation Assisted Stress Corrosion Cracking: *Gary S. Was*[1]; Jeremy Busby[1]; Matthew Sowa[1]; Ryan Dropek[1]; Mark Hash[1]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., 1921 Cooley Bldg., Ann Arbor, MI 48109-2104 USA

Irradiation has a profound effect on the stress corrosion cracking propensity of austenitic alloys in high temperature water. Irradiation assisted stress corrosion cracking (IASCC) has been well documented both in the laboratory and in service over the past two decades. Numerous studies have shown that the degree of intergranular stress corrosion cracking increases with dose. However, the microstructure is simultaneously changing in several ways (dislocation loops, voids, segregation and hardening) and, not surprisingly, they all correlate with increased cracking susceptibility. As a consequence of their simultaneous development, the attribution of IASCC to one or more of these features has been difficult to establish. This paper will examine the changes in microstructure, microchemistry and hardening in austenitic alloys as a result of irradiation and how they can affect stress corrosion cracking individually and collectively. Potential mechanisms by which such changes could impact stress corrosion cracking susceptibility in high temperature water will be presented.

2:45 PM
Interaction of Defects with Grain Boundaries and Implications for Microstructure Evolution in Irradiated Materials: *Maria J. Caturla*[1]; Alison Kubota[1]; Jaime Marian[1]; Brian D. Wirth[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., PO Box 808, L-353, Livermore, CA 94550 USA

Void denudation close to grain boundaries has been observed in irradiated materials. In some cases grain boundary migration also occurs during irradiation. To gain basic understanding of these phenomena we have performed atomistic simulations of the interaction between defects and grain boundaries for different defect types (vacancies and interstitials) and sizes as well as different grain boundary types. The effect of He in these interactions is also discussed. We study changes in grain boundary configuration as the defect density increases. A simple kinetic Monte Carlo model of defect evolution close to grain boundaries is developed. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

3:05 PM
Ab Initio Atomic-Scale Modeling of the Zr-I Interactions in Conditions of Stress-Corrosion Cracking: *Christophe Domain*[1]; Alexandre Legris[2]; [1]EDF R&D, MMC, Site des Renardieres, Route de Sens, Ecuelles, Moret sur Loing 77818 France; [2]LMPGM, USTL, Bat C6, Villeneuve DíAscq 59655 France

We have investigated the iodine-zirconium interactions using ab initio atomic-scale calculations in order to elucidate possible mechanisms of stress corrosion cracking. Our calculations show that for the gas pressures estimated during the reactor power transients, the reduction of the zirconium effective surface free energy induced by the gas adsorption is significant (up to 50%). In particular, for a given iodine partial pressure, the surface reduction for the basal planes is higher than for the prismatic ones, in agreement with the experimental observation of a cleavage along the basal planes. We have also estimated the iodine surface diffusion coefficient, and its high value (about 10-6 cm2/s at 600K) indicates that iodine is mobile enough to follow the crack tip during the cracking experiments reproduced in laboratory conditions. Our results clearly rules out the influence of absorbed iodine during the cracking process, its concentration being totally negligible.

3:25 PM
Effect of Irradiation on the Microstructure and Microchemistry of Grain Boundary-Engineered Austenitic Alloys: *Ryan Dropek*[1]; Gary S. Was[1]; James I. Cole[2]; Jian Gan[2]; Todd R. Allen[2]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., 2940 Cooley Bldg., Ann Arbor, MI 48109-2104 USA; [2]Argonne National Laboratory, Nucl. Tech. Div., Idaho Falls, ID 83403 USA

Changes to the composition and structure of grain boundaries have the potential to improve the resistance to irradiation assisted stress corrosion cracking of austenitic stainless steels. Steels of composition Fe-18Cr-8Ni-1.25Mn and Fe-16Cr-13Ni-1.25Mn were chosen for modifications of the grain boundary composition and structure. Composition changes included alloying additions of Zr to the 18Cr-8Ni alloy and Mo and P to the 16Cr-13Ni alloy, followed by the application of special heat treatments designed to segregate Cr to the grain boundary prior to irradiation. Structure changes entailed enhancing the coincident-site lattice boundary fraction over that in an annealed structure by a combination of deformation and thermal treatment. Composition and structure changes were applied individually and collectively to selected alloys that were then irradiated with 3.2 MeV protons to 1 dpa at 400∞C. The effect of grain boundary engineering on microchemistry, along with associated changes in microstructural development due to bulk composition changes are reported.

3:45 PM Break

4:15 PM
The Importance of Chemical Disorder in Radiation-Induced Amorphization of SiC: *Xianglong Yuan*[1]; Linn W. Hobbs[1]; [1]Massachusetts Institute of Technology, Depts. of Matls. Sci. & Eng. & Nucl. Eng., Rm. 13-4054, 77 Massachusetts Ave., Cambridge, MA 02139-4307 USA

Structural freedom considerations based on rigidity theory argue that the multiply corner-sharing network of silicon carbide (four tetrahedra sharing each corner) should not be easily amorphized, contrary to experimental results from electron and heavier particle irradiation experiments. We have used a combination of molecular dynamics simulation (with an empirical potential) and topological analysis to show that SiCís facile amorphizability is controlled by the potential for chemical disorder in this material, with a chemical disorder threshold (defined by the ratio of homonuclear to heteronuclear bonds) about 0.3. Furthermore, we have shown that fully chemically-disordered SiC (random site occupation) undergoes a reversible glass transition, with a MD-model glass transition temperature about 3000 K. The implications for radiation-induced amorphization of SiC and other isostructural compounds are addressed.

4:35 PM
Effect of Dislocation Network on Void Swelling: *Sergei L. Dudarev*[1]; A. A. Semenov[2]; C. H. Woo[2]; [1]EURATOM/UKAEA Fusion Association, Culham Sci. Ctr., Oxfordshire OX14 3DB UK; [2]Hong-Kong Polytechnic University, Dept. of Mechl. Eng., Hung Hom, Kowloon Hong-Kong

We found that competition between nucleation of voids and (three-dimensional) diffusion of vacancies, interstitial atoms and interstitial atom clusters in the presence of heterogeneously distributed dislocations in the vicinity of a grain boundary in an alloy under irradiation can be responsible for the experimentally observed effect of heterogeneous void swelling. The new model is able to describe a number of significant features characterising the effect, including the formation of void denuded zones, the variation of the density of dislocations as a function of distance to the grain boundary and the formation of unusually large voids in the immediate vicinity of grain boundaries. Profiles of void swelling calculated using the new model are found to agree quantitatively with experimental observations. This finding shows that the spatial distribution of the dislocation component of microstructure may play a determining part in the formation of heterogeneous profiles of voids swelling near grain boundaries.

Products, Applications, and Services Showcase: Reduction Technology & Laboratory Analysis

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: David V. Neff, Metaullics Systems Company, Solon, OH 44139 USA

Wednesday PM — Room: 5A
March 5, 2003 — Location: San Diego Convention Center

Session Chair: TBA

2:00 PM
CLAYBURN DRI-BARRIER MIXÆñIts Application as a Barrier Lining in Reduction Cells: *Ron Smith*[1]; [1]Clayburn Industries, Ltd., Al Prod. Div., 33765 Pine St., Abbotsford, British Columbia V2S 5C1 Canada

CLAYBURN DRI-BARRIER MIXÆ is a unique, penetration resistant dry vibratable barrier lining developed specifically for use below the cathode in aluminum electrolytic cells. In our presentation we will discuss the developmental history as well as the technical application and installation of Dri-Barrier MixÆ. In addition, we will discuss Clayburnís successful production and installations of Dri-Barrier MixÆ at smelters in the emerging Chinese aluminum industry.

2:20 PM
New Busbars for Smelters: *Serge Risser*[1]; Gerard Hudault[1]; [1]Aluminium Pechiney Lannemezan, Lannemezan F-65300 France

With increasing number of cells in state of the art aluminum smelting technology, smaller busbars are being replaced by bigger ones, which allows an accurate electrical balance and better cooling efficiency. Aluminium Pechiney has improved its welding technology and has developed a new busbar production route for its AP18, AP30 & AP50 technology. The recently upgraded horizontal continuous casting machine at Lannemezan plant supplies busbars that meet customersí highest quality requirements. Other achievements at Dunkerque, Tomago and Alouette smelters are expected to develop further applications of this technology. The presentation will review updated market information, the use of busbars, the different aluminium grades for conductors, alloys for stems, and key parameters for a safe and successful cast. Furthermore, technical information about facilities will be given.

2:40 PM
The MOELLER Direct Pot Feeding System for a Smooth and Constant Pneumatic Transport of Secondary Alumina to the Electrolyte Cells: *C. Duwe*[1]; [1]Moeller Materials Handling GmbH, Pinneberg Germany

The MOELLER Direct Pot Feeding is a high efficient and full automatically facility for a smooth and wear-resistant constant pneumatic transport of secondary alumina to the electrolyte cells. This pneumatic transport system is a combination of a dense phase feeding from a storage silo to intermediate bins at the electrolyte cells and a SFS (Super feeding system) air slide system directly to each of the electrolyte cells. The TURBUFLOW dense phase feeding as well as the SFS (Super feeding system) air slide system are approved systems which preserves the particle size distribution and the flow ability of the alumina. The main conveying air volume is separated in the intermediate bin and do not influences the situation of feeding secondary alumina from intermediate bin via point feeders into the electrolytic cell. The mass flow of secondary alumina is blocked automatically when the bunker of an electrolytic cell is full and the bulk material cone level has reached the fill spout discharge opening. The fluidising of the bulk material inside the SFS air slide system still works and ensures no remarkable variations of the bulk density. When secondary alumina is removed from the bunker of the electrolytic cell, the pneumatic transport starts again automatically and a constant and reliable mass federate to the pots is ensured. This most competitive system for the pneumatic feeding of secondary alumina to electrolytic cells is one of the components to ensure the quality of the aluminium production on the highest possible level.

3:00 PM
New Generation of Tapping Vehicles: *Knut Prestnes*[1]; [1]HMR Group AS, Prestnesvegen 68, Husnes 5460 Norway

In effort to increase productivity, industry looks for more effective ways to deliver smelted aluminium to the cast house. Capacity of tapping is the key. But in tapping vehicles, capacity increase is limited by two factors: maximum load per axle and lifting height with vacuum tapping. A new tapping concept elaborated and tested in one of the smelters in Norway, reduces these obstacles. A new generation of tapping vehicles has been developed. World patented system allows tapping up to 13 tons (theoretically up to 18-20 tons) of aluminium per one cycle. With this capacity one tapping vehicle can tap from the pots and deliver to the cast house, located at 1 km distance, 12 tons of smelted aluminium per hour. This is industry worldwide record. Additionally the whole process is one-man operated and controlled. Together with simplified crucible cleaning system and quick tapping-, siphon-tube changing they constitute important cost reduction factors.

3:20 PM Break

3:40 PM
Rockwell, Vickers or Nanoindentation?: *Seth Downs*[1]; Dehua Yang[1]; Oden Warren[1]; Thomas Wyrobek[1]; [1]Hysitron, Inc., 5251 W. 73rd St., Edina, MN 55439 USA

Recent trends in miniaturization of devices and demands for high-performance materials dictate a change in the way that materials are characterized. Understanding of the structure, properties and role of processing at the nanoscale is crucial to achieving the desired performance, regardless of the size scale of the final product. Nanomechanical characterization is presented as a new solution for materials testing, expanding on the range of capabilities of traditional hardness testing. Nanoindentation provides superior lateral and vertical resolution, allowing testing of surface properties or single phases of multi-phase materials. Typical nanoindentation and nanoscratching provide measurement of mechanical properties such as modulus, hardness, fracture toughness, friction coefficients and wear resistance. New techniques, such as acoustic emission monitoring and stiffness and modulus mapping, allow investigation of the initial stages of fracture and characterization of interfaces. An overview of the capabilities and techniques offered by Hysitronís nanomechanical characterization instruments will be presented. Comparison and interrelationships of Rockwell, Vickers and nanoindentation will also be discussed.

4:00 PM
Management, Molten Metal and Protective Clothing: *Larry Stinson*[1]; [1]Silver Needle Inc., 402 Main St., Kellogg, ID 83837 USA

This presentation will combine all the strategic and economic goals that work together to make a safety program not only succeed for a company but to actually be a benefit and asset as well. Topics to be covered include: 1. Liability; 2. Location; 3. Personnel; 4. Clothing Committee and 5. Economics.

4:20 PM Cancelled
Pulsar: Total Analytical Flexibility for Solids, Powders and Solutions: *Charles Belle*[1]; Phil Bennett[1]; Bruce MacAllister[1]; [1]Leeman Labs, Inc., 6 Wentworth Dr., Hudson, NH 03051 USA

Recycling - General Sessions: General Recycling

Sponsored by: Light Metals Division, Jt. LMD/EPD-Recycling Committee
Program Organizers: Han Spoel, Spalco Metals Inc., Toronto, Ontario M5R 1W8 Canada; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday PM — Room: 3
March 5, 2003 — Location: San Diego Convention Center

Session Chair: Han Spoel, Spalco Metals Inc., Toronto, Ontario M5R 1W8 Canada

2:00 PM Announcements

2:10 PM
PickingñAn ìOldî Process for Separation of Non-Ferrous Metals?: *Stefan Mutz*[1]; Thomas Pretz[1]; [1]Aachen University of Technology, RWTH Aachen, Wuellnerstr. 2, Aachen D-52062 Germany

The term ìSORTINGî implies the separation of a blended material into two or more products, all or both of which may be classified by the different material characteristics in the preparation technology. The oldest sorting procedure, by handpicking, is today used infrequently to separate different material mixtures, because of the considerable cost of labour and the strict hygiene regulations at the workplaces in Europe. In principle, manual sorting is only used to solve separation tasks, in which mechanised sorting could not be utilised. It should be noted that picking is one of the most selective processing routes. Current developments in the fields of computers systems and sensor engineering makes the creation of automatic picking devices possible, especially where efficiencies far beyond the realms of handpicking are desired. In the future therefore, automatic picking applications will not only be utilised in the already common applications, such as waste glass assortment by colour, but also in a variety of complex material mixtures e.g. old shredded cars. Thus, automatic picking, supported by high performance computer systems and new sensor technology, becomes an attractive alternative in many areas of waste processing. However, in order to merge the application of the automatic sorting procedures with the complex material flows of the recycling industry, a substantial research and development need still exists. The demands of the users are to sort increasingly larger mass flows, which themselves constitute larger material varieties. A further demand is the need to accurately identify and sort the single grains as the mass flow rises. This lecture compares the traditional sorting procedures, such as hand picking with todayís state of the art methods of classification within the range of automatic picking. Apart from the potential technological developments, some operational issues and future opportunities for this technology are illustrated especially in the fields of NF-metals.

2:40 PM
Recycling Rates of Waste Home Appliances in Taiwan: *Esher Hsu*[1]; Chen-Ming Kuo[2]; [1]National Taipei University, Dept. of Stats., 67 Sec., 3 Min-Sheng E. Rd., Taipei 104 Taiwan; [2]I-Shou University, Dept. of Mechl. Eng., 1 Sec., 1 Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

On July 5, 1997, Environmental Protection Administration of Taiwan publicized the recycling regulation of waste home appliances that include four items, namely, television, refrigerator, washing machine, and air conditioner. It is believed that this regulation pioneers the law enforcement of waste home appliances in the world. The objective of this study is to provide helpful base for amending recycling rates of waste home appliances. This study investigates the collection and recycling costs of waste home appliances in accordance with these newly established routes and facilities, respectively. Cost survey was conducted among collectors and recycling plants of waste home appliances; consequently, the collection and recycling costs were analyzed, correspondingly. Results provide some suggestions regarding recycling route, recycling system, recycling rates paid by manufactures and importers, recycling subsidies to collectors and recyclers. Those suggestions may provide useful policy implications to EPA for the future decision-making regarding the recycling of waste home appliances.

3:10 PM

Economic Assessment of the European Union Directive for Recycling of End-of-Life Vehicles: *Randolph E. Kirchain*[1]; Adriana Diaz-Triana[1]; [1]Massachusetts Insitute of Technology, Matls. Sys. Lab., 77 Massachusetts Ave., E-40 Rm. 202, Cambridge, MA 02139 USA

The Materials Systems Laboratory at the Massachusetts Institute of Technology (MSL) is conducting research on the economic and environmental implications of End-of-Life Vehicle (ELV) recycling, in the context of the recent European Community Directive 00/53/EC. This Directive sets explicit recycling targets for automakers. Specifically, recycling/recovery must be increased to 80/85% by 2006 and to 85/95% by 2015. Two key goals for the Directive are: 1) to prevent waste, preferably through improved design, but also through more intensive recovery and 2) to improve the environmental performance of those operators involved in the treatment of ELVs. MSL has developed a framework for the analysis of vehicle recycling in the US, through the use of process-based Technical Cost Models (TCMs). TCMs describe the recycling system, with different arrays for operators and their interactions, taking into account technical information about all relevant processes. TCMs have proven to be powerful tools for evaluating cost implications when looking at different materials and/or processing alternatives, providing the decision-maker with an accessible means for comparing distinct approaches as to inform on technical decision. MSL has recently developed recycling TCMs to reflect the economic and environmental dimensions of vehicle recycling in two different European Member States: Portugal and The Netherlands. The infrastructure for ELV processing in Portugal is based on dismantling and limited shredding and materials separation activities, while the more mature Dutch system promotes increased dismantling of materials for recycling. The activity level of the dismantling, shredding and materials separation operators and the associated costs are forecasted in the context of each country. The analysis of these different ELV recycling infrastructures through the development of appropriate TCMs is a valuable approach to understand key economic factors often overlooked when considering the implementation of recycling policies.

3:40 PM Break

3:55 PM

Radionuclide Surrogate Decontamination of Metal Using Electroslag Remelting: *David K. Melgaard*[1]; Gregory J. Shelmidine[2]; James A. Van Den Avyle[2]; Martin A. Molecke[3]; [1]Sandia National Laboratories, Div. 1835, MS 0889, PO Box 5800, Albuquerque, NM 87185-0889 USA; [2]Sandia National Laboratories, Div. 1835, MS 1134, PO Box 5800, Albuquerque, NM 87185-1134 USA; [3]Sandia National Laboratories, Div. 6141, MS 718, PO Box 5800, Albuquerque, NM 87185-0718 USA

A significant emerging recycling, waste minimization problem stems from production of power using nuclear fuel. Premium quality metals used in nuclear plants become contaminated and are unsuitable for reuse. Consequently decontamination studies were conducted to evaluate the decontamination capacity and partitioning effectiveness of Electroslag Remelting (ESR). Eight laboratory melts were conducted to evaluate the effectiveness of using ESR to transport radionuclides from the liquid metal phase to the liquid flux or slag phase. To determine the decontamination effectiveness, several surrogate compounds for uranium and plutonium were added to the melts. During the melts, the melting rate was varied, typical slag additives were used, and two different slag compositions were evaluated. The resultant post-test ingots and slag were then analyzed to determine the partitioning of the surrogates. ESR generally proved very effective in removing contaminates from the metal with a few noted exceptions.

4:25 PM

A Process to Recycle Glass Fibers from Glass Manufacturing Waste: *B. J. Jody*[1]; J. A. Pomykala[1]; E. J. Daniels[1]; J. C. Wells[2]; C. E. Davis[2]; [1]Argonne National Laboratory, Energy Sys. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Saint-Gobain Vetrotex America, 4515 Allendale Rd., Wichita Falls, TX 76310-2199 USA

This paper describes a one-step thermal process, developed at Argonne National Laboratory, to clean and purify the glass waste that is generated during the glass forming process so that the waste could be recycled into new glass fibers or new glass products without adversely impacting the operations of the industry or the quality of its products. The process involves shredding the waste to a manageable size and then processing it at temperatures below its melting point to remove the polymer-coating layer. Preliminary cost analysis of the process shows a potential payback of less than 2 years. The glass industry generates more than 60,000 tons of glass manufacturing waste annually in the glass forming process alone. Recycling of this waste stream, can keep this non-biodegradable waste out of the landfills. In addition use of recycled glass lowers energy costs, by an average of about $5 per ton compared to using virgin raw materials to make virgin glass fibers. This will also reduce the emission of CO_2 by 17,400 tons a year and will also reduce the emission of nitrogen oxides.

4:55 PM

Para-Eco Incinerator Ash Processing System for Cleaning Slag with Recovering Metals: *Kiyoshi Takai*[1]; Ken-ichi Ohkura[1]; Masao Suzuki[2]; [1]Rasa Corporation, Chitose Bldg., 2-9-4 Nihonbashi Kayabacho, Chuo-ku, Tokyo 103 Japan; [2]AI-Tech Associates, Tokura 3-38-13, Kokubunji-shi, Tokyo 185-0003 Japan

Para-Eco Incinerator Ash Processing System for cleaning slag has been developed through a pilot plant study of 7 years. The major part of the system is an electric furnace, heating to 1773K. The feed material consists of a municipal solid waste incinerator ash mixture with the reducing agent of coke and inorganic flux of calcium and magnesium. With this furnace system three kinds of products are produced. They are an inorganic molten salt that is slag, molten iron alloy absorbing non-ferrous metals, and fly ash. Due to a reducing atmosphere in the furnace, the slag does not contain any metal components as well as chlorine component just like a natural rock and/or aggregate. A retreatment plant modifies this slag into a practically useful aggregate. The plant consists of reheating step followed by a slow cooling step for crystallizing the slag. The iron components in the ash are reduced to molten iron droplets, which concentrate at the furnace bottom. The non-ferrous metal components such as copper, chromium, and others are reduced to each individual metallic state. Accordingly, they are absorbed into the molten iron but not into the molten slag. Volatile metals of lead, zinc, cadmium and others as well as sodium, potassium, and chlorine go into the fly ash again. A solvent extraction method and/or other appropriate procedures are applicable to extract metal components from this fly ash.

Science and Technology of Magnetic and Electronic Nanostructures: Functional Nanostructures: Ferroic Materials

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee

Program Organizers: Ramamoorthy Ramesh, University of Maryland, Department of Materials and Nuclear Engineering, College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Wednesday PM   Room: 15A
March 5, 2003   Location: San Diego Convention Center

Session Chair: Timothy D. Sands, Purdue University, Sch. of Matls. Eng., W. Lafayette, IN 47907 USA

2:00 PM

Bringing Oxides into the Silicon World: Strategies to Integrate Functional Oxides Epitaxially with Silicon: *Darrell G. Schlom*[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., 108 MRI Bldg., Research Park, University Park, PA 16802-6602 USA

Oxides possess many unparalleled properties, but we live in the silicon age. Hybrid structures in which the functional properties of oxides can be exploited in combination with silicon offer exciting opportunities for devices. The question is how to integrate such chemically different materials without degrading either material to create an electrically-useful interface. Which oxides are better suited for integration and how does one go about integrating them with silicon? This question is the focus of this talk. A methodology will be described, based on thermodynamics and oxidation kinetics, for the integration of functional oxides with silicon with the goal of achieving an abrupt heteroepitaxial interface. Examples illustrating functional oxides that can be grown epitaxially on silicon in a low temperature/excess oxidant regime will be presented, as well as functional epitaxial oxide/silicon heterostructures that make use of the integration of epitaxial oxide layers with silicon.

2:45 PM
Interfacial Structure and Properties of Nanoscale Ferroelectrics: *Xiaoqing Pan*[1]; Haiping Sun[1]; Wei Tian[1]; Jeffrey Haeni[2]; Darrell G. Schlom[2]; [1]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16803-6602 USA

In this talk, we will present our recent work on the interfacial structure and properties of nanoscale ferroelectrics. Well-controlled ferroelectric thin films and heterostrucures were grown by reactive molecular beam epitaxy (MBE). The strain relaxation and misfit dislocation formation mechanisms of epitaxial BaTiO3 on (001) SrTiO3 substrates were studied using transmission electron microscopy (TEM) techniques. In-situ TEM studies showed that the threading dislocations climb toward the film/substrate interface during annealing at hight temperatures and form extended misfit dislocations to relax the strain in the epitaxial ultra-thin film. Quantitative high-resolution TEM was employed to examine the atomic positions of cations and anions in the coherent BaTiO3/SrTiO3 heterostructures. It was found that the relative static displacement of cations to anions is much greater than that of bulk BaTiO3, indicating the strain-induced elevation of spontaneous polarization in BaTiO3 thin films that may responsible for the theoretical prediction of giant dielectric constants for layered ferroelectric structures.

3:30 PM
Atomic Layer Controlled Growth of Epitaxial Magnetic Oxide Heterostructures by PLD with In Situ High Pressure RHEED: *Chang-Beom Eom*[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., 1500 Engineering Dr., Madison, WI 53706 USA

Complex oxide materials possess an enormous range of electrical, optical, and magnetic properties. For instance, insulators, high quality metals, dielectrics, ferroelectrics, semiconductors, ferromagnetics, colossal magnetoresistance materials, superconductors, and nonlinear optic materials have all been produced using oxide materials. A major challenge is the atomic layer controlled heteroepitaxial growth of various complex oxide materials so that these properties can be fully utilized in novel devices. We have grown SrRuO3-SrTiO3-SrRuO3 epitaxial ferromagnetic oxide heterostructures on TiO2- terminated (001) SrTiO3 substrates using pulsed laser deposition including in-situ high pressure RHEED. SrRuO3 is a negatively spin polarized ferromagentic oxide with a lattice parameter of 3.93≈ i.e., a lattice mismatch with (001) SrTiO3 substrates of 0.64%. Our RHEED intensity data and AFM images suggest that the SrRuO3 films on SrTiO3 substrate grow in the step-flow mode with a transition from 2-dimensional layer-by-layer mode into step-flow mode after covering one monolayer of SrRuO3. The origin of the growth mode transition can be attributed to a change in mobility of ad-atoms and switching of a surface termination layer from the substrate into the film. Transmission electron microscopy images of a cross-sectional SrRuO3-6 unit cell SrTiO3-SrRuO3 heterostructure show that the trilayer is single domain with atomically sharp interfaces between SrRuO3 and SrTiO3. Such an atomic layer control of the interfaces and barrier layers in ferromagnetic oxide (FM-I-FM) trilayer junctions allows for quantitative studies of spin-polarized transport across the ferromagnetic oxide (FM-I-FM) junction interfaces.

4:15 PM
Nano and Micro Elastic Domains in Constrained Ferroelectric Films: *Alexander L. Roytburd*[1]; [1]University of Maryland, Matls. & Nucl. Eng, College Park, MD 20742 USA

We present results of experimental and theoretical studies of 90∞ domains and their effect on electro-mechanical properties of PZT tetragonal films. Engineering of constraint is used to design different polydomain structures of elastic domains. Mechanical interaction between a FE film and a substrate is engineered through changing misfit between a FE film and different substrates, creating vicinal structures of the substrate surface, patterning FE films with formation of stripes and islands. Important role of elastic domains in switching of 180∞ FE domains as well as in relaxation of piezostress created by 180∞ domains is discussed. The mobility of elastic domains and their switching in nano-islands resulting in giant direct and converse piesoeffects are demonstrated. The possibility to obtain nanoscale domain structures in constrained films and bulk crystals is explored.

Surface Engineering in Materials Science - II: High Temperature Materials and Surface Alteration

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA; A. Agarwal, Plasma Processes, Inc., Huntsville, AL 25811-1558 USA; Narendra B. Dahotre, University of Tennessee-Knoxville, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; C. Suryanarayana, University of Central Florida, Mechanical, Materials & Aerospace Engineering, Orlando, FL 32816 USA

Wednesday PM — Room: 7A
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Y. Sohn, University of Central Florida, MMAE/AMPAC, Orlando, FL USA; P. Kuppusami, National Institute for Materials Science, High Temp. Matls. Grp., Tsukuba Science City, Ibaraki Japan

2:00 PM
Development of Oxidation Resistant Coatings Based on Gamma TiAl: *Antonis Zaroulias*[1]; Guosheng Shao[1]; Panos Tsakiropoulos[1]; [1]University of Surrey, Sch. of Eng. (H6), Mechl., Matls. & Aeros. Eng., Guildford, Surrey GU2 7XH England

The development of gamma TiAl base alloys has addressed their oxidation and mechanical properties. The selected alloying additions to gamma and gamma + a2 alloys do not result in continuous Al2O3 scale formation in air. An intermixed Al2O3/TiO2 scale continues to form in the more complex alloys, but the rate of growth of this scale has been reduced. Alloys with Cr additions at a level generally above 8 to 10 at% can form a two-phase (gamma + Laves phase) microstructure and are capable of continuous Al2O3 scale formation in air. Furthermore, the Ti-Al-TM tau phases form protective Al2O3 in air, with the best oxidation behaviour exhibited by TM = Cr. Thus, the Ti-Al-Cr system offers unique opportunities for the design of coatings that are mechanically compatible with the substrate and also oxidation resistant. The paper will report results of our experimental study of Ti-Al-Cr coatings applied by PVD on gamma base substrates. The coatings have been designed for oxidation resistance using thermodynamic databases and microstructure models developed at the University of Surrey. A distinct feature of our study is the deposition of amorphous coatings with high crystallisation temperatures. The design and processing of coatings will be presented. The microstructures and properties of the as deposited and nanocrystalline coatings will be discussed.

2:20 PM
Long-Term Oxidation Behavior of Aluminized CMSX-4 Superalloys: *Nan Mu*[1]; Yongho Sohn[1]; Irene L. Nava[2]; [1]University of Central Florida, Adv. Matls. Procg. & Analysis Ctr., PO Box 162455, 4000 Central Florida Blvd., Plando, FL 32816-2455 USA; [2]Solar Turbines, Inc., PO Box 85376, 2200 Pacific Hwy., San Diego, CA 92186-5376 USA

Thermally grown oxide (TGO) scale on aluminized CMSX-4 single crystal superalloy has been studied after long-term oxidation testing to examine the spallation behavior of TGO using photo-stimulated luminescence spectroscopy (PSLS) and microscopy. Twelve disk-shaped (2.54 cm dia. and 0.32 cm thick) specimens were oxidized at 788∫C (1450∫F), 871∫C (1600∫C), 954∫C (1750∫F) and 1010∫C (1850∫F) for 5,000, 7,500 and 10,000 hours. Spallation of TGO scale along the NiAl(B2) grain boundary ridges was observed for specimens exposed to higher temperature and longer time. In contrast, at the lowest temperature and the shortest time of oxidation, a significant amount of metastable gamma- and theta-alumina was observed by PSLS along with some spallation. This type of spallation did not occur along the grain boundary ridges, but within the grains themselves.

2:40 PM
Enhancing the Corrosion Resistance of AISI 304 Stainless Steel by Laser Surface Melting: *S.-H. Wang*[1]; J.-Y. Chen[1]; G. Campbell[1]; L. Xue[1]; [1]National Research Council of Canada, Integrated Mfg. Tech. Inst., 800 Collip Cir., London, Ontario N6G 4X8 Canada

AISI 304 stainless steel is widely used in industry for numerous applications requiring corrosion resistance. Despite its good corrosion resistance, the steel is prone to sensitization by heating, which significantly reduces its resistance to localized corrosion, especially intergranular corrosion. In this study, a thin layer of sensitized AISI 304 stainless steel surface was melted using a CW CO2 laser, to study the effect of laser surface melting on its corrosion resistance. Laser surface melting produced a thin protective surface layer with extremely fine microstructure, homogeneous distribution of alloying elements and clean microstructure boundaries, which contributes to the desensitization of the stainless steel and enhances its resistance to pitting and intergranular corrosion as well as general corrosion. The laser-melted layer also showed resistance to the sensitization and deterioration of the enhanced corrosion resistance when subjected to post heating.

3:00 PM Break

3:20 PM
Optical Emission Spectroscopy Correlations to Plasma Nitriding Surface Treatments for Process Optimization and Control: R. B. Young[1]; S. Patankar[1]; K. A. Prisbrey[1]; *F. H. (Sam) Froes*[1]; [1]University of Idaho, Inst. of Matls. & Adv. Processes, Col. of Eng., Matls. Sci. & Eng. Dept., Moscow, ID 83844-3024 USA

Optical emission spectroscopy is inexpensive, and gives reliable information about chemical states and operating parameters during plasma nitriding surface treatments of titanium, tool steels, and stainless steels. The objective was to correlate plasma nitriding variables, optical emission spectra, material property improvement, and metallographic, SEM, and XPS surface characterization. The procedure was to measure these variables during and after nitriding in inductively coupled radio frequency plasma. Plasma variables included plasma power, electron temperature, ion density, pressure, gas flow, and others. Pattern recognition algorithms coupled with ab initio molecular modeling calculations helped identify key wavelengths in optical emission spectra. Neural networks and fuzzy logic methods allowed implementation of practical control methods and online optimization of process parameters. Our results included correlations of plasma state parameters with photon emissions, and correlations of nitriding performance with process parameters. These results yielded methods for better process optimization, design, and control.

3:40 PM Moved to Materials Lifetime Sciences Symposium
Physically-Based Models for the Prediction of Fatigue and Dwell Fatigue Crack Growth in Ti-6242: Comparison of Models with Experiments: F. McBagonluri[2]; Chris Mercer[1]; E. Akpan[1]; W. Shen[1]; *W. O. Soboyejo*[1]; [1]Princeton University, Princeton Matls. Inst., D404 E. Quad., Princeton, NJ 08544 USA; [2]University of Dayton, Dept. of Matls. Eng., Dayton, OH 45390 USA

4:00 PM
Influence of Ta Content on Microstrucure and Mechanical Properties of Ir-Ta as Diffusion Barrier Coatings on Nickel Base Single Crystal Superalloy TMS-75: *Parasuraman Kuppusami*[1]; Hideyuki Murakami[2]; Takahito Ohmura[3]; [1]National Institute for Materials Science, High Temp. Matls. Grp., High Temp. Matls. 21 Proj., 1-2-1, Sengen, Tsukuba Science City, Ibaraki 305-0047 Japan; [2]The University of Tokyo, Dept. of Matls. Eng., Sch. of Eng., 7-3-1, Hongo, Bukyo-ku, Tokyo 113-8656 Japan; [3]National Institute for Materials Science, Steel Rsrch. Ctr., 1-2-1, Sengen, Tsukuba Science City, Ibaraki 305-0047 Japan

Ir- Ta alloy coatings have been proposed as a novel diffusion barrier material on nickel base single crystal superalloys owing to their high yield strength at high temperatures. This investigation has been undertaken to generate the database of mechanical and oxidation resistance properties of Ir-Ta alloy coatings with varying Ta contents. DC magnetron sputter deposition of Ir-Ta coatings from a composite target of Ir-Ta was carried out at 573 K on <100> oriented Ni- base single crystal superalloy TMS-75 substrates. The influence of tantalum contents of 16.5, 23.9, 39.8 and 65.1at % in Ir-Ta coatings on the microstructure and mechanical properties has been investigated. Nanohardness and microhardness measurements indicated that addition of Ta alters the mechanical properties such as hardness and Youngís modulus significantly. Further, a comparative study on the thermal cyclic oxidation resistance of the Ir-Ta modified aluminide coatings is presented.

4:20 PM
Sulfidation Behavior of Fe20Cr Alloys: *Lalgudi V. Ramanathan*[1]; Marina F. Pillis[1]; [1]Instituto de Pesquisas Energeticas e Nucleares (IPEN), Matls. Sci. & Tech. Ctr., Av. Prof. Lineu Prestes 2242, Cidade Universitaria, Sao Paulo 05508-000 Brazil

High temperature corrosion of structural alloys in sulfur bearing environments is many orders of magnitude higher than in oxidizing environments. Increase in oxidation resistance of various alloys with addition of reactive elements is well known. The effect of yttrium on sulfidation behavior of Fe-Cr alloys is still inconclusive. This paper reports the effect of yttrium on sulfidation behavior of Fe-20Cr alloys in H2-2%H2S environment at 700∞C. In the presence of Y, the sulfidation rate of the alloy decreased by an order of magnitude, the sulfide grain size was smaller and the sulfide growth mechanism predominantly anionic, after an initial cation diffusion controlled process. The sulfide layer on the FeCrY alloy was significantly thinner than that formed on the FeCr alloy. The alloy/sulfide interface on the FeCr alloy was planar where as that on the FeCrY alloy was undulated, with large regions of internal sulfidation.

The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research: Electronic, Magnetic, Optical, Piezoelectric Materials, Surface Treatment

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Japan Institute of Metals, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
Program Organizers: Julia R. Weertman, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Katherine T. Faber, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Morris E. Fine, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Wayne King, Lawrence Livermore National Laboratory, San Ramon, CA 94583-2496 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Ben Mori, Tokyo 168-0081 Japan

Wednesday PM Room: 9
March 5, 2003 Location: San Diego Convention Center

Session Chairs: A. K. Vasudevan, Office of Naval Research, Matls. Sci. Div., Arlington, VA 22217 USA; P. W. Voorhees, Northwestern University, Matls. Sci. & Eng. Dept., Evanston, IL 60208 USA

2:00 PM Invited
Relation Between Stress-Induced Voiding and Film Texture in Cu Thin Films: *Junichi Koike*[1]; Atsuko Sekiguchi[1]; Makoto Wada[1]; Kouichi Maruyama[1]; [1]Tohoku University, Dept. of Matls. Sci., 02 Aobayama, Aoba-ku, Sendai, Miyagi 980-8579 Japan

Cu thin films are new materials for interconnect lines in advanced integrated circuits. Because of thermal expansion misfit between Cu and its surroundings, thermal stress arises and causes stress-induced voiding that leads to device failure. Since the elastic property of Cu is highly anisotropic, voiding tendency is expected to depend on local crystallographic texture. We investigated the relationship between the voiding tendency and the film texture using transmission and scanning electron microscopy and electron back scattering analysis in conjunction with finite element calculation of stress distribution. We found that voids were formed at interfaces and corners of incoherent twins under a large stress concentration in a (111) oriented films. By changing texture to a random or a (100) oriented texture, stress-induced voiding could be avoided because of the absence of the incoherent twins. Possible ways to control the film texture and the voiding tendency will also be presented.

2:30 PM
TEM Observation of Phase Transformation and Magnetic Structure in Ferromagnetic Shape Memory Alloys: *Koichi Tsuchiya*[1]; [1]Toyohashi University of Technology, Production Sys. Eng., Tempakucho Hibarigaoka 1-1, Toyohashi, Aichi 441-8580 Japan

Ni-Mn-Ga ferromagnetic shape memory alloys have been attracting much attention as a new class of magnetic actuator materials. It was reported that in Ni-Mn-Ga strain up to 5% can be obtained by magnetically-induced twinning in the martensite phase. This report describes the results of in-situ TEM observations of martensitic transformation and preceding intermediate phase transformation. It was revealed that the intermediate phase is characterized by the formation of satellite spots at 1/6g220 positions, which suggests the formation of micro domains accompanied by the modulation of a {110}<1-10> type with 6 layes period. Transformation from the intermediate phase to the martensite phase was continuous. The Lorentz microscopy was also used to investigate the magnetic domain structures in the martensite and its relation to the martensite microstructures such as the variant and microtwins. It was revealed that microtwins form the magnetic domains and the twin boundaries have a character of 90∞ walls.

2:50 PM

Mapping of Magnetization Patterns Through Phase Reconstructed Lorentz Microscopy: *Marc De Graef*[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

Lorentz microscopy has for many years been a qualitative observation technique, providing images of magnetic domain walls (through the out-of-focus or Fresnel mode) and magnetic domains (through the Dark Field or Foucault mode). Recently, it has become possible to use the out-of-focus Fresnel images to compute the phase of the electron wave. From this phase, the magnetization pattern can be computed by a simple gradient operation. The method relies on the so-called Transport-of-Intensity Equation (TIE), which relates the spatial variation of the phase to the longitudinal derivative of the image intensity. Simple and fast solution algorithms are now available to solve the equation almost in real-time. In this presentation we will describe the foundations of the method, and provide several illustrations using the following material systems: patterned Permalloy islands, ferromagnetic shape memory alloys, and hard magnetic materials.

3:10 PM

Electron Microscopy of Combined Reaction Processed L10 FePd Based Ferromagnets: *Amal A. Al-Ghaferi*[1]; *Anirudha R. Deshpande*[1]; Jorg M. Wiezorek[2]; Huiping Xu[1]; [1]University of Pittsburgh, MSE, 864, Benedum Hall, Pittsburgh, PA 15213 USA; [2]University of Pittsburgh, MSE, 848, BEH, Pittsburgh, PA 15213 USA

The Extraordinary magnetic properties in FePd based (L10) intermetallic alloys depend strongly on the microstructure and defect structure. The tetragonal ordered L10 phase forms from an FCC (Fe,Pd) solid solution via a first order transformation between 650-790°C (Tc) depending on Pd content. In conventionally processed FePd alloys dodecahedrally conjugated polytwin morphologies develop with submicron dimensions. The presence of polytwins is detrimental for the technical magnetic properties resulting in considerably lower MR and HC than theoretically predicted for (L10) FePd. Combined reaction processing exploits the competition between recrystallization and the ordering transition during annealing of cold worked Fe-Pd alloys and enables microstructural control to taylor technical properties. Combined reaction processing has been used to produce FePd alloys with fourfold increased coercivity with respect to the conventionally processed material. Here SEM and TEM are used to identify the microstructural origins of the enhanced magnetic properties and study the microstructures of combined reaction processed FePd permanent magnet alloys. Support from NSF (DMR-0094213) with Dr. Murthy as program manager is acknowledged.

3:30 PM Invited

Electrolytic Graining of Aluminum Lithographic Printing Plates: *Chao-Sung Lin*[1]; Gee-Chen Chiou[1]; [1]Da-Yeh University, Dept. of Mechl. Eng., 112 Shan-jiau Rd., Da-Tsuen, Chang-hwa 51505 Taiwan

AA1050 Al lithographic plates were electrograined in nitric and hydrochloric acids using various ac current waveforms. Detailed etch pit morphology and etch film microstructure were characterized by cross-sectional transmission electron microscopy (TEM). After electrograining, an etch film deposited on the Al surface and masked its real topography, which exhibited a pitted surface after removing the etch film. The etch film morphology strongly depended on the electrolyte composition, and the frequency and waveform of the ac current. For example, the etch film formed in nitric acid exhibited a layered structure, whereas that formed in hydrochloric acid was a single layer. Moreover, the layer thickness of the etch film decreased with increasing frequency and anodic/cathodic charge ratio of the ac current, indicating that the etch film formed during the cathodic half cycle. The layered etch film thus developed when the etch film overlaying the aluminum surface underwent dissolution/peeling during successive cycles.

4:00 PM

The Role of Analytical Electron Imaging in Semiconductor Device Development and Manufacturing: *Jerzy Gazda*[1]; Peter Liu[1]; [1]Advanced Micro Devices Corporation, Process Characterization & Analytl. Lab., Austin Wafer Fab Div., MS-613, 5204 E. Ben White Blvd., Austin, TX 78741 USA

In this overview we will show examples of the three most important roles of electron and ion microscopy to semiconductor device manufacturing. We will illustrate the current role of SEM tools in Fab manufacturing process verifications, concentrating on discussion of challenges faced by this technique when used for inspections of 65 nm-node and smaller devices. Secondly, we will illustrate the role of focused ion beam (FIB) tools in defect inspection and specimen preparation for SEM and TEM. Finally, we will discuss the currently occurring changes affecting the role of TEM-based techniques as they move from predominantly defect inspection to a larger role in thin film metrology.

4:20 PM

Field-Driven In-Situ TEM Observations of Domain Boundary Cracking in Relaxor-Based Piezoelectric Crystals: *J. K. Shang*[1]; X. Tan[1]; Z. Xu[2]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA; [2]City University of Hong Kong, Dept. of Physics & Matls. Sci., Tat Chee Ave., Kowloon, Hong Kong China

Relaxor-based piezoelectric crystals have recently received much attention because of their unusually large piezoelectric strains. However, these crystals are highly susceptible to field-induced cracking after repeated electric cycling.In this study, micromechanisms of electric-field induced cracking were examined in relaxor-based piezoelectric crystals by in-situ transmission electron microscopy and in-situ polarized optical microscopy. Field-induced cracking of domain boundaries was observed as incompatible piezoelectric deformations developed in the domains of different polarizations. Under repeated bipolar electric cycling, domain colonies developed near the crack tip and electric fatigue crack grew along the colony boundaries.

4:40 PM

Observation of GeO2 Redistribution in the Oxidized Poly-Si1-xGex Films by Electron Beam Irradiation: *Han-Byul Kang*[1]; Jun-Ho Lee[1]; Cheol-Woong Yang[1]; [1]Sungkyunkwan University, Sch. of Metallurgl. & Matls. Eng., 300 Chunchun-dong, Jangan-Gu, Suwon, Kyunggi-Do 440-746 Korea

We have observed the irradiation effect in oxidized poly-Si1-xGex films under electron induced in the Transmission Electron Microscope. Poly-Si1-xGex films with various compositions and about 1000≈ in thickness were deposited on Si wafers. The oxidation were carried out in a conventional tube furnace at 800°. Cross sectional samples of oxidized poly-Si1-xGex films were irradiated with LaB6 cathode operated in the TEM. In order to analyze the redistribution of elements, we used the Energy Dispersive X-ray Spectroscopy. The amount of current densities measured by GATAN slow scan CCD camera. After irradiation, we can know that the changing of the GeO2 distribution in the oxide layer. For the oxidized poly-Si0.6Ge0.4 films, the accumulation of GeO2 was observed at the surface. And, for the oxidized poly-Si0.4Ge0.6 films, the crystallization of GeO2 was occurred in the oxide layer. Ge lattice fringes and twinning were observed in the oxide layer.

5:00 PM

Electron Backscatter Diffraction for Studies of Localized Deformation: *R. H. Geiss*[1]; R. R. Keller[1]; [1]NIST, Matls. Reliability Div., Boulder, CO 80305 USA

Electron backscatter diffraction (EBSD) was used to study localized deformation in constrained volume materials. We will present a study of deformation in narrow aluminum interconnects after low frequency, high current density AC cycling. Joule heating and differential thermal expansion caused cyclic thermal straining, resulting in thermomechanical fatigue. By quasi in situ AC testing, we determined the evolution of the crystallography of all grains and boundaries in the lines, allowing us to formulate a mechanistic understanding of the deformation process, including slip line formation and grain growth. In a second study, we present an analysis of diffraction patterns from selectively oxidized, multilayered AlGaAs/GaAs structures. Elastic strains accompanying AlGaAs oxidation were characterized by EBSD. Image quality maps revealed the resulting strain field about oxide growth fronts which compared with FE simulations. Quantitative strain measurements were made comparing diffraction band width measurements of processed images.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Alloys Properties

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday PM Room: Solana
March 5, 2003 Location: San Diego Marriott Hotel

Session Chairs: Shigeatsu Nakazawa, Tohoku University, Dept. of Metall., Sendai 980-8579 Japan; Adolf Mikula, Institute of Iorganic Chemistry, Waehringerstrasse 42, Vienna A-1090 Austria

2:00 PM Keynote
ZincñA Possible Component in Lead-Free Solders: *Adolf Mikula*[1]; [1]Institute of Iorganic Chemistry, Waehringerstrasse 42, Vienna A-1090 Austria

Lead-tin solders are commonly used in electronic packaging due to their unique combination of electrical, chemical, physical, thermal and mechanical properties. Since lead and lead containing alloys cause great environmental concern and health hazards it is necessary to replace lead in solder materials. One of the main driving forces to eliminate lead in solder joints is the fact that the disposal of great amount of electronic equipment in landfills can cause lead to leach out and contain the underground water and subsequently find its way into the human body. Besides environmental concerns also new technological developments require new solders with a variety of improved or new properties. The miniaturisation of electronic devices need smaller and smaller interconnections and a reliable joining must be achieved with smaller volumes of solder materials. In most cases these new materials will be ternary tin or indium alloys. The second element could be Bi, Cu, Ag, and Au and as third elements Zn, Ni, Sb, Al and P must be considered. All these elements have an influenze on certain properties. Small amount of zinc will improve in most cases the mechanical properties and the strength of such solders. We investigated the thermodynamic properties and the oxidation behavior of several ternary zinc systems: Al-Sn-Zn, Cu-In-Zn, Ag-Sn-Zn and In-Sn-Zn.

2:35 PM
Activity Measurement of the Constituents in Molten Sn-Ag-In and Sn-Zn-Mg Ternary Lead Free Solder Alloys by Mass Spectrometry: *Takahiro Miki*[1]; Naotaka Ogawa[2]; Tetsuya Nagasaka[1]; Mitsutaka Hino[1]; [1]Tohoku University, Dept. of Metall., Grad. Sch. of Eng., Aoba-yama 02, Aoba-ku, Sendai 980-8579 Japan; [2]Tohoku University (Now at NKK Corporation), Sendai 980-8579 Japan

The increase of demand for smaller and lighter portable electronic devices has made interconnecting densities and packaging technologies more important. Soldering material widely used is Sn-Pb alloy, which have low melting point and excellent electrical, strength properties and wettability. However, Pb is one of the toxic elements, which is undesirable due to environmental and safety reasons, thus Pb-free alternative alloy is preferred for new soldering material. Sn-Ag and Sn-Zn based alloys were viewed as very promising candidates, among many potential substitutes. Addition of third element to these alloys will decrease the melting point of the alloy. Hence, Sn-Ag-In and Sn-Zn-Mg alloys are expected to be suitable for replacing Sn-Pb solder alloy. In order to design new Pb-free soldering materials, precise understanding of thermodynamic properties and phase diagrams for alloy systems are crucial. In the present work, ion current ratios of Ag to In and Mg to Zn were measured for Sn-Ag-In and Sn-Zn-Mg alloy by mass spectrometry, respectively. Also, the authors reviewed the thermodynamic properties of terminal binary alloys determined by other researchers and evaluated a thermodynamic function to express the excess Gibbs free energy of each binary alloy. Thermodynamic function to express the excess Gibbs free energy of liquid Sn-Ag-In and Sn-Zn-Mg ternary alloy were determined, utilizing the assessed Gibbs free energy of terminal binary alloys with the measured ion current ratios using mass spectrometer.

3:00 PM
Effect of Additives on Viscosity of Molten Nickel Base Alloys: *Yuzuru Sato*[1]; Koji Sugisawa[2]; Daisuke Aoki[2]; Masayoshi Hoshi[2]; Jong-Il Kim[2]; Tsutomu Yamamura[2]; [1]Tohoku University, Dept. of Metall., Aramaki aza Aoba, Sendai 980-8579 Japan; [2]Inha Technical College, Dept. of Metallurgl. Eng., Inchon 402-752 Korea

Viscosity has been measured for various binary alloys of nickel with iron, cobalt and chromium to clarify the behavior of viscous flow of molten nickel alloys. Entire concentration range for iron and cobalt, and 0-60at% for chromium were studied. Method for the measurement was an oscillating cup viscometry using an alumina crucible that was precisely machined. Temperature was up to about 1880K for all the measurements. A furnace used was carefully controlled to keep best temperature uniformity that was within 0.5K for entire length of the crucible to prevent the convection in the melt. All the results represented good Arrhenius relationship although some measurements included overcooled temperatures. The isothermal viscosities increased monotonously with an addition of iron and chromium into nickel. On the other hand, the addition of cobalt showed slight decrease, and then viscosity increases in the progress of addition of cobalt.

3:25 PM
Microstructure Analysis of ZA Alloy Rod Directionally Solidified by Heated Mold Continuous Casting: *Ying Ma*[1]; Yuan Hao[1]; [1]Gansu University of Technology, Matls. Sci. & Eng., 85 Langongping Rd., Lanzhou, Gansu 730050 China

The as-cast and heat treatment microstructure of ZA alloy rod directionally solidified by continuous casting has been analyzed. The results show that the microstructure of the ZA alloy lines is the parallel directional dendritic columnar crystal. Every dendritic crystal of eutectic alloy ZA5 is composed of many layer eutectic , and Á phase. The microstructure of hyper eutectic ZA alloys is primary dendritic crystal and interdendritic eutectic structure. The primary phase of ZA8 and ZA12 is , phase, but the primary phase of ZA22 and ZA27 is · phase.

3:50 PM Break

4:10 PM Invited
Activities of Bi and In in the Bi-In Liquid Alloy Measured by Using Vacuum-Sealed Quartz Cell/Atomic Absorption Spectrophotometer Combination: *Shigeatsu Nakazawa*[1]; Minoru Sunada[2]; Takeshi Azakami[3]; Tetsuya Nagasaka[1]; [1]Tohoku University, Dept. of Metall., Grad. Sch. of Eng., Aoba-yama 02, Sendai 980-8579 Japan; [2]Sunada Kogyo Company, Ltd., Kosugi 406, Tonami 939-1357 Japan; [3]Saitama Institute of Technology, Dept. of Matls. Sci. & Eng., Grad. Sch. of Eng., 1690 Fusaiji, Okabe-machi, Osatogun 369-0293 Japan

Activities of Bi and In in the Bi-In liquid alloy were measured over the whole range of composition at the temperature from 850K to 1050K. An alloy was vacuum-sealed in a quartz cell and heated at the temperature of interest. The absorption for 307nm radiation from Bi lamp was measured for Bi atom vapor in the cell. By heating a pure metal as a standard and measuring the absorbance as a function of the temperature, an analytical curve for Bi atom vapor was constructed and used for conversion of the absorbance to the vapor density. Bi activity was determined as the ratio of the Bi atom vapor density over the alloy to that over a pure metal. The same procedure was applied to In vapor and In activity was determined independently of Bi. Results were used to reevaluate the thermodynamics of the Bi-In system.

4:40 PM
Thermodynamic Properties of Several Ternary Zinc Alloys: Adolf Mikula[1]; *Sabine Knott*[1]; [1]University of Vienna, Inst. f¸r Anorganische Chemie, W‰hringerstrasse 42, Vienna A-1090 Austria

In this presentation I would like to show the importance of good, reliable and consistent thermodynamic data for industry and for research. The thermodynamic properties of several ternary liquid zinc alloys were determined using emf and calorimetric measurements. The Au-Sn-Zn, Ag-Sn-Zn, Cu-In-Zn and Al-Sn-Zn alloys were investigated because they are possible candidates for new lead-free solder materials. In these systems the thermodynamic data can be used to improve phase diagram calculations and will be helpful for some theoretical models to improve the calculation of physical or mechanical properties. In the Au-Sb-Zn, Ag-Sb-Zn and Cu-Sb-Zn the temperature dependence of the thermodynamic data was used to look for the existence of Chemical Short Range Order (CSRO) in the melt of these alloys.

5:05 PM
Defect Formation and Mechanism of ZA Alloy Made in Continuous Casting by Heated Mold: *Ying Ma*[1]; Feng-yun Yan[1]; Hong-jun Liu[2]; [1]Gansu University of Technology, Sch. of Matls. Sci. & Eng., Lanzhou 730050 China; [2]Huazhong University of Science and Technology, Sch. of Matls. Sci. & Eng., Wuhan 430074 China

The continuous directional solidification technique of five kinds of special ZA alloys with eutectic, peritectic and eutectoid transformation under the condition of continuous casting by heated mold was studies. The mechanism of surface defects appearing in ZA alloy line was discussed and the structure of some defects were analyzed by SEM. The results show that only when the fitting of various technique factors, pressure head, outlet temperature, pulling speed and cooling condition is reasonable under a certain range, can the ZA alloy smooth line be continuously pulled out. Unreasonable technique will result in hot tear, rough surface, mush outlet, leaking and other defects. The shape and location of solid-liquid zone have the importance influence on the forming of above defects. When the solid-liquid zone is located in the mould outlet or inner mould, the surface of solidification will protrude into the inner mould. If the left liquid cannot counteract the solidification contract, the rough surface will appear. If the solidification surface further into the mould, the ingot in mould will have a big friction force when pulling and form hot tear. When the solid-liquid zone is moved out of mould, the solidification surface will turn into a plane, which made it easy to form leaking. The vibration or unstable operation will make the ingot winding during pulling.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Materials Processing Technologies: General II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday PM — Room: Point Loma
March 5, 2003 — Location: San Diego Marriott Hotel

Session Chair: Thomas P. Battle, Dupont, Edge Moor, DE 19809 USA

2:00 PM Invited
Development of Closed Recycling Process for Low Grade Scrap of Al-Based Composite Materials: *Hiroyuki Sano*[1]; Shinichi Kato[2]; Tatsuya Motomura[2]; Toshiharu Fujisawa[1]; [1]Nagoya University, Rsrch. Ctr. for Adv. Waste & Emission Mgt., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Dept. of Matls. Sci. & Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

Closed recycling process for low grade scrap of Al-based composite materials was developed. Flux treatment with water soluble halide is a key technique in this process. In the present work, flux treatment conditions were discussed from the viewpoint of separation and recovery of matrix, reinforce and flux materials. Optimum separation condition was obtained for the NaCl-KCl-KF flux treatment. Recovered aluminum alloy has enough quality for using various materials. Used flux is recyclable for the process by adding a small amount of KF.

2:35 PM
Two-Way Shape Memory Effect of Ferromagnetic Ni-Mn-Ga Sputter-Deposited Films: *Makoto Ohtsuka*[1]; Minoru Matsumoto[1]; Kimio Itagaki[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 2-1-1, Katahira, Aoba-ku, Sendai 980-8577 Japan

The ternary intermetallic compound Ni_2MnGa is an intelligent material, which has a shape memory effect (SME) and a ferromagnetic property. Use of shape memory alloy films for an actuator of micromachines is very attractive because of its large recovery force. The Ni-Mn-Ga films with a thickness of nearly 5 μm were deposited with a radio-frequency magnetron sputtering apparatus. They were heat-treated at 1073 K for 36 ks for homogenization and ordering. The purpose of the present study is to investigate the effects of fabrication conditions on the properties of the films such as chemical composition, crystal structure, microstructure and transformation temperature. Furthermore the effect of plastic deformation and of constraint aging on SME is also investigated. The two-way SME was shown after plastic deformation. The intensity of X-ray diffraction peaks from stress-induced martensitic phase was found. The constraint-aged films also showed the two-way SME by thermal cycling.

3:00 PM
Fabrication of BaNd2Ti4O12/Bi4Ti3O12/BaNd2Ti4O12 Laminated Ceramics by Spark Plasma Sintering: *Yong Jun Wu*[1]; Naofumi Uekawa[1]; Kazuyuki Kakegawa[1]; [1]Chiba University, Ctr. for Frontier Elect. & Photonics, Dept. of Matl. Tech., Chiba 263-8522 Japan

Laminated ceramics have received wide scientific and commercial attention because its properties, such as mechanical properties and electrical properties, can be tailored by adjusting the structure, thickness and composition of the different layers. For example, ferroelectric Bi4Ti3O12 (BIT) which has a positive temperature coefficient of dielectric constant (te) can be used to modify the negative te of typical microwave dielectric ceramic BaNd2Ti4O12 (BNT). However, BaNd2Ti4O12/Bi4Ti3O12/BaNd2Ti4O12 (BNT/BIT/BNT) laminated ceramics could not be prepared by the general method because the optimal sintering temperature of BaNd2Ti4O12 ceramic (around 1350°C) was much higher than that of Bi4Ti3O12 ceramic (around 1100°C). In this research, a new method of spark plasma sintering (SPS) combined with post-sintering was successfully proposed to prepare BNT/BIT/BNT laminated ceramics. It was divided into three steps. In first step, the BaNd2Ti4O12 ceramics were prepared by conventional sintering method. In second step, the calcined and pre-pressed Bi4Ti3O12 powders were sandwiched between BaNd2Ti4O12 ceramic pellets and spark plasma sintered at 900°C for 10min to synthesize BNT/BIT/BNT composite ceramics. In last step, a post-sintering was employed to re-oxidize the partially reduced BNT/BIT/BNT composite ceramics. The results of scanning electron microscopy (SEM) and electron probe micro-analysis (EPMA) showed that BNT layer and BIT layer were well bonded and no significant interfacial infiltration between them was observed. The sandwiched BNT/BIT/BNT ceramic with 10.7% BIT in volume has a dielectric constant of 92.8, a low dielectric loss of 0.0068 and a small temperature coefficient of dielectric constant of 35 ppm°C at 1 MHz.

3:25 PM
Preparation of Infrared Materials from Ultrafine Quartz Powder: *Huaming Yang*[1]; Jianhong Chao[1]; Weiqin Ao[1]; Guanzhou Qiu[1]; [1]Central South University, Dept. of Minl. Eng., Changsha 410083 China

Infrared material is a special material widely used in military, biological engineering and chemical industry, but the problems, such as unstable property, high cost and higher thermal expansion coefficient, seriously restrict the development and wide application of traditional infrared materials with SiC or ZrSiO4 matrix. Ultrafine quartz powder was used to prepare infrared material by Hot-pressing technology. The aim of the paper is to investigate the thermal expansivity and effects of SiO2 size, addition of Al2O3 or Fe2O3 on infrared emissivity of quartz matrix infrared material(QMIM) in 8-25μm wavelength range. The results indicate that 89% of infrared emissivity and 0.4◊10-5/ of thermal expansion coefficient of QMIM can be obtained. Affecting mechanism of Al2O3 and Fe2O3 on infrared emissivity of QMIM were also discussed.

3:50 PM Break

4:10 PM
Deposition of Multilayered Titanium Thin Film by Nd: YAG Laser Ablation: *Takahiro Nakamura*[1]; Hideyuki Takahashi[2]; Katsutoshi Yamamoto[3]; Nobuaki Sato[3]; Atsushi Muramatsu[3]; Eiichiro Matsubara[2]; [1]Tohoku University, Dept. of Matl. Sci., Katahira 2-1-1, Aoba-ku, Sendai 980-8577 Japan; [2]Tohoku University, Inst. for Matl. Rsrch., Katahira 2-1-1, Aobaku, Sendai 980-8577 Japan; [3]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Katahira 2-1-1, Aobaku, Sendai 980-8577 Japan

The thin film of TiO2 by means of laser ablation has the nanostructure and would appear the new properties. Multilayered TiO2/Ti thin film on quartz substrate was synthesized by laser ablation of Ti and TiO2 targets using Nd: YAG pulse laser under the established conditions. Namely, Ti and TiO2 film was successively deposited on the substrates by the irradiation using laser light of the energy 500 and 400 mJ and the repetition rate 50 Hz for 15 and 6 sec with Ti and TiO2 target, respectively, in a high vacuum reaction chamber under the oxygen pressure of 10-8 Torr. Thicknesses of Ti and TiO2 in as-prepared film are 150 and 210 nm with growth rates of about 10 and 35 nm/sec, respectively. Preparation of TiS2/TiO2/Ti multilayered film was also examined in the presence of oxygen and H2S (or CS2) with Ti and/or TiO2 target.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Aqueous and Electrochemical Processing V

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday PM	Room: Pacific
March 5, 2003	Location: San Diego Marriott Hotel

Session Chairs: Paulo von Kr,ger, Federal University of Ouro Preto, Minas Gerais Brazil; U. B. Pal, Boston University, Dept. of Mfg. Eng., Boston, MA 02446 USA

2:00 PM Invited
Zirconia-Based Inert Anodes for Green Synthesis of Metals and Alloys: C. P. Manning[1]; A. Krishnan[1]; *U. B. Pal*[1]; [1]Boston University, Dept. of Mfg. Eng., Boston, MA 02446 USA

The research work demonstrates the technical viability of employing zirconia-based inert anodes for environmentally sound and cost-effective production of metals such as magnesium, tantalum, aluminum, etc., directly from their oxides. The inert anode consists of the oxygen-ion-conducting stabilized zirconia membrane in intimate contact on one side with a catalytically active electronic phase. The opposite (other) side of the zirconia membrane is placed in contact with an ionically conducting solvent phase containing the oxides of the desired metals. An inert cathode is placed in the solvent and an appropriate electric potential is applied between the electrodes to synthesize the metals from their oxides in the solvent. The full-benefit of the process can be realized if it is conducted at temperatures between 1200-1400∞C. At these temperatures the ohmic resistance drop across the stabilized zirconia membrane are low and therefore high current densities on the order of 1 A/cm2 or greater can be obtained. In addition, the process efficiency can be further increased by directly reforming hydrocarbon fuel over the anode. It should be noted that several attempts have been made earlier to employ this concept at temperatures below 1000∞C. However, these efforts have not been successful mainly because sufficiently high current densities could not be obtained through the zirconia membrane. This paper reports the recent progress of a continuing laboratory-scale investigation involving different types of zirconia-based inert anodes employed at temperatures between 1200-1400∞C. The topics covered include: stability of the zirconia membrane in the selected molten solvent (flux), volatility of the flux, potentiodynamic sweeps, electrolysis experiments, and analysis of the metals produced.

2:35 PM Invited
Hydrometallurgical Process for Recycle of Spent Nickel-Metal Hydride Secondary Battery: *Toshihiro Kuzuya*[1]; Takayuki Naitou[2]; Hiroyuki Sano[1]; Toshiharu Fujisawa[1]; [1]Nagoya University, Rsrch. Ctr. for Adv. Waste & Emission Mgmt., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan; [2]Nagoya University, Dept. of Matls. Sci. & Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

A hydrometallurgical process has been developed for recovery of metal values such as Co, Ni and rare earths from the electrode materials of spent nickel metal-hydride(Ni-MH) secondary battery. MmNi5 intermetallic compound could be separated from the electrode materials mixture by sedimentation. A typical chemical composition of MmNi5 was approximately, in mass%, 56.0%Ni, 13.4%Ce, 10.6%La and 7.9%Co. The time dependency of leaching intermetallic compound with sulfuric acid solution was examined with processing factors such as sulfuric acid concentration, temperature and agitation intensity. The leaching of rare earth metals proceeded very rapidly, reaching completion in less than 1.8ks. On the other hand, the slow leaching of nickel was observed. The percent of nickel leached at 328K could only reach about 70%.

3:05 PM
Rare Earth Separation and Recycling Process Using Rare Earth Chloride: *Testsuya Uda*[1]; Masahiro Hirasawa[2]; [1]California Institute of Technology, Matls. Sci., MC138-78, Pasadena, CA 91125 USA; [2]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls. (IMRAM), 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

We review our recent achievements of rare earth separation technique and new process idea for rare earth magnet recycling. (1) Binary chloride mixtures of rare earths were separated by new selective reduction-vacuum distillation process. According to our experimental results, apparent separation factors were 8.1 for Pr-Nd chloride mixture and 570 for Nd-Sm chloride mixture. These values are higher than conventional solvent extraction methods. (2) A study of recycling process of magnet sludge was carried out. The rare earths in the neodymium magnet sludge were extracted by chlorination with FeCl2. An activated carbon was used as a de-oxidation reagent. Metallic iron in the sludge was not chlorinated because the iron monochloride is not stable. The extracted rare earth chlorides were easily separated from Fe-alloy and the excess of FeCl2 by vacuum distillation. 96% of neodymium and 94% of dysprosium in the sludge were extracted into chloride phase. By the vacuum distillation, a mixture of neodymium and dysprosi um trichlorides of 99.2% purity was recovered. It was confirmed that the rare earth chlorides were converted to the corresponding oxides by a pyrohydrolysis reaction accompanied by gaseous HCl formation. The HCl gas can chlorinate metallic iron to FeCl2. Therefore, a new recycling process for rare earth magnet waste can be realized as a chlorine circulation type process. During the process, only carbon and water are consumed, and there are no toxic pollutants. Moreover, the obtained rare earth oxide can be directly used as raw material in the conventional oxide electrolysis process.

3:30 PM
A Novel Application of Electrorefining in a Membrane Cell to Reclaim Zinc from the Bottom Dross of Hot Dip Galvanization: *Levente Becze*[1]; Tam·s I. T˚r˚k[1]; [1]University of Miskolc, Dept. of Nonferrous Metall., Miskolc-Egyetemvaros, Miskolc 3515 Hungary

The zinc bottom dross is obtained as a by-product from hot dip galvanizing operations and contains at least 92% zinc, which is recycled mainly by pyrometallurgical processes. The overall costs of such a treatment, however, are often relatively high, and the very strict environmental protection regulations might also cause additional difficulties for the high temperature procedures in the future. Therefore, a novel hydo-eletrometallurgical process has been developed in our laboratory, which could reclaim zinc of high purity, while attempting to achieve low energy consumption and making allowances for all the environmental restrictions. This aqueous processing technique, based on electrolytic refining in a cell equipped with anion-exchange membrane to separate the anode and cathode compartments, allows the indirect purification of the anolyte. Zinc is dissolved from the anode along with some of the impurity elements, while the intermetallic compounds (Fe-Zn, Fe-Al-Zn) are accumulated in the anode slime.

3:55 PM Break

4:05 PM Invited
Fundamental and Applied Research on Tin Electrorefining, Employing Stainless Steel Cathodes: *Paulo von Kr,ger*[1]; Erivelto Luis de Souza[2]; [1]Federal University of Ouro Preto, Dept. of Met. Eng., Sch. of Mines, Minas Gerais Brazil; [2]FundaÁ„o Gorceix, Ouro Preto, Minas Gerais Brazil

This paper deals with the cell design and operation of a tin electrorefing 1:5 pilot plant. The purpose was the fitting of the basic parameters to be used in a full scale tankhouse, for a major Brazilian tin producer. The main difference was the use of permanent, stainless steel cathodes, instead of the conventional tin starting sheets. The results on both product quality and operating parameters are discussed.

4:40 PM
Separation of Elements in Stainless Steel by Electrorefining Process: *Toshihide Takenaka*[1]; Masahiro Kawakami[1]; Masao Kawaguchi[1]; [1]Toyohashi University of Technology, Dept. of Produc-

tion Sys. Eng., Hibarigaoka 1-1, Tempaku-cho, Toyohashi, Aichi-Prefecture 441-8580 Japan

Stainless steel is one of the major materials in a nuclear reactor. The management of radioactivated stainless steel will become an important subject in the near future. The half-life of radioactive elements of Fe and Cr are short in general, whereas those of Ni is very long. Therefore, the separation of the elements in stainless steel should be effective to reduce the management cost of the waste radioactivated stainless steel. In this study, the separation of the elements in stainless steel was investigated by an electrorefining technique in an aqueous solution. The electrochemical reactions of the elements in stainless steel were studied by voltammetry, and potentio-static electrolysis was carried out. A plate of austenitic stainless steel, SUS304, was used as an anode and a cathode. The anode was dissolved electrochemically with good anodic current efficiency in a chloride solution, while the anodic dissolution did not occur in a sulfate solution. The smaller the anodic overpotential was, the lower the Ni concentration in the solution was after electrolysis. The residue where Ni was concentrated was found on the anode. At the cathode, metallic electrodeposit was obtained by potentio-static electrolysis, and only a small amount of H2 gas was evolved simultaneously. The electrodeposit at the cathode consisted of Fe and Cr mainly, and the Ni content was less than 0.5wt% under the suitable condition. It is concluded that the electrorefining technique in a chloride solution can be applied for separation of the elements of stainless steel.

5:05 PM

Preparation of Fibrous Nickel Oxide Powder by Wet Chemical Precipitation Combined with Pyrolysis: *Zhang Chuanfu*[1]; Jing Zhan[1]; Xueyi Guo[1]; Masazumi Okido[2]; [1]Central South University, Col. of Metallurgl. Sci. & Eng., Yuelu Dist., Changsha, Hunan 410083 China; [2]Nagoya University, Ctr. Integrated Rsrch. in Sci. & Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

In this study, the process of wet chemical precipitation of nickel ions from the solution with oxalic acid followed with precursor pyrolysis has been employed to prepare the fibrous NiO powder. The influences of the pH value, reaction temperature, concentrations of reactants, the order of reagents adding and the addition of surfactant on the morphology of precursor were investigated for the wet chemical precipitation stage; and that of the morphology of precursor, temperature and time for decomposition, air flow, and the rate for heating on the morphology, particle size and specific surface area of the final obtained NiO particles during pyrolysis were addressed respectively. The results show that the fibrous NiO particles with 100~120 for axis-ratios and about 6.0 m2/g for specific surface area can be obtained under certain experimental conditions. Spherical nanometer NiO powder particles were obtained after further grinding of fibrous nickel oxide particles.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: Copper III

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday PM	Room: Santa Rosa
March 5, 2003	Location: San Diego Marriott Hotel

Session Chairs: Lauri Holappa, Helsinki University of Technology, Lab. of Metall. FIN-02015 HUT Finland; F. Kongoli, FLOGEN Technologies Inc., Montreal, Quebec Canada

2:00 PM

Effect of Oxygen Enrichment on the El Teniente Converter Productivity at Ilo Smelter, Per˙: *Jose Bengoa*[1]; Jose Palacios[2]; Mario Sanchez[3]; [1]Southern Peru Copper Company, Ilo Smelter, PO Box 35, Ilo Peru; [2]University of Atacama, Dept. Met. Eng., PO Box 240, Copiapo Chile; [3]University of Concepcion, Dept. Met. Eng., PO Box 53-C, ConcepciÛn Chile

Low metal prices and environmental restrictions are always a challenge for copper smelters operation and its productivity. Thus, in order to reduce production costs and control gas emissions, improvements of El Teniente Converter operation were made at Ilo Smelter Plant of Southern Peru Copper Co., Ilo, Peru, and the main objective was to increase its capacity for smelting copper concentrates without affecting normal operation. The present work corresponds to a recent study conducted during the normal industrial operation of El Teniente Converter in this plant and, it shows the effect of increasing air oxygen enrichment from 28 to 34% on its productivity. The results show an increasing of concentrate smelting rate from 800 to 1370 tpd and hence a notorious improving in copper production, and practically no effect on the refractory consumption. Additionally, the increment of the concentrate throughput increases SO2 concentration in the exhausting gas and the sulfuric acid production, showing the effort done by this operation in order to accomplish the environmental Peruvian regulation.

2:30 PM

Oxidation of Copper at Different Temperatures: *Gabriel Plascencia*[1]; Torstein Utigard[1]; [1]University of Toronto, Matls. Sci. & Eng., 184 College St., Toronto, Ontario M5S 3E4 Canada

Copper was oxidized in the temperature range from 300 to 1000∫C under different partial pressures of oxygen. In the range from 300 to 500∫C, copper oxidizes following the logarithmic rate law; while in the range from 600 to 1000∫C copper oxidizes following the parabolic rate law. Transition from logarithmic to parabolic growth occurs at 525∫C. X-ray diffraction was performed on oxide scale to account for the CuO/Cu2O ratio at different temperatures. Activation energies found in this work are in good agreement with those already reported.

2:55 PM

YCC ISASMELT ProjectñThe First Chinese ISASMELT Furnace: *Yun Li*[1]; Philip Arthur[2]; [1]Yunnan Copper Corporation, Wangjiaqiao, Western Hill Dist., Kunming, Yunnan 650102 China; [2]MIM Process Technologies, ISASMELT, Level 2, 87 Wickham Terrace, Brisbane, QLD 4000 Australia

In 1999 Yunnan Copper Corporation made a decision to carry out technical modification on the copper smelter and change the existing sinter plant/electric furnace process to ISASMELT/electric furnace slag cleaning. The ISASMELT furnace has been running smoothly since heatup in May 2002. The design capacity for the ISASMELT furnace is 600,000 tonnes of dry copper concentrate per year. The main aim of the project was to improve environmental performance and decrease energy consumption. Generally the project is going smoothly at the current stage, due to selection of reliable and successful technology. Good preparation work was done. The extensive training program for key people in Mount Isa Copper Smelter improved the technical peopleís understanding of the process greatly and ensured successful hand-over of the process technology. The paper describes the plant layout and initial operating data.

3:20 PM

Modelling of Chalcocite Concentrate Flash Smelting: *Zdzislaw Miczkowski*[1]; Jozef Czernecki[1]; Zbigniew Smieszek[1]; [1]Institute of Non-Ferrous Metals, SowiÒskiego 5, Gliwice 44-100 Poland

KGHM Polska Miedz S.A. applied single-stage process for copper production from chalcocite concentrates in flash furnace. The paper contains model of complex oxidation process of single concentrate grain smelted at KGHM. Description takes into consideration main chemical reactions, heat and mass transfer phenomena, inside the grain as well as between the grain and gas environment. Two zones of process course inside the grain were distinguished. The first zone moves faster within the grain and is caused by increase of temperature inside. Liberation of carbonate and organic carbon occurs there. Second zone penetrates the grain at lower rate. The rate is determined by oxygen presence. Second zone is the place where sulphur is eliminated and vapours of organic compounds are oxidised. On the basis of lab tests results, coefficients characterising oxygen transfer within the grain and determining the rate of chemical reactions were calculated. Model calculations results for single concentrate grain processed in the environment of various oxygen content and model calculations results for concentrate flux smelted in the lab reactor system are presented.

3:45 PM Break

3:55 PM Invited

Copper Converting Versus Steel ConvertingñA Critical Comparison: *Lauri Holappa*[1]; Heikki Jalkanen[1]; [1]Helsinki University of Technology, Lab. of Metall., PO Box 6200, Hut 02015 Finland

Copper converting is mostly performed in Peirce-Smith converters. The process is still very similar to the original one from the late 19th century: a batch process in a horizontal cylindrical reactor with air blowing via tuyeres along the length of the reactor side. This converter is sometimes called Bessemer converter as Bessemer proposed a similar converter to steelmaking. However, finally he came to a vertical pear-shaped converter with air bottom-blowing. In steel converting, a clear continuum can be perceived from air-blowing Bessemer &Thomas processes to oxygen converter processes (LD or BOP) using top-blowing via a lance, to oxygen bottom-blown converters (OBM or Q-BOP) with special tuyeres, and finally to nowadays combined blowing converters utilizing the advances of both top and bottom blowing. In copper converting similar ideas were missing or at least did not lead to large scale success, with the exception of top-blown rotary converters (TBRC or Kaldo) which were used in small extent both for steel and non-ferrous converting. On one respect copper converting has taken a leading position i.e. in the progress of continuous converting. Mitsubishi process as well some new processes like Ausmelt make continuous converting of matte to blister copper by oxygen-air blowing via top lances. A different principle for continuous converting is flash converting by Kennecott-Outokumpu which fully utilizes the exothermic reactions in smelting and converting solid fine matte particles in a reactor shaft similar to Outokumpu matte flash smelting. Although numerous ideas and principles have been tested for continuous steelmaking it seems to be still far from commercial breakthrough. The paper discusses principal physico-chemical and process technical similarities and differences in copper and steel converting. The main processes are reviewed and compared, and the trends are discussed in respect of eventual exchange of knowledge between ferrous and non-ferrous metallurgy.

4:20 PM

Effect of Electric and Magnetic Fields on Metallic Inclusions in a Liquid Slag: *Andrzej Warczok*[1]; *Gabriel Riveros*[1]; [1]Universidad de Chile, Av. Tupper 2069, Santiago 2777 Chile

Liquid slags produced in smelting of copper concentrates contain copper in a dissolved form as well as mechanically entrained inclusions of copper matte. Coalescence and sedimentation of inclusions during slag cleaning plays a key role in copper recovery. Liquid matte inclusion in a molten slag presents a system of metallic conductor in an ionic solution. Constant electric field induces migration of the matte droplet along current lines. Additional phenomena occur in a system under crossed electric and magnetic fields. Electromagnetic buoyancy force acting on the inclusion affects its settling rate. Electromagnetic buoyancy force of insulating and metal spheres has been measured in a liquid synthetic slag. It was found that depolarisation of metal sphere, related to the electrode reactions occurring at the surface, plays an important role in determination of direction and magnitude of the electromagnetic force. On the basis of existing theories and results of measurements the mathematical model, describing the behaviour of metallic inclusions in the liquid slag under the influence of crossed electric and magnetic fields, was developed. Simulation of behaviour of copper matte inclusions under crossed fields showed trajectory of motion in a liquid slag as a function of the slag properties and the inclusion size.

4:45 PM

Factors Affecting the Rate of Copper Reduction During Copper Refining: *Gabriel Riveros*[1]; Andrzej Warczok[1]; Leandro Voisin[1]; Tanai Marin[1]; [1]Universidad de Chile, Av. Tupper 2069, Santiago 2777 Chile

Fire refining of copper consists of two stages oxidation and reduction of copper. Very frequent the capacity of anode furnace is determined by the rate of copper reduction. Analysis of factors affecting the rate of oxygen reduction from a liquid copper with solid carbon or injected hydrocarbons pointed out the role of mass transfer in a liquid copper in final stage of reduction. Experimental results of copper reduction with graphite and injected natural gas in a crucible scale showed two stages of the reduction process with a sharp change of controlling mechanism. Estimated dynamic surface area of gas bubbles based on measured bubble frequency and gas flowrate allowed for determination of reaction constant as a function of gas injection intensity and degree of partial pre-combustion of air/natural gas mix. The variation of reductant utilisation as a function of oxygen concentration and temperature has been evaluated based on the determined kinetic model. Possibilities of intensification of copper reduction in fire refining has been discussed on the basis of obtained kinetic data and analysis of various factors determining the time of reduction.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Iron and Steel Making Fundamentals II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday PM   Room: Leucadia
March 5, 2003   Location: San Diego Marriott Hotel

Session Chairs: Fumitaka Tsukihashi, The University of Tokyo, Dept. of Adv. Matls. Sci. Grad. Sch. of Frontier Scis., Tokyo 113-8656 Japan; Mitsutaka Hino, Tohoku University, Dept. of Metall. Grad. Sch. of Eng., Sendai 980-8577 Japan

2:00 PM Invited

Simulation on the Formation, Dripping and Penetration Behavior of Primary Oxide Melt in the Pyrometallurgical Process: *Mitsutaka Hino*[1]; Atsushi Kumano[1]; Kenko Shimizuno[2]; Tetsuya Nagasaka[1]; [1]Tohoku University, Dept. of Metall., Grad. Sch. of Eng., Sendai 980-8579 Japan; [2]Fujitsu Company, Kawasaki Japan

Primary melt, which appears in the ore during heating, plays an important role in many pyrometallurgical processes. For example, Al_2O_3 is known to be enriched in the primary melt which is formed in the lower part of the ironmaking blast furnace, so that such primary melt could possibly penetrate into micro-porosity of sinter ore and result in the harmful effect on the reducibility of the ore. With keeping such background in view, it has been observed the dripping behavior of Fe_tO-CaO-SiO_2-Al_2O_3-MgO slag from the iron funnel, which has simulated the micro-porosity of sinter ore with the wide range of basicity, Al_2O_3, Fe_tO and MgO contents. The effect of MgO on slag dripping behavior has been summarized in this paper. Premelted slag sample was charged on the iron funnel, suspended in the furnace from thermobalance and heated under Ar stream at 4K/min. Dripping of melt from the funnel and slag hold-up were detected by weight change and its temperature was measured. It was found that the hold-up of the MgO free slag increased with increasing Al_2O_3 content, while this trend became weaker with increasing of the basicity. The addition of 2mass% MgO resulted in large increase of hold-up on the most of the conditions. When FetO content was less than 20mass% and the basicity was lower than 0.8, MgO lowered the hold-up even if the slag contained 10mass% Al_2O_3. Every slag hold-up showed the minimum at the basicity of less than unity. This trend became remarkable when the slag contained 2mass% MgO.

2:30 PM Invited

New Reactor Concepts for Direct Coal-Based Continuous Steelmaking: *Noel A. Warner*[1]; [1]The University of Birmingham, Cheml. Eng., Edgbaston, Birmingham B15 2TT UK

Modern steelmaking is based on direct use of oxygen in high intensity batch reactors. A totally new approach is proposed in this paper, based on the view that pursuit of high intensity is unlikely to lead to successful coal-based continuous steelmaking. By substituting carbon dioxide and water vapor as the oxidants rather than using oxygen directly, the intensity is greatly reduced. Accordingly, subsurface formation of carbon monoxide bubbles can be avoided by careful manipulation of the three participating rate processes, gaseous diffusion, interfacial chemical kinetics and liquid phase mass transfer. Steelmaking can then be carried out without explosive ejection of molten droplets and the generation of micro-spray but rather in a controlled and fumeless fashion. The vital component is the introduction of generic melt circulation technology. Three melt circulation loops in series are envisaged. Starting with a composite feed of iron ore fines, pulverized

WEDNESDAY PM

coal and lime flux, a metallized solid raft floating on molten iron is propelled out of the ironmaking loop onto the first of two downstream steelmaking loops. In the first, a liquid slag is formed, primary decarburization is undertaken not with oxygen but rather with CO2 and H2O and the melt is desulfurized. The semi-product stream enters the third melt circulation loop, where open-channel decarburization is effected along with dephosphorization to yield a low carbon steel product. Alternatively, the melt leaves the last loop to irrigate a packed tower countercurrent to argon at around 100 mbar to continuously produce ultra-low carbon (ULC) steel.

2:55 PM

Kinetics and Morphological Studies of a Carbon Composite Briquette Aiming the Emergent Ironmaking Technologies: *JosÈ Carlos DíAbreu*[1]; JosÈ Henrique Noldin[2]; Karla de Melo Martins[1]; [1]Catholic University, DCMM, Rua Marques de S„o Vicente 225, Sala 542L, G·vea, Rio de Janeiro, RJ 22453-900 Brazil; [2]Catholic University/Tecnored, Ltd., R. Gal. Garzon, 22, 308, Rio de Janeiro, RJ 22470010 Brazil

The first part of this work presents the effects of temperature, gas flowrate and external atmosphere over the reduction rates of a carbon composite briquette (CCB), aiming its use as a burden in some of the new alternate ironmaking technologies, such as Tecnored and RHFs. Conversions were obtained determining the metallization degree, being the pre-exponential factor and the apparent activation energy calculated using the Arrhenius equation. It is reported that raises in temperature, decreasing in N2 flowrate and the use of a CO atmosphere, increase the reaction rate. Into the second part, a morphological study of the iron metallization process during the reduction of these briquettes is presented. The main objective of this investigation was to assess the morphology of the metallic iron formed into the cross section of the samples, for different temperatures and times of reduction, using Optical and SEM images. It was possible to verify that three types of iron morphology occurred: an external, dense and continuous iron layer, and the presence of iron globules and whiskers at the briquetteís core. The measurements of the carbon content on the globules and the external iron layer are also presented.

3:20 PM

Phase Relations During Sintering of Iron Ore and Fluxing Effect of Minor Components: *Florian Kongoli*[1]; Ian McBow[1]; Akira Yazawa[2]; [1]FLOGEN Technologies, Inc., Matls. Tech. Dept., 5757 Decelles, Ste. 511, Montreal, Quebec H3S 2C3 Canada; [2]Tohoku University, 16-32, Niizaka, Aoba-ku, Sendai 981-0934 Japan

The production of a homogeneous self-fluxing sinter is an important step in iron making processes. A good sinter should have good permeability and reducibility and has to keep those characteristics for a certain time. An early melt down of the sinter in the blast furnace, where its solid state reduction is essential, would cause many problems such as low permeability and reductibility. Nevertheless, the important factors that influence these characteristics, such as the chemistry of the sinter and the fluxing effect during sintering and sinter reduction conditions, have not yet been clarified. In todayís new reality where many new minor components such as Al2O3 and MgO enter to the sinter through raw materials, the quantification of the fluxing effects during sintering becomes even more important. In this work, the fluxing effect of many sinter major and minor components has been quantified through the characterization of phase relations in the CaO-FeO-Fe2O3-SiO2 system at sintering conditions and the quantif ication of the effect of Al2O3 and MgO by the means of some practical diagrams which can directly help the optimization of the sintering processes.

3:45 PM Break

4:05 PM Invited

Thermodynamics of Removal of Tramp Elements from Steel Scrap: *Fumitaka Tsukihashi*[1]; [1]The University of Tokyo, Dept. of Adv. Matls. Sci., Grad. Sch. of Frontier Scis., Tokyo 113-8654 Japan

Increasing amounts of steel scrap are being used as resources for steelmaking. Therefore, there has been a growing interest in the removal of harmful tramp elements such as antimony, zinc, tin, arsenic, bismuth, lead and copper from molten steel. The removal of these elements from molten steel by an oxidative treatment is basically impossible and is feasible by using basic fluxes in a reductive refining process. The problem that should be solved is the removal treatment of tramp elements economically in steelmaking process and production of high quality steel by reducing the effect of contaminated tramp elements with satisfying the environmental issues. In this paper, the available thermodynamic data of the tramp elements in molten steel such as the activities of tramp elements, the partition ratio of them between molten steel and sulfide and calcium based fluxes, and the Gibbs free energy of compounds of tramp elements are summarized. Furthermore, the possibility of the removal of tramp elements from molten steel by using various flux systems is thermodynamically estimated by using these thermodynamic data.

4:35 PM Invited

Thermodynamics of Mold Powder: *Hiroyuki Fukuyama*[1]; Kazuhiro Nagata[1]; [1]Tokyo Institute of Technology, Dept. of Chem. & Matls. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

In continuous casting of steel, mold fluxes play some important roles. In particular, horizontal heat transfer from strand to mold significantly affects surface quality of steel. A crystalline layer in slag films yields larger thermal resistance by scattering infrared radiation from steel and forming air gap at the flux/mold interface. Cuspidine (3CaO∑2SiO2∑CaF2) is one of the most important compounds crystallized in slag films during the casting. In order to discuss the crystallization behavior of cuspidine, the present authors have experimentally determined the CaO-SiO2-CaF2 phase diagram around cuspidine. Moreover, the thermodynamic properties of cuspidine are essentially required to discuss the stability field of cuspidine in multi components systems of mold flux. However, no experimental data are currently available on the Gibbs energy of formation of cuspidine. Thus, the present study aims for experimentally determining the Gibbs energy of formation of cuspidine by both electromotive force method and transpiration method.

5:00 PM

Influence of Reduction-Carburization Conditions on the Rate of Iron Carbide Formation: *Abdel-Hady El-Geassy*[1]; Mahmoud Ibrahim Nasr[1]; Mohamd Bahgat Sedikk[1]; [1]Central Metallurgical Research & Development Institute, Iron-Making Div., PO Box 87-Helwan, Cairo Egypt

High grade iron ore fines rejected from the DRI plants were isothermally reduced in H2/CO gas mixtures at 550-850°C. The freshly reduced reaction products were then subsequently carburised in either H2/CO or H2/CO/S. Thermogravimetric analysis technique was used to follow up both of reduction and carburisation reactions as a function of time. The carburised samples were characterized by X-ray phase identification, Mossbaure and carbon and chemical analyses. The influence of temperature, H2:CO ratio in the gas mixtures and carburisation time were intensively investigated and correlated. A conversion extent of iron to Fe3C was taken as a measurable index for the efficiency of carburisation reaction under the different experimental conditions. The presence of sulfur greatly stabilize the Fe3C formation resulting 96% conversion extent in 40%H2/CO mixture at 650°C.

15th International Symposium on Experimental Methods for Microgravity Materials Science - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee
Program Organizers: Robert Schiffman, R. S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica & Instituto Nazionale di Fisica della Materia, Bologna 40127 Italy

Thursday AM Room: 10
March 6, 2003 Location: San Diego Convention Center

Session Chair: Reza Abbaschian, University of Florida, Matls. Sci. & Eng., Gainesville, FL 32611-6400 USA

8:30 AM
Ampoule Design Experimentation for Microgravity Processing of Metal Alloys: *D. A. Downs*[1]; J. B. Andrews[1]; [1]University of Alabama-Birmingham, Microgravity Solidification Lab., Dept. of Matls. Sci. & Eng., Rm. 254 BEC, 1150 10th Ave. S., Birmingham, AL 35294 USA

The Coupled Growth in Hypermonotectics (CGH) experiment involves the directional solidification of aluminum-indium alloys and is designed to investigate the fundamental mechanisms of monotectic solidification. Initial experiments carried out during the STS-78 Life and Microgravity Spacelab (LMS) Mission used specially designed aluminum nitride ampoule assemblies. The specialized design of these ampoules is critical to obtaining correct, consistent, processing conditions. Past and current ampoule development efforts have highlighted ampoule design features that promote desirable performance as well as features that proved troublesome. The results of continued furnace testing of different ampoule configurations will be presented.

8:50 AM
New Optical Method for Measuring Densities of Containerless Processed Liquid Metals: *J¸rgen Brillo*[1]; Ivan Egry[1]; [1]Deutsches Zentrum f¸r Luft-und Raumfahrt e.V. (DLR), Inst. f¸r Raumsimulation, Linder Hˆhe, Kˆln-Porz D-51174 Germany

We have developed a new experimental technique for density determination of electromagnetically levitated metallic liquids. This method employs an enlarged beam of parallel laser light to produce a shadow image of the sample. The shadow is recorded by a digital CCD-camera and the images are analysed using an edge detection algorithm. The circumference is fitted by Legendre polynomials which allows to calculate the volume. The advantage of this procedure compared to those techniques recording the sample image directly is that interferences caused by the samples movement are being avoided. Hence there is no change of the apparent size of the sample. In addition, effects that might occur from the sampleís radiation are suppressed as there is a constant contrast between the shadow and the background. A detailed description will be given and first results on measurements of the Ni-Cu- Fe- system will be presented.

9:10 AM
Thermophysical Property Measurements of Molten Metals and Ceramics by Electrostatic Levitation: *Takehiko Ishikawa*[1]; Paul-Francois Paradis[1]; Jianding Yu[1]; Tomotsugu Aoyama[1]; Shinichi Yoda[1]; [1]National Space Development Agency of Japan, 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan

Thermophysical property measurements, in particular density, vapor pressure, surface tension, and viscosity, proved to be very challenging by means of electrostatic levitation when dealing with refractory metals (e.g. Mo, Ta), high vapor pressure metals (e.g. Ti, Mo), and ceramics (e.g. oxides). The main issues to be addressed are the levitation initiation, the time required to achieve a molten state of a sample, the high luminosity of the droplet, and, sometimes, severe evaporation. This paper briefly summarizes the procedures and methods adopted by the National Space Development Agency of Japan with an electrostatic levitator furnace to enable thermophysical property measurements of the aforementioned materials. Typical data that would have been impossible to obtain without the use of these techniques and procedures, namely the density of oxides (e.g. $BiFeO_3$, YAG), the vapor pressure of Ti, and the surface tension and viscosity of Mo are reported for superheated and undercooled phases.

9:30 AM
Accurate Thermophysical Property Measurements of Liquid Si by Electrostatic Levitation: *Zhenhua Zhou*[1]; Won-Kyu Rhim[1]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., Pasadena, CA 91125 USA

Several improvements have been done with the high vacuum electrostatic levitator in Caltech. Four laser beams with the tetrahedral arrangement heating technology was developed to greatly reduce the temperature gradient inside the levitated sample. A new image processing technology was adopted to get precise sample volumes. The DOAP (Division-of-amplitude photopolarimeter) was installed and used to measure the spectral emissivity of the liquid Si down to the highly undercooled region. The spectral emissivity data together with the single color pyrometer can give the accurate temperature of the levitated Si sample. Based on these new improvements, accurate temperature dependent thermophysical properties such as density, thermal expansion coefficient, spectral emissivity, specific heat over total hemispherical emissivity, viscosity and surface tension of liquid Si can be obtained. The contactless measurements and the uniform temperature distribution inside the levitated sample together with the accurate temperature measurement will result in more reliable data than before.

9:50 AM
A Passive Thermal Carrier (Lotec) for Temperature Sensitive Materials Being Moved to and from the International Space Station and for Shuttle (Sts) Flights: *F. C. Wessling*[1]; J. M. Blackwood[1]; H. R. Holt[1]; [1]The University of Alabama-Huntsville, Dept. of Mechl. & Aeros. Eng., Huntsville, AL 35899 USA

A Low Temperature, low Energy Carrier (LoTEC) maintains steady state temperatures without the use of power for transporting temperature sensitive materials to and from the International Space Station. LoTEC uses a combination of high thermal resistance insulation, careful thermal design and phase change materials to maintain temperature. It has been tested with phase change materials for three different interior temperatures (-16C, 0C, +4C). These temperatures are usable for preservation of many biological materials during transport. The exterior dimensions of LoTEC are approximately 250.7 x 436.9 x 514.1 mm. Thus, LoTEC fits into a standard mid-deck locker, an Express Rack, a SpaceHab rack or a rack in the Multi-Purpose Logistics Module (MPLM). LoTEC has an empty mass of approximately 9.6 kg and an internal volume of 22 Liters. This volume is reduced by the amount and type of phase change material (PCM) used. For example, eight kg of water ice and its containers would take up approximately half of the internal volume, leaving 11 Liters of space for the payload. Several different configurations have been considered to accommodate different payload dimensions. The interior dimensions of LoTEC are approximately 406 x 343 x 158 mm. Measured energy gain by LoTEC is approximately 0.14 watts per degrees Celsius. Performance data in the form of temperature versus time curves are included. Eight kg of PCM gives approximately eight to ten days of constant temperature at typical STS interior temperatures. The temperature distribution in LoTEC is dependent on the PCM containers, the PCM used, and the location of the containers in LoTEC. Small, stand-alone data loggers record the temperatures.

10:10 AM Break

10:30 AM
Development Status of Electrostatic Levitation Furnace(ELF) for Kibo: Keiji Murakami[2]; Takehiko Ishikawa[2]; Kazunori Kawasaki[1]; Shoji Muramatsu[1]; Hiroaki Asahi[1]; *Takahiro Nishimura*[1]; [1]IHI Aerospace Company, Ltd., Space Sys. Dept., 900 Fujiki, Tomioka, Gunma 370-2307 Japan; [2]National Space Development Agency of Japan, Space Utilization Rsrch. Ctr., 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan

The development status of Electrostatic Levitation Furnace (ELF) system for KIBO is introduced. Several new functions and properties are studied. (1) Small and Vacuum Chamber: It succeed in the miniaturization of chamber by adoption of small components and the device of a layout. The chamber will provide the pressurized condition as well as the very high level vacuum condition. (2) Tetrahedral Laser Heating: Four lasers are placed as tetrahedral configuration to accomplish uniform heating and sample rotation suppression. (3) Cage Exchangeability: In order to meet the various user requirements and refreshing electrodes, the chamber consists of the duplex structure made from the chamber itself and the exchangeable sample cage. (4) High-speed Sample Position Sensing: High-speed sample position sensing system makes

1kHz (maximum) interval sample position control is provided. The parabolic flight test and some ground tests using the Bread Board Model have been performed in the current development phase.

10:50 AM

ATENñA Multi-Purpose Furnace for Material Science Research on the International Space Station: *C. Casgrain*[1]; L. Misener[2]; [1]Canadian Space Agency, St-Hubert, Quebec J3Y 8Y9 Canada; [2]Millenium Biologix Inc., Kingston, Ontario K7M 7G3 Canada

Canada is a partner of the International space station (ISS), providing the Mobile servicing system, a sophisticated robotic arm used to assemble and maintain the station. In exchange for its contribution the Canadian scientists will have the right to use 2.3% of the non-Russian portion of the International Space Station for research over the fifteen years of its expected life. For the Microgravity Science Program (MSP) and its scientific community, this includes the continuous use of one double middeck locker. One important mandate of MSP is to develop state-of-the-art payloads to support world class research as well as a facility to improve the microgravity environment quality. An effective use of MSPís double locker on ISS is to devote the bottom part to the isolation system against vibrations (MIM Base Unit) and the top part for small exchangeable payloads. We are presently developing the fourth generation of the CSAís locker-sized furnace payload called ATEN (Advanced Thermal ENvironment). ATEN will be the first MSP payload to fly on the MIM Base unit. The ATEN project started in February 1999 with a survey of the scientific community and four feasibility studies. In January 2001, Millenium Biologix Inc in Kingston Ontario was selected to design and build the ATEN furnace in a two-year timeframe. The ATEN furnace is designed to meet a wide range of scientific requirements. It will allow investigators to do fundamental studies in diffusion, Ostwald ripening and particle pushing as well as improving material processing techniques to grow semi-conductors, ceramics and glasses of better quality. Therefore ATEN will provide different modes of operation such as isothermal, gradient heating and melt zone heating. ATEN will be able to process specimens up to 10 mm in diameter and 80 mm in length. The furnace will operate in isothermal mode at temperatures varying from 100∫C to 1300∫C and in temperature gradient mode with a gradient varying from 5∫C/cm to 50∫C/cm. The facility will also provide melt zone mode. The temperature accuracy will be within ±0.25% at high temperature with a repeatability of ±1∫C. One of the keys to successful experiments in space is extensive ground preparation. The great advantage of ATEN is that it is a compact payload and Ground Units (GU) will be made available to scientists for mission preparation and experiment fine-tuning. As mentioned, ATEN will fit into the Microgravity vibration Isolation Mount Base Unit (MIM Base Unit) enclosure. The MIM Base Unit will provide all necessary mechanical and electrical interfaces, power, cooling, data processing capabilities, data storage and communication interface for tele-operation in addition to providing isolation from vibrations. In December 2001, ATEN successfully underwent its Preliminary Design Review (PDR) at CSA headquarters in St-Hubert. The CDR is planned for November 2002 and the final delivery is planned for the summer of 2003 while the first flight opportunity is foreseen in 2005. The aim of the present paper is to present the ATEN payload technical features as well as the major challenges undertaken during the design of the payload.

11:10 AM Cancelled

German Facility Developments for Microgravity Materials Sciences: *Rainer Kuhl*[1]; Horst Binnenbruck[1]; [1]German Aerospace Center, Space Mgmt., DLR, Microgravity Rsrch. & Life Scis., Bonn 53227 Germany

11:30 AM

Determination of Nucleation Kinetic Parameters of Metallic Melts Using Electrostatic Levitation Techniques: Melissa J. Wert[1]; *William H. Hofmeister*[1]; Robert J. Bayuzick[1]; Jan Rogers[2]; Thomas Rathz[2]; Glenn Fountain[2]; Robert Hyers[3]; [1]Vanderbilt University, Cheml. Eng., Box 1604, Sta. B, Nashville, TN 37235 USA; [2]NASA, Marshall Space Flight Ctr., MC SD47, Huntsville, AL 35812 USA; [3]University of Massachusetts, Dept. of Mechl. & Indl. Eng., Amherst, MA 01002 USA

Electrostatic levitation is an elegant way of processing metals in a high vacuum environment and is extremely useful in obtaining undercooling data required to determine nucleation kinetic parameters of metallic melts. Bulk zirconium samples (~2 mm diameter) of varying oxygen content are levitated at vacuum levels in the range of 1 x 10-8 Torr, minimizing contamination by container walls or residual gasses in the system. Numerous undercooling cycles (>100) are obtained for each sample, generating an undercooling distribution representative of the sampleís kinetic behavior. A statistical analysis is performed on the undercooling distributions of each sample, resulting in values for the kinetic prefactor, K_v, and work of formation of a critical nucleus, ΔG^*, using the Spaepen-Turnbull nucleation rate equation. Glow discharge mass spectrometry is used to determine oxygen content of each sample. Nucleation kinetic parameters are found to increase with increasing sample purity.

11:50 AM

Preparation of Al-Li Alloy with Electric Magnetic Force Simulation Facility: *Da Daoan*[1]; Hao Weixin[1]; Geng Gruihong[1]; [1]Lanzhou Institute of Physics, Gansu 730000 China

By adopting the usual smelting technology, serious segregation will occur when the content of Li exceed 3%. We discovered that the impact of gravity can be counteracted by Lorentz force generated by the electric magnetic force simulation facility. The contents of Li in the Al-Li alloy can be increased to 10% with density of 2.0g/cm, of which the non-uniformity of Li and Al element distribution at lower than 3%. The hardness measurement result of Al-Li alloy with 7% Li content shows that Vickers hardness can reach to 103.9.

Aluminum Reduction Technology: Pot Control

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Martin Segatz, VAW Aluminum Technology, Bonn D-53117 Germany; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Thursday AM — Room: 6B
March 6, 2003 — Location: San Diego Convention Center

Session Chair: Gary Tarcy, Alcoa Inc., Hall Process Improv. Alcoa Techl. Ctr., Alcoa Ctr., PA 15069-0001 USA

8:30 AM

Aluminum Fluoride Control Strategy Improvement: Abdelhamid Meghlaoui[1]; *Najeeba Hassan Aljabri*[1]; [1]Dubai Aluminum Company, Ltd., Process Control & Dvlps., Jabel Ali, Dubai UAE

The AlF_3 is an important control parameter that has a considerable impact on the cell performance. An AlF_3 control strategy is developed on the basis of Entner model using the fuzzy logic to predict the daily AlF_3 concentration required for calculating the AlF_3 additions. Entner model is based on the AlF_3 dynamic reactive mass balance taking in consideration the time lag reaction. The main model input-parameters are the AlF_3 concentration in the bath and the historical additions. Because of economical considerations, the bath analysis is not performed daily but estimated from bath temperature daily measurements using fuzzy logic. This paper presents the control algorithm and the methodology to select adequately the model input-parameters as well as the fuzzy logic membership functions using a plant superheat data. Results from different cell technology show that fuzzy Entner based method improves dramatically the cell performance compared to the plant existing control strategy.

8:55 AM

Reduction Cell Noise Analysis: *Larry Banta*[1]; Congxia Dai[1]; [1]West Virginia University, Mechl. & Aeros. Eng., PO Box 6106, Morgantown, WV 26506 USA

In most reduction cell control schemes, anode position is a compromise between maintaining the lowest possible cell voltage and prevention of unacceptable levels of ìnoiseî. Exactly what constitutes noise varies from controller to controller, however, and there are many metrics used to quantify the noise level in a pot. It has long been known that cell voltage noise has a variety of sources, and at least some cell controllers make no attempt to distinguish one type of noise from another in determining control actions. This paper describes work underway at WVU to discriminate different noise sources by analysis of the types of noise signals they generate, and to suggest appropriate control strategies based on noise type as well as noise levels.

9:20 AM

Neural Network Qualifier of Noises of Aluminium Reduction Cells: Alexander I. Berezin[1]; *Peter V. Poliakov*[2]; Oleg O. Rodnov[1]; Igor V. Mezhubovsky[1]; Igor V. Gonebnyy[1]; [1]Mayak PKF, Ltd., Bograda st. 108, Krasnoyarsk 660021 Russia; [2]STC ìLight Metalsî, Vavilova st. 60, Krasnoyarsk 660025 Russia

The raw voltage signals from the aluminium reduction cells contain fluctuations, named in industrial practice by ìnoiseî, which can be used to identify problems with the cell operation. It is known, that each type of technology disturbances is characterized by unique noise. We have developed the Neural Network Qualifier of noises of aluminium reduction cells for use in control systems. Qualifier allows to distinguish noises caused by technology disturbances, and, thus, to identify them at an early stage of occurrence. Before the beginning of work the Qualifier is training for adaptation to noises of a concrete cell designs. For this purpose the network studied the images of noises, which were registered on cells with known disturbances. Trained Qualifier is used for the analysis of noises on working cells. Seeing similarity between known examples and real images of noise, the network identifies 6 technological ìillnessesî of cells.

9:45 AM

Analysis and Correction of Heat Balance Issues in Aluminum Reduction Cells: *Philip Biedler*[1]; Larry Banta[1]; [1]West Virginia University, PO Box 6106, Morgantown, WV 26506 USA

The heat balance in an aluminum reduction cell is a complex process composed of interrelated variables including power input, bath composition, bath and metal pad depth, cover depth, material properties and cell geometry. Because the process is dynamic, cells often drift from the targeted heat balance. When this happens, actions must be taken to correct the problem. However, the most appropriate action and the consequences of that action are not always known. To address these issues, a 2-D finite element model was developed to model the thermal, electrical, and chemical processes occurring in the cell. This model was used to study the interrelation of the variables affecting the heat balance and to analyze the effect of corrective actions taken to rectify several heat balance issues.

10:10 AM Break

10:20 AM

Fully Distributed Computer Control System for Aluminum Electrolysis: *Fengqi Ding*[1]; *Jie Li*[1]; Yinggang Jiang[1]; Wengen Zhang[1]; Zhong Zou[1]; Yexiang Liu[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

A fully distributed computer control system for aluminum electrolysis was developed. In the system, the supervision level made up of workstations equipped in the computer station and workshop sections and the direct control level consisting of cell controllers were connected with CAN bus to form a control network, with every workstation being able to supervise the cell controllers in its permitted supervision range; on the other hand, every workstation on the supervision level was linked into the plant LAN to meet the needs of setting up plant MIS. The cell controller was also designed in the form of network structure in which several intelligentized modules were connected with inner CAN bus to realize their data exchange and multi-CPU cooperation. Till now about 2000 cell controllers of this kind have been successfully applied at more than 20 aluminum plants in China.

10:45 AM

Increased Current Efficiency and Reduced Energy Consumption at the TRIMET Smelter Essen Using 9 Box Matrix Control: *Torsten Rieck*[1]; Martin Iffert[1]; Peter White[2]; Renuka Rodrigo[3]; Rik Kelchtermans[2]; [1]Trimet Aluminium AG, Aluminiumallee 1, Essen D-45356 Germany; [2]Heraeus Electro-Nite International NV, Centrum-Zuid 1105, Houthalen B-3530 Belgium; [3]Heraeus Electro-Nite Company, 9901 Blue Grass Rd., Philadelphia, PA 19114 USA

Energy consumption is one of the key issues in the production of primary aluminium. It is therefore a great challenge for aluminium smelters worldwide to increase current efficiency and at the same time reduce energy consumption. At the Essen Smelter a new type of energy and mass balance control algorithm has been developed and successfully implemented on an industrial scale. Central to this new strategy are superheat measurements. Supporting measurements include bath temperature and cathode voltage drop. Measurement data are input into a newly developed 9 box matrix control algorithm. The concept utilizes pot voltage adjustments as the primary control tool to drive the bath temperature into a predefined control band. As a secondary control, aluminium fluoride additions are based upon the liquidus trend. Through combined stabilization of energy and mass balance a stable cell operation is achieved, leading to the benefits of higher current efficiency and reduced energy consumption.

11:10 AM

Estimation of a Technological Condition of the Aluminium Reduction Cells on the Basis of its Daily Energy Balance: Oleg O. Rodnov[1]; *Peter V. Poliakov*[2]; Alexander I. Berezin[1]; Pavel D. Stont[1]; Igor V. Mezhubovsky[1]; [1]Mayak PKF, Ltd., Bograda st. 108, Krasnoyarsk 660021 Russia; [2]STC ìLight Metalsî, Vavilova st. 60, Krasnoyarsk 660025 Russia

It is necessary for smelters to have expert systems of analysis, which will permit to conduct deep evaluation of the technology of aluminium electrolysis with the purpose to determine the reasons, which provoke technology disturbances, to investigate and optimize operations. We offer methodology to analyze of the statistical data of electrolysis parameters permitting to determine daily an energy balance of the cell. The technique establishes interrelation of technological parameters of electrolysis by means of the introducing of new technological criterion, specially designed for the solution of put problems. In activity is submitted the conclusion of criterions describing interrelation of technological parameters of electrolysis and permitting to identification ìhealthî and ìillî of the cell. The conclusion of technological criterions bases on a theory of similarity and dynamical model of the aluminium reduction process.

11:35 AM

A Supervised System for Aluminum Reduction Cell: *Zeng Shuiping*[1]; [1]North China University of Technology, Inst. of Automatic Control, Jiyanzhang Rd. 5, Shijingshan Dist., Beijing 100041 China

This paper studied a online supervised system for aluminum reduction cell, which include the sample of data related with the process, the real-time display of some operation parameters and technical remarks, the control command for the process, alarm of abnormal condition and production management. This system can also analyze the operation techniques, judge the system conditions and give out the evaluation for the present status. The system is made with the tool of Visual Basic and the tool of Structured Query Language in database control. With the open architecture and the existing interface of I/O, the software can realize the special tasks required by individual operator, which performed well in actual production.

Cast Shop Technology: Foundry Technology II

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; David H. DeYoung, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Seymour G. Epstein, The Aluminum Association, Inc., Washington, DC 20006 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Thursday AM — Room: 6C
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: Daryoush Emadi, National Research Council of Canada, Matls. Techl. Lab., Ottawa, ON K2B 8L4 Canada; John Grandfield, CSIRO Australia, Preston, Victoria 3072 Australia

8:30 AM Cancelled

Thermal Two-Phase Finite Element Flow Model for Materials in the Semi-Solid State: *FrÈdÈric Pineau*[1]; [1]National Research Council Canada, Process Modlg. & Instrumentation, 75 de Mortagne Blvd., Boucherville, QuÈbec J4B 6Y4 Canada

8:55 AM

Hydrogen Gas Pick-Up of Al-Alloy Melt During Lost Foam Casting: *Seung-ryoul Shin*[1]; Zin-hyoung Lee[1]; [1]KAIST, Matls. Sci. & Eng., 373-1, Kusung-dong, Taejon, Yusung-gu 305-701 Korea

The hydrogen gas pick-up problem that can occur during LFC (Lost Foam Casting) was investigated. The gas content of the melt was measured by reduced pressure test and the proper test pressure was determined. The hydrogen pick-up increased with the increased amount of polystyrene that was replaced by melt. The hydrogen pick-up depended on the initial hydrogen content of the melt and the contact time of the melt with the decomposed gas phase. The effects of evacuating the chamber containing the mold, pouring system and coating thickness were investigated on hydrogen concentration and flow length of melt. Also the solidification of the melt and the gas formation were monitored using a high speed camera through the quartz window mount on the mold during the process.

9:20 AM Cancelled

Vacuum Die-Casting of Wrought 7050 & 7150 Aluminum Alloys: *J. T. Staley*[1]; *Greg N. Colvin*[1]; [1]Alcoa Inc., Howmet Rsrch. Corp., 1500 S. Warner St., Whitehall, MI 49461 USA

9:45 AM

Numerical Modeling of Squeeze Casting Magnesium Alloy AM50 with Natural Convection: *Alfred Yu*[1]; *Henry Hu*[1]; *Naiyi Li*[2]; [1]University of Windsor, Mechl., Matls. & Auto., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ford Motor Company, Mfg. Sys.

Dept., Ford Rsrch. Lab., Rm. 2349, MD3135, 2101 Village Rd., Dearborn, MI 48124 USA

Natural convection has received less attention than conduction in metal solidification processes. It may play an important role in solidification due to its potential influence on solutes distribution in melts and grain structure. A coupled mathematical model has been developed to simulate the solidification phenomena of squeeze cast magnesium alloy (AM50). An analysis based on a Control-Volume Finite Difference approach and an Enthalpy Transforming Model was employed to understand the heat transfer and fluid flow in a cylindrical casting during solidification. The effect of natural convection on the total solidification process of squeeze casting AM50 was examined under various processing parameters, and comparison results are presented and analyzed.

10:10 AM Break

10:20 AM

Directional Solidification of Aluminum-Copper Alloys: Alicia Esther Ares[2]; Rubens Caram[3]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Scis., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]CONICET/University of Florida, Dept. of Matls. Sci. & Eng., 131 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA; [3]State University of Campinas, Dept. of Matls. Eng., Campinas, Sao Paulo 6122 Brasil

The columnar to equiaxed transition (CET) was studied in Al-2wt%Cu, Al-4wt%Cu, Al-10wt%Cu, Al-20wt%Cu, Al-33.2wt%Cu alloys, which were solidified directionally from a chill face. The main parameters analyzed include the temperature gradients, solidification velocities of the liquidus and solidus fronts, and grain size. The transition was observed to occur when the temperature gradient in the melt decreased to values between -3.89∞C/cm to 1.25∞C/cm. In addition, there is an increase in the velocity of the liquidus front faster than the solidus front, which increases the size of the mushy zone. The size of the equiaxed grains increase with distance from the transition, an observation that was independent of alloy composition. The results are compared with both, obtained previously in lead-tin alloys and obtained by other authors.

10:45 AM

Analysis of Grain Size Obtained During Directional Solidification of Aluminum Based Alloys: Alicia Esther Ares[2]; Rubens Caram[3]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Scis., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]CONICET/University of Florida, Dept. of Matls. Sci. & Eng., 131 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA; [3]State University of Campinas, Matls. Eng. Dept., Campinas, Sao Paulo 6122 Brasil

In the present work measurements of columnar and equiaxed grain size were carried out. The measurements were made from the macrography of aluminum alloys samples such as Al-Cu, Al-Mg, Al-Zn, Al-Si, Al-Si-Cu unidirectionally solidified and which presented a columnar to equiaxed transition. The results of the analysis are compared with those obtained in a previous work in alloys with low melting point, Pb-Sn. The length of the columnar grains and the size of the equiaxed grains are correlated with dynamic parameters derived from temperature measurements, such as, cooling rate, temperature gradients and velocity and position of the liquidus and solidus fronts. The results obtained are presented and discussed.

11:10 AM

Spheroidization of Silicon and its Influence on the Mechanical Properties of the Eutectic Al-12% Si Alloy: *Ruyao Wang*[1]; Weihua Lu[1]; Hsienyang Yeh[1]; Henry H.E. Yeh[1]; [1]Donghua University, Dept. of Mechl. Eng., 1882 W. Yanían Rd., Shanghai 200051 China

A procedure has been developed to vary the morphology of silicon in eutectic Al-12%Si alloy (US patent pending). This technology consists of two steps: firstly, adding small amount of designed modifier into melt, then heating the treated alloy at temperature as in solution treatment. Depending on the holding time at soaking temperature, the silicon morphology varies from connected flake to bar-like shape to, finally, nodule. Experiment showed that the heating temperature designated is much lower than that required in conventional Al-Si alloy, not resulting in coarsening or clustering of silicon particles and making spheroidization of silicon possible. The separated bar-like silicon is capable of remarkably reinforcing the matrix of Al-Si alloy, raising the tensile strength by 30% compared to that in Al-Si alloy with connected silicon. However, the high level of spheroidization of silicon particle doesnít greatly increase tensile strength and ductility, but dramatically raises the wear resistance by 150%.

11:35 AM Cancelled

Hypereutectic Al-Si Alloys with a Completely Self-Modified Eutectic Structure: *Ruyao Wang*[1]; Weihua Lu[1]; [1]Donghua University, Shanghai China

Computational Phase Transformations: Engineering Applications

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)

Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Thursday AM Room: 11B
March 6, 2003 Location: San Diego Convention Center

Session Chairs: J. P. Simmons, Air Force Research Laboratories, Dayton, OH 45433 USA; Zi-Kui Liu, Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

8:30 AM Invited

PrecipiCalc: An Efficient Numerical Precipitation Code for Materials and Process Design: *G. B. Olson*[1]; [1]Northwestern University, Dept. Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The Langer-Schwartz theoretical framework for precipitation kinetics is extended to multicomponent systems by a numerical code built on the Thermocalc/DICTRA system for multicomponent thermodynamics and diffusion. Under the DARPA-AIM program on Accelerated Insertion of Materials the code is linked to finite-element simulations of heat treatment of turbine discs to simulate the spatial variation of gí precipitation in IN100 and Rene88DT superalloy discs, with model validation by high resolution microanalytical studies. The code is being tested on several precipitation systems, including the devitrification of metallic glasses under the DARPA-SAM initiative on Structural Amorphous Metals.

9:00 AM

The Modelling of Phase Transformations in the Context of a Generalised Materials Property Capability: *Nigel Saunders*[1]; Xiuqing Li[2]; Alfred Peter Miodownik[1]; Jean-Philippe Schille[2]; [1]Thermotech, Ltd., Surrey Technology Ctr., The Surrey Rsrch. Park, Guildford, Surrey GU2 7YG UK; [2]Sente Software, Ltd., Surrey Tech. Ctr., The Surrey Rsrch. Park, Guildford, Surrey GU2 7YG UK

As part of the development of a new software programme (JMatPro) for calculating general materials properties, work has been undertaken on phase transformations for Al-, Fe-, Ni- and Ti-alloys. Current capability exists for the calculation of non-equilibrium solidification, TTT/CCT diagrams, Ms, gammaí/gammaî coarsening, etc. Examples will be presented on how information from a variety of other subject areas (e.g. thermodynamics, physical and mechanical property modelling) has been combined with phase transformation modelling to provide a more general materials property capability. Examples will include, the combination of solidification models with physical properties calculations, to provide density, volume changes, thermal conductivity etc. and the calculation of gamma/gammaí lattice mismatch as a function of temperature in Ni-based superalloys, which is controlled not only by thermal expansion differences but also by potential changes in gamma/gammaí equilibrium. Further extensions will be discussed including the effect on secondary creep rate of both coarsening of gammaí and the time dependent depletion of heavy elements in gamma due to formation of TCP phases such as sigma.

9:20 AM Invited

On Modeling the Microstructure and Coarsening Kinetics of γí Precipitates in Ni-Base Alloys: *Alan J. Ardell*[1]; [1]University of

THURSDAY AM

California-Los Angeles, Matls. Sci. & Eng., 6531-G Boelter Hall, Los Angeles, CA 90095-1595 USA

The γí (ordered Ni_3Al) precipitate microstructure and its evolution during coarsening in aged binary Ni-Al alloys is a favorite son of the computational modeling community. Many features of the precipitate microstructure have been captured beautifully, e.g. precipitate alignment along elastically soft <001> directions, and their cuboidal shapes. However, several aspects of coarsening have not been investigated by computational methods. These include the anomalous decrease of the coarsening rate with increasing volume fraction, f; the suggestion that γí precipitate shapes might depend on f when f is small, and the concave interfaces of large individual γí precipitates when f is small (< 0.04). Furthermore, many computations use generic values of physical parameters, and therefore cannot reproduce differences among experimentally observed precipitate microstructures; e.g. why Ni_3Ge and Ni_3Al precipitates differ so in shape and spatial distributions. Useful targets for future efforts of computational modeling will be discussed in this presentation.

9:50 AM
Coarsening of Misfitting Particles: Ostwald Ripening and Coalescence: *K. Thornton*[1]; Norio Akaiwa[2]; P. W. Voorhees[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; [2]National Research Institute for Metals, Tsukuba Japan

We present the results from two-dimensional simulations of Ostwald ripening of second-phase particles in an elastically anisotropic, homogeneous system with cubic symmetry. We utilize advanced numerical methods such as the fast multipole method to include a large number of particles to provide sufficient statistics. Calculated microstructures exhibit experimentally observed features like particle alignment and four- and two-fold symmetric particles. We characterize the microstructure evolution through analyses of the particle morphology, the size distribution, and the spatial distribution. Although the evolution of the system is not self similar, there is strong evidence that the system, including its kinetics, is approximately characterized by the ratio between the elastic energy and the interfacial energy. At high volume fractions, particle separations become so small that coalescence is possible. We discuss the influence of coalescence on the evolution of large systems of misfitting particles undergoing coarsening.

10:10 AM Break

10:25 AM Invited
Phase Field as an Engineering Tool for Heat Treatment Applications: *Jeff P. Simmons*[1]; Youhai Wen[2]; Chen Shen[3]; Yunzhi Wang[3]; [1]Air Force Research Laboratory, AFRLMILLMP, Matls. & Mfg. Direct., WPAFB, Dayton, OH 45433 USA; [2]UES, Inc, Dayton, OH 45432 USA; [3]The Ohio State University, Dept. of Matls. Sci. & Eng., Columbus, OH 43210 USA

The Phase Field model has been demonstrated to have a very wide range of applications to microstructural modeling phenomena, with applications that include both diffusional and shear transformations as well as grain growth phenomena. Like all microstructure simulation techniques, it produces all microstructural information, including particle size distributions, interparticle spacings, particle morphologies, as well as unusual (far from mean) events. In order to develop the Phase Field model into an engineering tool, two practical considerations must be made: (1) how to perform simulations under conditions of concurrent nucleation and growth and (2) quantitative comparison with microstructural descriptors such as volume fraction vs. time, coarsening rates, and particle size distribution effects. This presentation summarizes progress to date towards developing the Phase Field model into a tool suitable for engineering shop practice.

10:55 AM
Phase Field Method and Quantitative Microstructure Modeling: *C. Shen*[1]; Q. Chen[1]; Y. Wang[1]; Y. Wen[2]; J. P. Simmons[3]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [3]Air Force Research Laboratory, AFRL/MLLM, WPAFB, Dayton, OH 45433 USA

Phase field method has been developed for decades. It uses spatially distributed (field) variables to describe kinetic evolutions of material systems containing spatial non-uniformity in material properties. Its capabilities to describe complex microstructural patterns make it attractive for engineering applications. In this presentation we discuss a number of issues related to quantitative applications of the phase field method to engineering systems, including the incorporation of thermodynamic and mobility databases and necessary scaling of energy, length and time. Examples will be given on the development of γ/γí microstructures in Ni-base superalloys by nucleation, growth, and coarsening under isothermal and non-isothermal conditions.

11:15 AM
Modeling of Microstructure Evolution During the Beta to Alpha Transformation in Ti-6Al-4V Alloy: *Qing Chen*[1]; Chen Shen[1]; Kaisheng Wu[1]; Yunzhi Wang[1]; [1]The Ohio State University, Dept. Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

A combination of phase field method and classic nucleation theory has been developed to simulate the formation of grain boundary and Widmanst‰tten alpha structures during the beta to alpha transformation in Ti-6Al-4V alloy. The dependence of non-equilibrium chemical free energy on composition, temperature and long-range order parameter was established on the basis of available CALPHAD thermodynamic description. Newly assessed diffusion mobility data for alpha precipitates and beta matrix have been used in the phase field simulation. The roles of elastic strain energy, interfacial energy and interface mobility in the formation of Widmanst‰tten alpha plates have been investigated. This work is supported by AFRL under MAI contract.

11:35 AM Cancelled
Prediction of Phase Transformation Kinetics and of Ferritic Microstructures in Low-Carbon Steels: *Matthieu Kandel*[1]; [1]IRSID, Physl. Metall. Dept., Voie Romaine, BP30320, MaiziËres-les-Metz 57283 France

11:55 AM
The Quaternary Ni-Al-Mo-Ta Thermodynamic Database for Ni-Base Superalloys: *Shihuai Zhou*[1]; Longqing Chen[1]; Zi-Kui Liu[1]; [1]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., Steidle Bldg., University Park, PA 16802 USA

The thermodynamic database of the Ni-Al-Mo-Ta system is developed for the Ni-base superalloys with special attention to the stability of the topologically close-packed phases. With the CALPHAD technique using a computerized optimization procedure, the thermodynamic descriptions of the compounds δ-NiMo in Ni-Mo system, the ternary compounds in the ternary Ni-Al-Mo, Ni-Al-Ta and Ni-Mo-Ta systems are evaluated. The thermodynamics of the γ and γí phases are modeled with a single Gibbs energy function taking into account the crystallographic relation between these two phases. Various phase diagrams calculated with the current database will be presented and compared with experimental data.

Friction Stir Welding and Processing II: Friction Stir Processing

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Thomas J. Lienert, University of South Carolina, Mechanical Engineering Department, Columbia, SC 29208 USA; Rajiv S. Mishra, University of Missouri-Rolla, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Thursday AM　　Room: 7B
March 6, 2003　　Location: San Diego Convention Center

Session Chair: Rajiv S. Mishra, University of Missouri-Rolla, Metallurgl. Eng., Rolla, MO 65409-0340 USA

8:30 AM
Microstructure Evolution During Friction Stir Processing of an As-Cast Nickel Aluminum Bronze Material: *Terry R. McNelley*[1]; Asunta M. Cuevas[1]; Douglas L. Swisher[1]; [1]Naval Postgraduate School, Mechl. Eng., 700 Dyer Rd., Monterey, CA 93943-5146 USA

The evolution of microstructure during friction stir processing (FSP) of an as-cast Nickel Aluminum Bronze material (Cu-9Al-5Ni-4Fe), which is widely utilized for cast components in marine applications, has been evaluated by orientation-imaging microscopy (OIM) as well as other microanalysis methods. FSP is a novel method for surface modification of deformable metals by severe plastic deformation of the surface layers of the material using a specially designed, rotating tool. The deformation is accompanied by adiabatic heating and so the use of FSP on a cast metal allows the microstructure of near-surface regions to be converted from a cast to a wrought and annealed condition. The process results in large local strains (~10) and pronounced strain, strain rate and temperature gradients, with corresponding microstructure and property gradients that depend on material, tool and processing conditions. At the boundary between base metal and the thermo-mechanically affected zone (TMAZ) there is a characteristic region of grain elongation and lattice rotation. A {111} tends to align with the plane of shear at this boundary. Further on inside the TMAZ, there are regions of grain refinement (~5mm grain/subgrain size) accompanied by microstructural homogenization, followed by regions characterized by recrystallization and grain growth at locations still closer to the centerline of the TMAZ. These regions will be described and the extent and location of each will be related to the tool geometry and FSP conditions.

8:55 AM

Friction Stir Processing for Microstructural Modifications of Aluminum Casting: Z. Y. Ma[1]; *R. S. Mishra*[1]; M. W. Mahoney[2]; [1]University of Missouri, Dept. of Metallurgl. Eng., 218 McNutt Hall, Rolla, MO 65409 USA; [2]Rockwell Scientific, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA

Friction Stir Processing (FSP) can be used to locally tailor the microstructure for enhancement of a specific set of properties. FSP has been applied to cast aluminum plates to modify the microstructure for enhancement of mechanical properties. The influence of FSP parameters on microstructural changes and microstructure-mechanical property correlations will be presented. FSP leads to homogeneous microstructure and eliminates porosity completely. This results in significant enhancement of mechanical properties. This concept can be used to locally enhance the properties of castings.

9:20 AM

Using Grain Growth as an In-Situ Flow Marker During Friction Stir Welding: *William J. Arbegast*[1]; [1]South Dakota School of Mines and Technology, Adv. Matls. Procg. Ctr., 501 E. Saint Joseph St., Rapid City, SD 57701 USA

Friction Stir Welds in aluminum alloys have shown a tendency to undergo abnormal grain growth when subjected to high temperature thermal treatments after FSW processing. This can result in significant reduction in formability during subsequent cold and hot working operations. Experiments were conducted to evaluate the effect of time and temperature on this abnormal grain growth behavior. One Autogenous 2195 FSW and two Hybrid (4043 and 2319 Filler) FSW were prepared and subjected to temperatures ranging from 750°F to 1050°F for various times. Several distinct zones of abnormal grain growth were observed with the degree of growth a function of exposure time and temperature. The FSW nugget zone composition also had an effect on the rate of growth, but did not seem to influence the distinct origination locations. The results of these experiments are presented with discussions regarding the cause of the distinct origination points of the abnormal grain growth.

9:45 AM

Over-Stirring Hole Defects During Thermomechanical Stir Processing: *Saptarshi Mandal*[1]; Keith M. Williamson[1]; Gene J.-W. Hou[1]; [1]Old Dominion University, Dept. of Mechl. Eng., Rm. 238, Kaufman Hall, Norfolk, VA 23529 USA

This paper investigates the critical size of a hole-defect that can be resolved by a stir tool without compromising the mechanical properties of the weld. Hole-defects may occur during tool breakage or from solidification impurities in a work-piece. Results from these experiments show that hole-defects up to 12.5% of the nib diameter are virtually invisible to the thermomechanical stir process. This confirms the robustness of the stir process and suggests that an empirical relationship exists between joint fit-up and the diameter of the nib. These experiments were carried out on lap welds in Aluminum 1100 and 3000 series alloys by ìover-stirringî machined holes of various diameters. The study also determined that the critical hole depth was also a function of nib geometry; however, the full effect of the hole length is more difficult to quantify. Similar results were obtained, when the same experiment was carried out on defective stir welds with cracks of the same scale as the machined holes.

10:10 AM Break

10:20 AM

Effect of Microstructural Evolution on Mechanical and Corrosion Behavior of Friction Stir-Processed Aluminum Alloys: *P. R. Subramanian*[1]; Paul Sudkamp[2]; Alpesh K. Shukla[3]; Lisa M. Young[1]; David P. Mika[1]; Paul L. Dupree[1]; Nirm V. Nirmalan[1]; [1]General Electric, Global Rsrch. Ctr., PO Box 8, Schenectady, NY 12301 USA; [2]General Electric Aircraft Engines, 1 Neumann Way, Cincinnati, OH 45215 USA; [3]Rensselaer Polytechnic Institute, Matls. Rsrch. Ctr., 110 8th St., Troy, NY 12180 USA

Friction stir welding (FSW) is an enabling solid-state joining process for fabrication of low-cost lightweight structures. The microstructural evolution in FSW-processed alloys is closely connected to the local thermomechanical cycles imposed through the combination of frictional heating and mechanical working by the tool material. The mechanical and corrosion properties of the resultant joints, in turn, are intimately linked to the evolved microstructures. In an effort to understand these relationships, FSW simulations were conducted on monolithic aluminum alloy 2195 plates. Thermal fields produced during FSW were determined using infrared (IR) imaging and embedded thermocouples. This presentation will cover the link between the thermal fields and the resultant microstructural evolution in the various FSW zones. The influence of these microstructural features on tensile, fatigue, and corrosion behavior will be highlighted. (This work was sponsored (in part) by the Air Force Office of Scientific Research, USAF, under grant number F49620-01-1-0300).

10:45 AM

The Effect of Friction Stir Processing (FSP) on the Spatial Heterogeneity of Discontinuously-Reinforced Aluminum (DRA) Microstructures: *Jonathan E. Spowart*[1]; Zong-Yi Ma[2]; Rajiv S. Mishra[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]University of Missouri-Rolla, Dept. of Metallurgl. Eng., Rolla, MO 65409 USA

In order to introduce different levels of microstructural heterogeneity into an experimental DRA material, three 6061/SiC/25p DRA extrusions were produced, using established P/M techniques. Each contained F-600 grade SiC particles (median diameter, d_{50} = 13.4 µm) however, the median matrix particle size was varied in a controlled manner, by careful screening of the 6061-Al powder stock. The range of matrix particle sizes that were chosen (26.4 µm, 42.0 µm and 108.6 µm) introduced increasing levels of spatial heterogeneity into the DRA microstructures, as quantified using the Multi-Scalar Analysis of Area Fractions (MSAAF) technique. The DRA materials were further processed, using FSP, in order to ascertain the effect of FSP on the homogeneity of the as-extruded microstructures. It was found that FSP introduces a significant amount of solid-state mixing which reduces the amount of microstructural spatial heterogeneity, even in the worst DRA specimens. In addition, mini-tensile tests were carried out on the as-extruded and FSP DRA materials, and these results are presented in the context of the effects of microstructural heterogeneity on tensile mechanical properties.

11:10 AM

Finite Element Simulation of Plane-Strain Thick Plate Bending of Friction Stir Processed 2519 Aluminum: *Michael P. Miles*[1]; Tracy W. Nelson[1]; Murray W. Mahoney[2]; Rajiv S. Mishra[3]; [1]Brigham Young University, Eng. & Tech., 265 CTB, Provo, UT 846002 USA; [2]Rockwell Scientific, A-25, Thousand Oaks, CA 91360 USA; [3]University of Missouri-Rolla, Dept. of Metallurgl. Eng., B37 McNutt Hall, Rolla, MO 65409-0340 USA

Friction stir processing (FSP) is used to modify surface microstructures to enhance the ability to bend thick plate 2519-T8 aluminum. Plate, 25.4 mm thick and 50.8 mm wide, is friction stir processed across the pre-tensile surface 6.3 mm deep. The plates are then bent at 200C, still below the precipitate dissolution temperature, over a punch with radius 38.1 mm into a v-shaped die to progressively greater angles, until the outer surface of the material begins to crack. Parent metal bending results are compared to material with the surface microstructure modified by FSP. Experimental and predicted results are compared with the plate bending process modeled using an elasto-viscoplastic finite element program. The mesh is divided into two zones, i.e., one zone for the unaffected parent material and another zone for the FSP material. The purpose of this initial study is demonstrate the enhanced formability associated with FSP and to validate the modelís accuracy to predict strain for a given bend angle.

11:35 AM

Fatigue Life Improvement of an Aluminum Alloy FSW with Low Plasticity Burnishing: *N. Jayaraman*[1]; Paul S. Prevey[1]; Murray Mahoney[2]; [1]Lambda Research, 5521 Fair Ln., Cincinnati, OH 45227 USA; [2]Rockwell Science Center, 1049 Camino dos Rios, Thousand Oaks, CA 91360 USA

Friction stir welding provides a new technology for solid state joining a wide variety of aluminum alloys that cannot be joined with conventional fusion welds. However, recent work has shown that significant tensile residual stresses are developed in the stirred region with local tension maxima at the transition between the stir and heat-affected zones. Residual tension at the edges of the stir zone has been associated with stress corrosion cracking and corrosion fatigue crack initiation. This fatigue debit has been overcome using low plasticity burnishing (LPB) to introduce a deep surface layer of compressive residual stress. LPB processing after friction stir welding has increased the high cycle fatigue endurance of aluminum alloy FSW by 80%. However, the LPB processing parameters have not yet been optimized to produce the maximum achievable fatigue life. A linear elastic fracture mechanics approach is applied to calculate the fatigue crack growth rates and fatigue lives of friction stir welded 2219-T8751 aluminum with initiation from salt fog pitting. The analysis is performed with and without the including of the deep compressive residual stresses produced by low plasticity burnishing (LPB). Calculated fatigue lives are compared to fatigue data developed in four-point bending at R=0.2 following 100 hr. salt fog pitting corrosion. The results indicate that the improved fatigue life achieved with LPB after friction stir welding can be explained by retardation of crack initiation and growth in the deep compressive layer produced by LPB. The fatigue crack growth analysis provides a theoretical basis for understanding the improved fatigue life realized with LPB and for estimating the residual stress distribution that will provide the highest achievable fatigue strength.

High Temperature Alloys: Processing for Properties: Alternate Materials

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee
Program Organizers: Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA; Jacqui B. Wahl, Cannon-Muskegon Corporation, Muskegon, MI 49443-0506 USA

Thursday AM Room: 13
March 6, 2003 Location: San Diego Convention Center

Session Chair: Gerhard E. Fuchs, University of Florida, Dept. of Matls. Sci. & Eng., Gainesville, FL 32611-6400 USA

8:30 AM
Processing, Characterization and Properties of Laser Deposited Compositionally Graded Titanium Alloys: *Peter C. Collins*[1]; Rajarshi Banerjee[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

An upcoming technology for laser deposition of full-density near-net shape components from a powder feedstock is the Laser Engineered Net Shape (LENSô) process. One of the benefits of the LENSô process is its ability to re-create a designed compositional gradient within a single component, and thus fabricate functionally graded materials. Thus, using this processing technique coupled with concepts of combinatorial materials science, it is possible, to explore the functional relationships between microstructure and properties in a variety of alloys of metallurgical importance such as titanium alloys. This study focuses on the processing and characterization designed graded titanium alloys. These composition gradients have been designed around commercially relevant compositions. Additionally, the change in properties will be addressed. Finally, it will be demonstrated that while simple thermodynamic and kinetic models can be used to predict aspects of the microstructural development, a more detailed understanding of competing phase transformations in these titanium alloy systems is required in order to accurately predict the properties.

8:50 AM
Reaction Forging of Iron AluminidesñA Lower Energy Approach to Processing: J. Rodriguez[1]; J. Wall[1]; S. Moussa[1]; *K. Morsi*[1]; [1]University of Missouri, Mechl. & Aeros. Eng., E3411 Eng. Bldg. E., Columbia, MO 65211 USA

The work presents a new low-energy forging approach which can be used to process a range of important aluminide intermetallics and their composites (e.g. titanium aluminides, nickel aluminides, iron aluminides and niobium aluminides). Reaction forging is combines synthesis (RS) with traditional forging technology to process intermetallics with minimal external energy inputs. The effect of varying processing parameters including green density, strain, and forging temperature on the developed microstructure and hardness is presented. Results confirm the feasibility of this new approach to forge iron aluminides at operating temperatures significantly lower than conventionally used.

9:10 AM
Thermal Conductivity of Uniaxially Cold-Pressed Fe2Mo Powders: Seshadri Seetharaman[1]; [1]Royal Institute of Technology, Matls. Sci. & Eng., Brinellvagen 23, Stockholm SE-100 44 Sweden

Fe2Mo intermetallic powders have been produced by H2 gas reduction of Fe2MoO4 at 973 and 1173K to obtain powders having two different average grain sizes. The powders have been pressed uniaxially between 500 and 2000MPa to obtain pellets having different porosities. The thermal diffusivity of the pellets having a relative density (1-porosity) between 0.515 and 0.710 has been measured at room temperature using the laser flash technique. An identical relationship has been found between the thermal diffusivity and the relative density regardless of average grain sizes. In order to understand the relationship betweeen the thermal conductivity and the relative density, a 5 resistor 2-D Ohmís law model has been formulated. It was found that the 5 resistor 2-D Ohmís law model successfully explains the experimental results in both the numerical and conceptual sense.

9:30 AM
Fabrication of TiC-(Al,Ni) Cermets by Reaction Assisted Infiltration for Application at Elevated Temperature: *Naum Frage*[1]; Hayim Prigogine[1]; Moshe P. Dariel[1]; [1]Ben-Gurion University, Dept. Matls. Eng., Beer-Sheva 85103 Israel

TiC based cermets with an intermetallic matrix (Ni3Al or Al3Ni) are known for their physical properties and wear resistance at elevated temperatures. The fabrication of such cermets by free infiltration with a liquid intermetallic phase requires temperatures above 1350¢™C. The aim of the present work was to determine ways for reducing the fabrication temperature by reaction-assisted infiltration. The preforms for infiltration were compacted from TiC and Ni powder mixtures (10, 15, 20wt% Ni) and sintered to different relative densities. The sintered preforms were infiltrated with liquid Al at 1150-1200¢™C under vacuum 10-4 torr. The interaction between the Ni particles and liquid Al leads to the intermatallic phase formation in the course of the infiltration stage. Several cermets with different compositions were fabricated and characterized. The mechanical properties of the cermets of various compositions will be discussed and related to their microstructure.

9:50 AM Break

10:10 AM
Effects of Fine a2-Ti3Al Precipitates on Quasi-static and Dynamic Deformation Behavior in Ti-6Al-4V Alloy: *Dong Geun Lee*[1]; Sung Hak Lee[1]; Chong Soo Lee[1]; [1]Pohang University of Science & Technology, Matls. Sci. & Eng., San 31, Hyoja-dong,, Nam-gu, Pohang, Kyungbuk 790-784 S. Korea

The effects of Ti3Al(·2) phases precipitation in · phase on the tensile and dynamic fracture characteristics were investigated in this study. Overaging heat treatments can precipitate the nano sized Ti3Al phases inside · phase of Widmanstatten and equiaxed. The important microstructural parameters of two microstructures were analyzed and compared those ëbeforeí with those ëafterí ·2 phases precipitation. Dynamic torsional tests were conducted using a torsional Kolsky bar for Widmanst tten and equiaxed microstructures precipitated by ·2 phases. And then the test data were compared with those of the microstructures without ·2 phases. By ·2 phases precipitation, critical dynamic fracture toughness(Ec) and the resistance to the dynamic shear fracture were increased. The possibility of the adiabatic shear band formation was analyzed from the energy and the difference of the void initiation behavior, which showed the increasing the resistance of dynamic fracture and adiabatic shear band formation.

Hot Deformation of Aluminum Alloys: Processing, Structure and Property - III

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Zhe Jin, Alcoa Technical Center, Thermomechanical Processing and Alloy Development, Alcoa Center, PA 15069 USA; Armand J. Beaudoin, University of Illinois at Urbana-Champaign, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA; Balasubramaniam Radhakrishnan, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6359 USA

Thursday AM Room: 6E
March 6, 2003 Location: San Diego Convention Center

Session Chairs: Thomas R. Bieler, Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 44824-1226 USA; Kenji H. Higashi, Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Sakai, Osaka 599-8531 Japan; Gorti B. Sarma, Oak Ridge National Laboratory, Compu. Sci. & Math. Div., Oak Ridge, TN 37831-6359 USA

8:30 AM
Lateral Spread, Temperature and Microstructure Evolution in Hot Flat Rolling of Aluminium Alloys: *Xinjian Duan*[1]; Terry

Sheppard[1]; [1]Bournemouth University, Sch. of Design, Eng. & Compu., 12 Christchurch Rd., Bournemouth, BH1 3NA England

Hot breakdown rolling is a complicated thermomechanical process. There is shape change, temperature variation and microstructural evolution. These three factors affect each other. In this communication, a three-dimensional FEM program, FORGE3®, is used to simulate a four-pass laboratory rolling pass schedule. The computed histories of temperature and subgrain size fit well with the measurements taken from the literature. The temperature change during water quenching is also simulated. The computed width variations agree with those predictions from an empirical formula which was proposed by the present authors. The prediction of recrystallisation is also conducted for this pass schedule by the use of JKAK equation. A new approach is proposed on the calculation of the Zener Hollomon parameter. The discussion focuses on the determination of friction coefficient and the heat transfer coefficient.

8:50 AM

The Effect of Dislocation Structure on Precipitation Behavior in Al-Mg and Al-Mg-Si Alloys: *Reza Shahbazian Yassar*[1]; David P. Field[1]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Spokane St., PO Box 2920, Pullman, WA 99164 USA

The precipitation behavior of a heat treatable Al-Mg-Si alloy (AA6022) and a non-heat treatable Al-Mg alloy (AA5657) were investigated by DSC, SEM and TEM. Selected samples were subjected to deformation before aging and the dislocation structures were characterized. The effect of deformation and aging treatment on precipitation kinetics and precipitate morphology was studied. Also the correlation between the DSC thermal events and SEM-TEM results is discussed.

9:10 AM

Microstructural Control During Hot Deformation Processing of Aluminum 7075 with Rapidly Engineered Dies: *Deepanker Mathur*[1]; *Kuldeep Agarwal*[1]; *Rajiv Shivpuri*[1]; [1]The Ohio State University, Dept. of Industl. & Sys. Eng., 1971 Neil Ave., 210 Baker Sys., Columbus, OH 43210 USA

The controlling driver in the aerospace industry is the manufacturing cost reduction for all components used to fabricate airframe structures, either for commercial or military purposes. The microstructure, texture and mechanical properties of precipitation hardenable Aluminum alloys undergo dynamic changes during thermomechanical processing (TMP). Hot forging of Al 7075 with dies made of stainless steel and bronze mixture with conformal cooling lines, produces a recrystallised microstructure. These dies are manufactured from innovative 3-D printing technology. Changing process conditions such as temperatures, speed of deformation, pressure loads gives flexibility in controlling the microstructure. Tests for determination of heat transfer coefficient are carried out on this material system. Scaled down forging tests representing rib web structures are performed on aluminum 7075 samples with these engineered dies and various cooling rates to study the influence of heat transfer on the microstructural evolution and properties in such a deformation.

9:30 AM

Hot Fracture Behavior of Al-Mg Alloy During Rolling: *Paul T. Wang*[1]; Zhe Jin[1]; [1]Alcoa Inc., Process Tech., Alcoa Techl. Ctr., Alcoa Ctr., PA 15668 USA

Al-Mg alloys were tested under tension and torsion conditions, and rolled at a lab-scale reversing mill to determine hot fracture behavior under various temperatures and strain rates. Two fracture modes were observed, ductile fracture and hot shortness. The corresponding surface morphology representing these modes is analyzed at a local level to reveal potential fracture mechanisms. A fracture criterion applicable to hot deformation is proposed to account for these two fracture modes. Its applications to fracture of free surface during hot rolling are discussed.

9:50 AM

Deformation and Failure Mechanisms in Commercial AA5083 Materials: *Mary-Anne Kulas*[1]; Paul E. Krajewski[2]; Terry R. McNelley[3]; Eric M. Taleff[4]; [1]The University of Texas at Austin, Matls. Sci. & Eng. Prog., MC C2201, Austin, TX 78712 USA; [2]General Motors Corporation, R&D Ctr., 30500 Mound Rd., Warren, MI 48090 USA; [3]Naval Postgraduate School, Dept. of Mechl. Eng., 700 Dyer Rd., Monterey, CA 03043 USA; [4]The University of Texas at Austin, Dept. of Mechl. Eng., MC C2200, Austin, TX 78712 USA

AA5083 aluminum sheet materials have been studied for the determination of deformation and failure mechanisms over a range of temperatures and strain rates. In addition to relatively standard methods of deformation mechanism determination, the characterization of transient creep behaviors has been used for the identification of deformation mechanisms. Both grain-boundary sliding and solute-drag creep are found to contribute to deformation within the range of temperatures and strain rates evaluated. The mechanisms of tensile failure observed include cavitation-induced ductile fracture and cavitation-induced ductile fracture accelerated by neck growth. The failure mechanisms of samples elongated in tension have been quantitatively evaluated using a new parameter, Q. This parameter provides a measure of the contributions to failure from necking and cavitation and is found to provide insight into the deformation mechanisms active prior to failure in the AA5083 materials.

10:10 AM

Constituent Particle Break-Up During Hot Rolling of AA 5182: *Julian H. Driver*[1]; Antoine Baldacci[1]; Annabelle Bigot[2]; Helmut Klocker[1]; [1]Ecole des Mines de St Etienne, SMS, 158 Cours Fauriel, St. Etienne 42023 France; [2]Pechiney, CRV, 725 rue Aristides Berges, Voreppe 38341 France

The break-up of large (> 100 µm3) intermetallic particles during reversible hot rolling has been investigated by experimental studies of particle evolution during hot deformation and by Finite Element micromechanical analyses. The experiments involve controlled hot plane strain compression tests and industrial hot rolling up to strains of 2 or 3. The particle sizes and shapes have been characterized at various strains both by SEM observation after selective dissolution of the Al matrix and, quantitatively, by 3-D synchrotron X-ray tomography. The two types of constituent particles typical of this alloy (Al-Fe-Mn and Mg2Si) are shown to exhibit different behaviours; e.g. the iron-rich particles break up more quickly but also develop wider size distributions. The influence of particle morphology has also been examined by 3-D FE analysis of the stress/strain distributions around complex, ramiform particles. It is shown that the branches of the particles should break off rapidly (e<<1) from the outer branches inwards. The influence of strain rate on particle break-up is also evaluated.

10:30 AM

Hot Deformation and Fracture of an Al-Zn-Mg-Cu Alloy: Christopher Roberts[1]; *Zhe Jin*[1]; [1]Alcoa Technical Center, Alcoa Ctr., PA 15069 USA

A systematic characterization of hot-torsion-deformed samples of a cast Al-Zn-Mg-Cu aluminum alloy was carried out to understand the hot deformation and hot fracture behavior of the alloy over temperatures ranging from 650F to 900F and strain rates from 0.0025/s to 5/s. The torsion tests were conducted using Gleeble machine and samples were quenched immediately after the completion of deformation. The grain structure after hot deformation was found to vary depending on temperature and strain rate. The soluble constituents existing in the starting microstructure were also seen to change primarily depending on the test temperature. A transition from a transgranular failure to a brittle intergranular failure was observed as temperature increases at high strain rates. At low strain rates, however, the fracture appeared to be dominated by decohesion along the recovered subgrain boundaries, which was observed to be independent upon the temperatures studied.

10:50 AM

Mechanical Behavior of Open-Cellular Al Foams at Ambient and Intermediate Temperatures: *I. Nieves*[1]; F. Arceo[1]; T. G. Nieh[2]; J. C. Earthman[1]; [1]University of California-Irvine, Dept. of Cheml. Eng., Biocheml. Eng. & Matls. Sci., Irvine, CA 92695 USA; [2]Lawrence Livermore National Laboratory, Matls. Sci. Div., Livermore, CA 94551-0808 USA

Several open-cellular 6101-T6 Al foams (Duocell) were tested to failure in tension and compression at 293K, 423K, and 523K. These tests were used to examine the effects of temperature, density and macroporosity on the yield stress and elastic compression modulus and to access the effectiveness of density compensation model in predicting elevated temperature cellular behavior. In general, the yield strength values agreed with model predictions at 293K but were noticeably greater than expected at 423K and 523K. The experimental elastic modulus values also agreed with the room temperature predictions but were considerably lower at elevated temperatures. Both yield and modulus data correlated with the SEM results, which revealed increased ductility at elevated temperatures. These results suggest that density compensation alone is inadequate for predicting the mechanical behavior of cellular materials at intermediate temperatures. Accordingly, the researchers have developed new models whose predictions are in better agreement with the experimental results. These improved predictions are compared with those of the Gibson-Ashby density compensation model and the reasons for the discrepancies explained. The new predictions are further used to elucidate the mechanisms governing cellular behavior at intermediate temperatures.

11:10 AM
Structure Property Relationships of Al-(Sc,Zr) Alloys at 24 and 300°C: *Christian B. Fuller*[1]; David N. Seidman[1]; David C. Dunand[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

A study of the relationship between the microstructural and mechanical properties of Al-(Sc,Zr) alloys containing < 0.01% of $Al_3Sc_{1-X}Zr_X$ precipitates is presented. Classical precipitation-strengthening deformation mechanisms are observed at 24°C, where a transition between precipitate shearing and Orowan looping was calculated to occur at an average precipitate radius of 2-3 nm. For alloys aged above 300°C, Zr additions produce longer lifetimes, but have no effect on the magnitude of the peak hardness. Creep experiments of Al(Sc,Zr) alloys at 300°C yield high stress exponents (27-50), and threshold stress values between 0.06 and 0.33 of the Orowan stress. The climb controlled threshold stress is dependent on the average precipitate radius, which is in agreement with Al(Sc) alloys and a model describing the interaction of dislocations and coherent precipitates. This research is supported by the US Department of Energy.

11:30 AM
The Effect of Microstructure on the Properties of AA5182 Alloy: *Zhengdong Long*[1]; [1]University of Kentucky, Ctr. for Aluminum Tech., 1505 Bull Lea Rd., Lexington, KY 40511 USA

The AA5182 alloy is widely used as beverage can end materials due to its excellent strength and formability. The Al-Mg alloy is traditionally believed as non-heat treatment, solid solution strengthened Aluminum alloy. Some investigations indicated that there are different phases existed in this alloy. In this paper, the phases in this alloy were investigated by SEM and TEM. The different heat treatments were applied to obtain different microstructure, and the effects of these treatments on the mechanical properties of hot band and cold rolled sheet were also further studied. The results indicated that the mechanical properties are greatly affected by microstructure and heat treatments.

11:50 AM
Effect of Forming and Treatment Parameters on Mechanical and Fatigue Properties of Al-Li Extrusions: *K. Sperlink*[1]; V. Ocenasek[2]; [1]AIE CR, Novotneho lavka 5, 116 68 Prague Czech Republic; [2]Research Institute for Metals, Panenske Biezany, 250 70 Odolena Voda Czech Republic

Since 1990, research and development of production and treatment technologies of several types of high strength Al-Li extruded shapes have been carried out in cooperation with Russian partners. The paper presents the results of extrusion experiments performed in real industrial conditions together with the results of mechanical, fatigue and fracture toughness testing. Extruded shapes produced from the alloys Al-Mg-Li (1420), Al-Cu-Mg-Li (1441) and Al-Cu-Li (1450) were prepared and tested. In several cases, the properties of Al-Li extrusions were compared with the properties of commonly used AA2124 and AA7075 extrusions. Very extensive work has been done in the investigation of the heterogeneity and anisotropy of large extrusions used in aircraft constructions. The results of property testing were completed with the results of extrusion texture analysis. Both the effect of extrusion conditions (application of direct or indirect method, extrusion ratio) and the effect of the parameters of thermo-mechanical treatment (small plastic deformation after quenching, temperature and duration of artificial ageing) were evaluated. The optimum conditions of the extrusion and consequent treatment of the studied alloys were determined according to these experimental results.

International Symposium on Gamma Titanium Aluminides: Advanced Alloy Design

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee
Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

Thursday AM — Room: 6F
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: Helmut Clemens, GKSS-Research Centre, Inst. for Matls. Rsrch., Geesthacht D-21502 Germany; David G. Morris, CENIM/CSIC, Physl. Metall., Madrid 28040 Spain

8:30 AM Invited
Recent Developments of TiAl Alloys Towards Improved High-Temperature Capability: *Fritz Appel*[1]; [1]GKSS Research Centre Geesthacht, Inst. for Matls. Rsrch., Max-Planck-Str. 1, Geesthacht D-21502 Germany

Demands for higher strength coupled with good oxidation resistance have led to the development of a new family of gamma -TiAl alloys with the base-line composition (at.%) Ti-45Al-(5-10)Nb+X, with X designating modest amounts of several other metallic and nonmetallic elements. Experimental data concerning strength, creep resistance and defect structures of such alloys have been analysed to validate their potential for engineering applications. Under high-temperature exposure, microstructural changes are perhaps more likely to occur in this more complex alloy system because quaternary or higher elements are likely to modify the various phase stabilities. To overcome this problem requires a tight optimisation of alloy composition and processing conditions. Engineering alloys can be identified that exhibit excellent creep and oxidation resistance at 700-800∞C and that are capable of carrying tensile stresses of 1000 MPa at room temperature combined with plastic elongations of 1-2%.

9:00 AM
Designing Gamma TiAl Alloys for Enhanced High-Temperature Performance: *Young-Won Kim*[1]; [1]UES, Inc., Matls. & Processes, 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Gamma TiAl alloys are emerging as a pervasive structural materials technology. For the last several years, ever-increasing need of higher temperature materials has resulted in worldwide efforts in the development of such gamma alloys. The main thrust was to enhance resistance to high cycle fatigue deformation and failure, creep deformation and oxidation, and much of this has been accomplished through the refinement of lamellar structures/grains, microalloying, chemistry modification, process development, and/or their combinations. These improvements have led to the introduction of a series of gamma alloys, which are to be used at temperatures up to 800∞C under application stress conditions. Another alloys having higher-temperature capability are expected to emerge from an extended effort in identifying more stable dispersoids and more oxidation-resistant alloying additions. Technological challenges ahead are enormous, however, and they are: fixing a few engineering alloy compositions, applying them in various application-specific product forms, and developing industry scale, cost-effective processing practice. This paper discusses the status of current alloys, advances made in wrought processing, and the design methodology and future outlook of emerging high-temperature alloys.

9:30 AM Invited
A Potential γTiAl-Deduced High Temperature Intermetallic Alloy Based on a New Intermetallic Compound γ1 in TiAl+Nb System: *Guoliang Chen*[1]; Xiaodong Ni[1]; Jingwen Xu[1]; Yanli Wang[1]; Xiping Song[1]; [1]University of Science & Technology-Beijing, State Key Lab. for Adv. Metals & Matls., Beijing 100083 China

Nb atom in TiAl is proved to be substitutional atom of Ti and preferentially occupies on the Ti sublattice by experimental and theoretical methods such as ALCHEMI technique. For the case of low Nb content the site occupation of Nb atoms is random in the Ti sublattice. As increasing Nb content the distribution of Nb atoms in the sublattice become ordering. This is a continuous ordering process occurred in the

TiAl + Nb system with increasing Nb and Al contents. The development of the continuously ordering finally lead to form a new ternary compound g1. The molecular formula of g1 phase was determined to be Ti4Nb3Al9. The unit cell of g1 phase contains 16 atoms and is 4 times of the g-TiAl phase with L1 0 structure. The relationship of lattice parameters between g and g1 phase is: ag1= ÷2 ag cg1=2cg The typical atom occupation in the unit cell as well as the reciprocal lattice of g1 phase has been shown in the paper. The x-ray diffraction pattern of stoichimetrical g1 phase, Ti4Nb3Al9, is shown in the paper too. The space group of the lattice is P4/mmm. Alloy based on the g1 phase contains in the g1 and h-(NbTi)Al3 two phases or in the g, g1and h-(NbTi)Al3 three phase region exhibit attractive high temperature mechanical properties. As an example, the high temperature compressive yield stress in air reaches 1100 C ~700 MPa at strain rate ?=10-2/s and 200 MPa at strain rate ?=10-5/s.

10:00 AM Invited

Microstructure Control Using β-Ti Phase for Wrought Gamma TiAl Based Alloys: *Masao Takeyama*[1]; Satoru Kobayashi[1]; Takashi Matsuo[1]; [1]Tokyo Institute of Technology, Dept. Metall. & Ceram. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Ti-Al-M ternary systems (M: β stabilizer for Ti) exhibit an unique composition range where thermodynamically stable β phase exists at higher and lower temperatures but not in between: β+α->α->β+γ. The presence of β phase at higher temperatures is useful for hot workability, whereas the β phase formed at low temperatures is effective in toughening the materials since the transformation of α->β+γ can produce fully lamellar structure having fine β particles within lamellae. Thus, the microstructure control involving β-Ti phase in this composition range makes it possible to develop a new class of wrought gamma alloys to be used at around 900 K. In this study, the alloying effects on the phase equilibria and kinetics of microstructure evolution along the pathway (α->β+γ) in ternary systems have been presented. The knowledge is extended to quaternary systems, and a design concept for development of multi-component wrought gamma alloys will be discussed.

10:30 AM Invited

High Temperature Properties of TiAl Alloys Produced by Elemental Powder Metallurgy: Y. Wu[1]; *S. K. Hwang*[1]; S. W. Nam[2]; N. J. Kim[3]; [1]Inha University, Matls. Sci. & Eng., 253 Yonghyun-Dong, Nam Gu, Incheon 402-751 Korea; [2]KAIST, Daejeon 305-701 Korea; [3]CAAM, POSTECH, Pohang 790-784 Korea

Through the fundamental research works in the last decade, it has been realized that elemental powder metallurgy (EPM) is a viable approach to produce sound TiAl alloys of dependable quality. Alloy design efforts resulted in a variety of chemical compositions such as Ti-46.6Al-1.4Mn-2Mo with assorted minor additions of C or Y. Fully lamellar microstructures with extremely fine lamellar thickness could be obtained and were shown to have about 3% in tensile elongation and close to 700MPa in tensile yield strength at room temperature. Furthermore, in high temperature air tests, the alloys exhibited a rather low secondary creep rate as well as a good oxidation resistance at 800?aC. The attractive properties have been attributed to the fine-scale lamellar microstructure, precipitation hardening and the formation of a surface oxide phase impervious to oxygen penetration.

11:00 AM Invited

Processing, Microstructure and Mechanical Properties of Directionally Solidified TiAl Based Alloy Reinforced by Al2O3 Particles: *Juraj Lapin*[1]; Mohamed Nazmy[2]; [1]Slovak Academy of Sciences, Inst. of Matls. & Machine Mechanics, Racianska 75, Bratislava SK-831 02 Slovak Republic; [2]Alstom Power, Haselstrasse 16, Baden 5401 Switzerland

The processing, microstructure and mechanical properties of intermetallic Ti-46Al-2W-0.5Si (at%) alloy directionally solidified (DS) in alumina moulds were studied. After directional solidification the microstructure consisted of regular a2- and g-lamellae, elongated B2 particles, Al2O3 particles and fine Ti5Si3 precipitates. Various volume fractions of Al2O3 particles and mean interlamellar spacings in DS ingots were achieved by variations of the growth rate, reaction time and cooling rate. The ingots with constant volume fraction of Al2O3 particles were prepared at a constant growth rate and various mean interlamellar spacings were achieved by subsequent solution annealing followed by cooling at constant rates. The volume fraction of Al2O3 particles increased with increasing temperature of the melt and reaction time. The mean interlamellar spacing for both DS and heat-treated (HT) ingots decreased with increasing cooling rate. In DS ingots, microhardness, compression strength, yield strength and deformation to fracture increased with increasing cooling rate. In HT ingots, microhardness and yield strength increased and compression strength and deformation to fracture decreased with increasing cooling rate. The yield stress increased with decreasing interlamellar spacing and increasing volume fraction of Al2O3 particles. A simple model including the effect of interlamellar spacing and volume fraction of Al2O3 particles was proposed for the prediction of the yield stress.

11:30 AM Invited

Development of a Hot Forged TiAl Alloy and Manufacturing of Components: *Toshimitsu Tetsui*[1]; [1]Mitsubishi Heavy Industries, Nagasaki R&D, 5-717-1 Fukahori-machi, Nagasaki 851-0392 Japan

In order to enlarge parts size and reduce material cost of gamma TiAl alloy, a new hot forgeable TiAl alloy has been developed. A composition of this alloy is Ti-42Al-5Mn (at%) and a specific gravity of 4, and a microstructure shows fine-grained lamellar + beta + gamma structure. In the manufacturing process a cast ingot is heated to the beta + alpha region, subsequently hot forged. After hot forging heat treatment of near 1200°C is applied for stress relief and softening. Due to the effect of the beta phase that does not appear in ordinary gamma TiAl alloy, the machinability of this alloy is improved and complicated shape can be machined without chipping. In this paper, properties of this alloy such as tensile, fatigue, fracture toughness and creep will be reported and also manufacturing of components such as blades and special structural parts will be demonstrated.

12:00 PM

Microstructure and Mechanical Properties of Extruded Gamma Met PX: *Susan L. Draper*[1]; Gopal Das[2]; Ivan E. Locci[1]; J. D. Whittenberger[1]; H. Kestler[3]; J. L. Smialek[1]; [1]NASA Glenn Research Center, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [2]Pratt & Whitney, 400 Main St., E. Hartford, CT 06108 USA; [3]Plansee AG, Reutte A-6600 Austria

The potential of Gamma Met PX, a high strength alloy based on TNB developed by GKSS, Germany, to be utilized as a compressor blade material is being assessed. The microstructure and mechanical properties of VAR cast Ti-45Al-X(Nb,B,C) (at.%) gamma TiAl hot extruded above the alpha transus temperature were evaluated. Heat treatments of the as-extruded rods have led to the development of a duplex microstructure at 1290∞C and a fully lamellar microstructure at 1340∞C, suggesting that the alpha transus temperature for the alloy is lower than 1340∞C. Both tensile and compression behavior of as-extruded and heat treated specimens were studied in the temperature range of RT to 871∫C. In general, the yield stress, ultimate tensile strength and the elastic modulus decreased with increasing deformation temperature. Compression creep testing between 727 and 1027∫C revealed that the properties were reproducible and predictable; while strengths reached superalloy-like levels at fast strain rates and lower temperatures, def ormation at slower strain rates and/or higher temperature indicated significant weakening. Microstructural evolution during heat treatment, identification of various phases, and deformation mechanisms for tensile, compression and creep processes will be discussed in terms of the current understanding of phase transformation and mechanical behavior in gamma TiAl alloys.

12:20 PM

Processing, Properties, and Microstructure of Gamma-TiAl Alloys with Niobium Ranging from 4 to 10 at. %: *Vinod Kumar Sikka*[1]; Tadeu Carneiro[2]; Edward A. Loria[3]; [1]Oak Ridge National Laboratory, Metals & Ceram., PO Box 2008, Oak Ridge, TN 37831-6083 USA; [2]Reference Metals Company, Inc., 1000 Old Pond Rd., Bridgeville, PA 15017 USA; [3]Consultant, 1828 Taper Dr., Pittsburgh, PA 15241 USA

The γ-TiAl-based alloys have been investigated extensively in literature. However, there is still a need for further development of γ-TiAl-based alloys with a unique combination of low-temperature ductility and high-temperature strength and oxidation resistance. The purpose of this study is to investigate the effect of varying niobium from 4 to 10 at. % for a base 46 at. % Al composition. The alloy compositions investigated are included in Table 1. The alloys for this study were prepared by nonconsumable-arc melting and extruding them to a reduction ratio of 16:1 at a temperature above α-transus of 1350∞C. The α-transus temperature was measured to vary from 1300 to 1315∞C for alloys with 4 to 10 at. % Nb.

12:40 PM

TiAl G4 Alloy: Investment Cast Processing and Mechanical Properties: *Grange Marjolaine*[1]; Thomas Marc[2]; Raviart Jean-Louis[2]; Belaygue Philippe[3]; Recorbet David[4]; [1]Snecma Moteurs, Service YKO, Ctr. de Villaroche, Moissy, Cramayel 77550 France; [2]ONERA, DMMP, 29 Ave. de la Division Leclerc, BP72, Chatillon, Cedex 92322 France; [3]TurbomÈca, DÈpt. MatÈriaux, Bordes, Cedex 64511 France; [4]Centre díEssais AÈronautique de Toulouse, Div. S, 23 ave. Henri Guillaumet, Toulouse, Cedex 31056 France

Over the last years, Snecma Moteurs has devoted extensive research effort in an advanced cast gamma TiAl french program, aimed at optimizing investment cast structure and at demonstrating the manufacturing of near net-shape turbine blades. In an attempt to improve the quality of cast products, a Ti-47Al-1Re-1W-0.2Si (at%) alloy, namely G4, has been used for this program in order to promote beta equiaxed-type solidification instead of the conventional alpha columnar-type solidification. Current status of investment casting process and of microstructure/property relationships of alloy G4 is highlighted in the present paper. Improved mechanical properties and environmental resistance of alloy G4 with respect to conventional near-gamma alloys have been assessed. In contrast to the commonly practiced subtransus heat treatment applied to conventional gamma aluminides to achieve nearly lamellar microstructure, a modified heat treatment condition leading to a duplex microstructure was found to considerably increase the creep resistance and the fatigue behaviour of this alloy.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C. T. Liu: Intermetallics VII–High Temperature Intermetallics II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Seetharama C. Deevi, Philip Morris USA, Research Center, Richmond, VA 23234 USA; Fritz Appel, GKSS Research Centre, Geesthacht D-21502 Germany; Robert W. Cahn, University of Cambridge, Materials Science and Metallurgy, Cambridge CB2 3QZ UK; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Guo Liang Chen, University of Science and Technology-Beijing, State Key Laboratory for Advanced Metals and Materials, Beijing 100083 China; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208-3108 USA; Shuji Hanada, Tohoku University, Sendai 980-8577 Japan; Linda Horton, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6132 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Dong-Liang Lin, Shangai Jiao-Tong University, Shangai 200030 China; T. G. Nieh, Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; Masaharu Yamaguchi, Kyoto University, Department of Materials Science & Engineering, Kyoto 606-8501 Japan

Thursday AM Room: 8
March 6, 2003 Location: San Diego Convention Center

Session Chairs: David P. Pope, University of Pennsylvania, Matls. Sci. & Eng., Philadelphia, PA 19104 USA; Y. Austin Chang, University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., Madison, WI 53706 USA

8:30 AM Invited

Damage Tolerance in Refractory Mo-Mo3Si-Mo5SiB2 Silicides at Temperatures up to 1300∞C: *R. O. Ritchie*[1]; H. Choe[1]; J. H. Schneibel[2]; [1]Lawrence Berkeley National Laboratory, Matls. Scis. Div., Berkeley, CA 94720 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6115 USA

There has been considerable interest of late in multiphase Mo-Si-B refractory alloys for potential higher-temperature advanced engine systems due to their high melting temperatures (> 2000°C) and relatively good oxidation resistance. In this study, we investigate the damage-tolerant properties, i.e., fracture toughness and fatigue-crack propagation behavior, of several such multiphase Mo-Si-B intermetallics. Specifically, the alloys Mo-12Si-8.5B, Mo-16.8Si-8.4B, and Mo-10Nb-12Si-8.5B (at.%), consisting of alpha-Mo, Mo3Si, and Mo5SiB2 (T2) as their primary phases, are examined at temperatures from ambient to 1300°C, with the objective of discerning salient toughening mechanisms. It is found that Mo-12Si-8.5B (at.%) alloy in particular displays relatively high intrinsic (crack-initiation) toughness both at ambient and elevated temperatures, which is attributed to a crack-trapping mechanism at coarse alpha-Mo particles. Moreover, both fracture toughness and fatigue-crack growth properties in this alloy actually improve as temperature increases up to 1300°C. This is principally due to the enhanced ductility of alpha-Mo phase at elevated temperatures, which promotes some degree of extrinsic toughening, specifically crack bridging, which in turn results in rising resistance-curve behavior at 1300°C.

8:50 AM Invited

Iridium Alloys for Electric Generators in Space: *E. P. George*[1]; C. G. McKamey[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6093 USA

Interplanetary spacecraft need onboard electric power that is reliable and stable over many years. Solar cells and fuel cells can be used for near-Earth missions but, for deep space missions, radioisotope thermoelectric generators (RTGs) remain the only viable choice. RTGs work by converting the heat generated during the radioactive decay of plutonia directly to electricity with the help of Si-Ge thermoelectric elements. Most recently, they were used onboard the Cassini mission to Saturn. This talk will describe C. T. Liuís pioneering contributions to the development of the iridium alloy that is currently used to contain the plutonia fuel in RTGs. He made the key discovery that alloying with ëppm levelsí of Th suppresses grain-boundary fracture and dramatically improves the high-temperature impact ductility of iridium. We will discuss the physical mechanisms responsible for the Th effect, including grain refinement and enhanced grain-boundary cohesion. The talk will conclude by describing current research at ORNL that builds on Liuís early work and is aimed at improving the weldability and impact ductility of iridium alloys as well as understanding the effects of trace impurities on mechanical properties. Research sponsored by the Office of Space and Defense Power Systems and the Division of Materials Sciences and Engineering at the Oak Ridge National Laboratory managed by UT-Battelle, LLC, for the US Department of Energy under contract DE-AC05-00OR22725.

9:10 AM

Effects of Nb on the Yield Strength and Ductility of the C11b Structure of MoSi2: *Adel A. Sharif*[1]; Amit Misra[2]; Terence E. Mitchell[2]; [1]California State University, Los Angeles, Col. of Eng., Compu. Sci., & Tech., Dept. of Mechl. Eng., 5151 State Univ. Dr., Los Angeles, CA 90032 USA; [2]Los Alamos National Laboratory, MST-8: Struct. Property Relations, MS G755, Los Alamos, NM 87545 USA

The effects of alloying with 1 at% Nb on the mechanisms of deformation of MoSi2 are investigated from room temperature to 1600°C. Nb alloying lowered the brittle to ductile transition of MoSi2 from about 900°C to room temperature and increased its yield strength at 1600°C by an order of magnitude. The dislocation substructure investigations are utilized to elucidate the source of concurring low-temperature solid solution softening and high-temperature solid solution hardening observed in the presence of Nb in the C11b structure of MoSi2.

9:25 AM Invited

Effects of Microstructure on the High Temperature Mechanical Properties of Al_3Ti-Mo_3Al Two-Phase Intermetallic Alloys: *Seiji Miura*[1]; Hiroyuki Shimamura[1]; Juri Fujinaka[1]; Tetsuo Mohri[1]; [1]Hokkaido University, Matls. Sci. & Eng., Kita 13, Nishi 8, Kita-Ku, Sapporo, Hokkaido 060-8628 Japan

We present the results of the study on the high temperature deformation behavior of a two-phase intermetallic-based alloy composed of the $D0_{22}$-Al_3Ti and A15-Mo_3Al phases with an equi-axed structure or a lamellar structure. It was found that the equi-axed structure formed by an isothermal heat treatment was very stable even after a long-term heat treatment and, moreover, the microstructure was retained even after a heavy deformation at high temperatures. Although the stress exponent is rather lower a steady state deformation was attained, and compressive deformability was found to be extremely high with the equi-axed structure. On the other hand, a lamellar structure was deterious after compression tests. The stress exponent of the specimens with a lamellar structure is rather high, and the resulting microstructure strongly suggested that the deformation was concentrated at boundaries between lamellar colonies.

9:45 AM

Controlling the Thermal Expansion Anisotropy of Mo_5Si_3 and Ti_5Si_3 Silicides: *Joachim H. Schneibel*[1]; Claudia J. Rawn[1]; E. Andrew Payzant[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

The silicides Mo_5Si_3 and Ti_5Si_3 are potential high-temperature structural materials with melting points in excess of 2100°C. Unfortunately, the ratio of their coefficients of thermal expansion in the crystallographic a and c directions, CTE(a)/CTE(c), is very high with values of 2.0 and 2.7, respectively. This pronounced anisotropy results in severe microcracking of polycrystals of these materials. Guided by the ab-initio model of Fu et al. [Phil. Mat. Lett. 80 (2000) 683-690)], ternary and quaternary alloying additions capable of reducing the CTE anisotropy were identified. Thermal expansion coefficients of promising alloys were determined by powder x-ray diffraction using a precisely controlled high-temperature stage. In addition, the site occupation of the ternary alloying additions, which is of crucial importance for interpreting the CTEs, is under investigation. Alloys with anisotropy values as low as 1.16 exhibiting dramatically reduced microcracking were obtained. This work was sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, and the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program, Oak Ridge National Laboratory. ORNL is operated by UT-Battelle, LLC, for the USDOE under Contract No. DE-AC05-00OR22725.

10:00 AM
Multi-Phase Microstructure Design Coupled with Thermodynamic Modeling in the Mo-Si-B-Ti System: *Y. Yang*[1]; Y. A. Chang[1]; [1]University of Wisconsin-Madison, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706-1595 USA

Based on the critically assessed literature information and our own experimental data, all ternary systems in the Mo-Si-B-Ti system have been thermodynamically modeled. A consistent thermodynamic description for the Mo-Si-B-Ti system is thus obtained via the descriptions of the low order systems. A few key quaternary alloys were made to validify the thermodynamic description. The calculations show good agreements with experimental observations. By means of this set of thermodynamic description, a reaction scheme at 1600∞C in metal-rich side is presented, the reaction scheme aims to serve as a guide for multi-phase microstructure design at extremely high temperature structural applications.

10:15 AM Break

10:30 AM Invited
Phase Equilibria in the Ternary Fe-Rh-Ti System: Jozef Balun[1]; Luiz Tadeu Fernandes Eleno[2]; *Gerhard Inden*[1]; Claudio Geraldo Schoen[2]; [1]Max-Planck-Institut f‚r Eisenforschung GmbH, Physl. Metall., Max-Planck-Str. 1, D¸sseldorf D-40237 Germany; [2]Escola Politecnica da Universidade de Sao Paulo, Computatl. Matls. Sci. Lab./Dept. Metall. & Matl. Eng., Av. Prof. Mello Moraes 2463, Sao Paulo-SP CEP 05508-900 Brazil

No information is available about phase equilibria in the ternary Fe-Rh-Ti system. In all three binary subsystems a strong atomic ordering tendency exists between unlike atoms leading to a very stable bcc B2 ordered structure around the equiatomic composition. It is thus expected that a wide B2 phase field should exist in the ternary system with continuous solubility across ternary sections between binary stoichiometric B2 phases. Experiments were carried out at 1273K and 1073K to determine the isothermal sections. Both diffusion couples and individual alloys with compositions within two- or three-phase fields were analysed. The resulting sections show a large ternary miscibility gap. Calculations were performed using the Cluster Variation Method in the irregular tetrahedron approximation in order to model the experimental observation. Pair and tetrahedron interactions were used to calculate prototype diagrams guiding towards a set of interaction parameters which provides a fairly good description of the experimental findings.

10:50 AM Invited
Microstructure and Oxidation Behavior of Mo-Si-B Intermetallic Alloys: Keith J. Leonard[2]; Brian Kowalski[1]; Madan G. Mendiratta[3]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, MS 6116, Oak Ridge, TN 37831-6116 USA; [3]UES, Inc., Air Force Rsrch. Lab., WPAFB, Bldg. 655, Dayton, OH 45433 USA

There is growing interest in refractory intermetallic alloys based on the Mo-Si-B system for very high temperature structural applications. These alloys is characterized by a multiphase microstructure consisting of α-Mo, Mo_3Si and Mo_5SiB_2 (T2) phases. In the present study, thermal effects on microstructure evolution in a Mo-7.44Si-8.51B (at.%) alloy were studied and the microstructures characterized by XRD, SEM, TEM and hardness measurements.. The results indicate that it is possible to exert some control over microstructure and properties by very high temperature heat treatments. Significant changes in volume fraction of α-Mo, Mo_3Si and T2 phases occur at temperatures ≥1700∞C. In addition, the cyclic oxidation behavior in air at temperatures between 800-1100∞C were studied in a three-phase Mo-12Si-12B (at.%) and near-single T2 phase Mo-12.5Si-25B alloy. The evolution of the oxidation process and products during the early, transient period was also studied and the microstructural changes characterized by XRD and SEM. The results indicate that catastrophic oxidation occurs in both alloys at/below 800-900∞C; performance and oxidation protection is better at ≥1000∞C. A porous, non-protective borosilicate/B-SiO_2 layer forms at low temperatures, which permits easy oxygen diffusion and increased weight loss through volatilization of Mo as MoO_3 gas. A stable, dense silica scale forms at/above 1000∞C, which provides protection from oxidation and reduced weight loss. The authors are thankful for financial support of this work by AFOSR (Grant # F49620-00-1-0080, Dr. Craig. S. Hartley, Program Monitor).

11:10 AM
Influence of Alloying Elements on the Mechanical Properties of FeCo-V Alloys: A Review: R. S. Sundar[1]; S. C. Deevi[1]; [1]Chrysalis Technologies, Inc., Rsrch. Ctr., Richmond, VA 23237 USA

Among the soft magnetic materials, ordered intermetallic alloys based on FeCo exhibit highest saturation magnetization, high Curie temperature, good permeability, low magnetic anisotropy and reasonable strength. The recent interest to design a imore electric aircraftî sparked interest in improving the high temperature mechanical properties of FeCo-V alloys while maintaining the excellent magnetic properties. FeCo-2V alloys offer the highest saturaization magnetization but do not meet the more electric aircraft design requirements of the yield strength and creep resistance. The aim of this paper is to review the influence of alloying elements and select the best possible alloying elements to enhance the strength and creep resistance without sacrificing the electrical and magnetic property requirements. In addition, we review the physical metallurgy of FeCo-V alloys and suggest the strengthening mechanisms based on which strength and creep resistance can be improved. We will also present limited experimental data indicating that the design requirements can be met without deteriorating the electrical and magnetic properties of FeCo-2V alloys.

11:25 AM
Microstructure of Nb Substrates Coated with Mo(Si, Al)$_2$-Al_2O_3 Composite and B-Doped Mo_5Si_3 Layers by Spark Plasma Sintering: *Takashi Murakami*[1]; Shinya Sasaki[1]; Kazuhiro Ito[2]; Haruyuki Inui[2]; Masaharu Yamaguchi[2]; [1]National Institute of Advanced Industrial Science and Technology, Trib. Grp., Inst. of Mechl. Sys. Eng., AIST Tsukuba E., 2-1, Namiki 1-Chome, Tsukuba, Ibaraki 305-8564 Japan; [2]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan

Nb alloys have attracted much attention as high temperature structural materials. However, their high-temperature oxidation resistance has yet to be much improved. In this study, Nb substrates were coated with Mo(Si, Al)$_2$-Al_2O_3 composite, B-doped Mo_5Si_3 and NiAl by spark plasma sintering, and microstructure and thermal stability of these coated specimens were examined. When thin Al foil was inserted between Mo(Si, Al)$_2$-60wt% Al_2O_3 powder and Nb substrate before sintering, Nb substrate was found to be coated with a thin Al_2O_3 interlayer and a Mo(Si, Al)$_2$-60wt% Al_2O_3 outlayer without forming any cracks. The Al_2O_3 interlayer is believed to be formed by partial oxidation of Al foil during sintering in a vacuum of 10Pa. Nb substrates can be coated with B-doped Mo_5Si_3 although a thin reactive layer was formed between the substrates and B-Mo_5Si_3. The results of coating Nb substrates with NiAl will be also shown in this presentation.

11:40 AM
Physical and Mechanical Properties of Single Crystalline $Mo_5X_{3+\alpha}$ (X=Si, B, C) and Related Mo Based Multiphase Compounds: *Kazuhiro Ito*[1]; Taisuke Hayashi[1]; Michiaki Kumagai[1]; Keisuke Ihara[1]; Katsushi Tanaka[1]; Masakuni Fujikura[2]; Masaharu Yamaguchi[1]; [1]Kyoto University, Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan; [2]Japan Ultra-High Temperature Materials Research Center, Ltd., 3-1-8 Higashi-cho, Tajimi, Gifu 507-0801 Japan

$Mo_5X_{3+\alpha}$ (X=Si, B, C) intermetallic compounds such as Mo_5SiB_2 ($D8_l$), Mo_5Si_3 ($D8_m$) and Mo_5Si_3C ($D8_8$) have a great potential for ultra-high temperature applications. Anisotropy of the coefficient of thermal expansion for Mo_5SiB_2 (α_c / α_a=1.2-1.6) is significantly reduced from about 2 of other two $Mo_5X_{3+\alpha}$. Thermal conductivity of Mo_5SiB_2 at room temperature is about 0.3W/cmK, which is a quarter lower than that of Mo due to complex structure. Of interest is that Mo_5Si_3C shows relatively lower thermal conductivity (0.098W/cmK) and higher electrical resistivity (150μΩcm). The creep strength of Mo_5SiB_2 is superior to those of $MoSi_2$ and Si_3N_4 based structural ceramics. Direct access to Mo_5SiB_2+ Mo_{ss} eutectic microstructure was succeeded in synthesis using an optical floating zone method at a growth rate of less than 5mm/h. The room temperature fracture toughness of the alloys is on the order of 16 MPa√m and substantially improved from that of the monolithic Mo_5SiB_2.

11:55 AM Concluding Remarks by Organizers

Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging: Interfacial Interactions, Intermetallics & Substrates

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee

Program Organizers: J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Srini Chada, Motorola, Department APTC, Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Jud Ready, MicroCoating Technologies, Atlanta, GA 30341 USA; Jin Yu, KAIST, Center for Electronic Packaging Materials 305-701 Korea

Thursday AM — Room: 15B
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: K.-N. Tu, University of California-Los Angeles, Matls Sci. & Eng., Los Angeles, CA 90095 USA; Ray Fournelle, Marquette University, Matl. Sci. & Eng. Prgm., Milwaukee, WI 53201 USA

8:30 AM Invited

Effect of Intermetallic Compound Layer Development on Mechanical Strength of Lead Free Solder Joints: *Kikuo Kishimoto*[1]; Masaki Omiya[1]; Masazumi Amagai[2]; [1]Tokyo Insitutute of Technology, Dept. of Mechl. & Control Eng., 2-12-1 O-okayama, Meguro-ku, Tokyo 152-8552 Japan; [2]Texas Instrument Japan, Ltd., Pkg. Dvlp., 4260, Kawasaki, Hiji-machi, Oita 879-1595 Japan

A mechanical reliability of the solder joints is a serious concern. Intermetallic compounds are formed during reflow process between solder and electrical pads and gradually grow in service. Due to its brittle manner, the reliability of solder joints are supposed to degenerate. By using the Cu-plates on which Cu or Ni or Ni/Au plating was deposited, the specimens of solder joints were fabricated with Sn-Ag-based lead free solders. After aging it in an isothermal chamber, tensile tests were performed and the relationships between solder joint strength and aging period were considered. From SEM microscope observation and EDX microprobe analysis, the growth and components of the intermetallic compounds layer were examined. To investigate the stress condition on the solder joint interface, the finite element analysis was also carried out.

8:55 AM

Mechanical Properties of Intermetallic Compounds Formed in Pb-Free Solder Joints During Isothermal Aging: H. Rhee[1]; F. Guo[1]; J. P. Lucas[1]; K. N. Subramanian[1]; [1]Michigan State University, Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

The intermetallic compound (IMC) layer that forms during reflow can play pivotal roles on reliability and failure of solder joints irrespective of whether failure occurs within the IMC or in the solder region adjacent to IMC. To understand the role of IMCs in small solder joints, common in microelectronics, their micro-mechanical properties were probed primarily by nano indentation testing (NIT). Fracture and flow properties were determined for interfacial IMCs and constituent IMC particles present in solder in unaged and isothermally-aged conditions in base and composite Pb-free solder alloys. Roles of hardness, modulus and creep property data obtained from such studies on the solder joint reliability will be presented. Project funded by National Science Foundation under grant No. NSF-DMR-0081796.

9:15 AM

Effect of Microelements Addition on the Interfacial Reaction Between Sn-Ag-Cu Solders and Cu Substrate: *Chiang Ming Chuang*[1]; Kwang Lung Lin[1]; [1]National Cheng-Kung University, Dept. of Matls. Sci. & Eng., Tainan 701 Taiwan

Two kinds of Sn-Ag-Cu lead-free solders, Sn-3.5Ag-0.7Cu and Sn-3.5Ag-0.5Cu-0.07Ni-0.01Ge (in wt%), were selected to explore the effect of microelements (Ni and Ge) on the interfacial reaction between solder and Cu substrate. After same time dipped, the thickness of the interfacial intermetallics of the Ni and Ge addition solder is several times as that of the Sn-3.5Ag-0.7Cu solder. Besides, the additional microelements would transfer the feature of interfacial intermetallics from pebble shape to worm shape. However, the negligible difference in XRD data of these two alloys suggests that both interfacial intermetallics have the same crystal structure. The major interfacial intermetallic formed with Sn-3.5Ag-0.7Cu solder is Cu6Sn5, while it is (Cux, Ni1-x)6Sn5 with Sn-3.5Ag-0.5Cu-0.07Ni-0.01Ge. The analytic results of Electron Probe Microanalyzer (EPMA) show that the aggregating Ni into the interfacial intermetallics plays the influential role on the difference of interfacial reaction rate and the morphology of interfacial intermetallics.

9:35 AM

Mechanical Properties of Sn-Based Intermetallics Measured by Nanoindentation: *Richard R. Chromik*[1]; Sarah L. Allen[1]; Michael R. Notis[1]; Rick P. Vinci[1]; [1]Lehigh University, Matls. Sci. & Eng., 5 E. Packer Ave., Bethlehem, PA 18103 USA

In solder joints prepared with Sn-based solders, one finds intermetallic compounds present as distributed phases in the bulk of the solder alloy, and as discrete layers present at the interface between the solder and the metallization or substrate. These intermetallics are typically metal-Sn alloy compounds, such as Ag_3Sn, Cu_6Sn_5, $AuSn_4$, and Ni_3Sn_4. In order to examine the role of intermetallic phases in determining the ultimate mechanical strength and lifetime of a solder joint, the first step is to characterize the mechanical properties of the intermetallics themselves. We report on our study using nanoindentation to characterize elastic and plastic behavior of many common intermetallic alloys. The measured mechanical properties will be used as a database for future finite element modeling (FEM) that takes into account the presence of the intermetallics and the experimentally observed microstructure of solder joints.

9:55 AM

Thickening Kinetics of Interfacial Cu_6Sn_5 and Cu_3Sn Layers During Reaction of Liquid Tin with Solid Copper: *Robert A. Gagliano*[1]; Morris E. Fine[2]; [1]Ondeo Nalco, Metall. Dept., 1 Ondeo Nalco Ctr., Aurora, IL 60563 USA; [2]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Thickening behavior of interfacial η (Cu_6Sn_5) and ε (Cu_3Sn) phase intermetallic layers was investigated in liquid tin/solid copper reaction couples over reaction times from 1 minute to 100 minutes and temperatures from 250°C to 300°C. Scanning electron microscopy (SEM) was used to quantify the interfacial microstructure at each processing condition. The η was always found to develop with a scalloped growth morphology, while the ε always grew as a somewhat undulated planar layer in phase with the ε. The thickness of each phase was quantitatively evaluated from SEM micrographs using imaging software. Thickening kinetics of the η and ε compounds were modeled using a time and temperature dependent empirical power law. From the model, values for the kinetic exponent, rate constant, and activation energy were established for each intermetallic layer. Measured values for the kinetic exponents and activation energies suggest that thickening of the η is controlled by a grain boundary diffusion mechanism, and growth of the ε occurs by simple solid state diffusion.

10:15 AM Break

10:30 AM Invited

Compound Formation for Electroplated Ni and Electroless Ni in the Under Bump Metallurgy with 42Sn-58Bi Solder During Aging: *Jenq Gong Duh*[1]; Bi Lian Young[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan

Ni-based under bump metallurgy (UBM) is of focused interests due to slower reaction rate with Sn-rich solders compared to Cu-based UBM. In this study, several UBM schemes using Ni as the diffusion barrier are investigated. Joints of Sn-58Bi/Au/electroless nickel (EN)/Cu/Al2O3 and Sn-58Bi/Au/electroplated nickel/Cu/Al2O3 were aged at 110∞C and 130∞C for 1~25 days to study the interfacial reaction and microstructural evolution. Sn-Bi solders react with the Ni-based multi-metallization, and form ternary Sn-Ni-Bi IMC during aging at 110∞C. Compositions of ternary IMC were Sn79Ni16Bi5, Sn78Ni16Bi6 and Sn80Ni12Bi8 for Sn-58Bi/Au/Ni-5.5wt%P/Cu, Sn-58Bi/Au/Ni-12wt%P/Cu and Sn-58Bi/Au/Ni/Cu, respectively. Elevated aging at 130∞C accelerates IMC growth rate and results in the formation of (Ni, Cu)3Sn4 and (Cu, Ni)6Sn5 adjacent to the ternary Sn-Ni-Bi IMC for the Sn-58Bi/Au/Ni-12wt%P/Cu and Sn-58Bi/Au/Ni/Cu joints, respectively. Cu contents in the (Cu, Ni)6Sn IMC are six times of those in the (Ni, Cu)3Sn4. Electroplated Ni fails to prevent Cu diffusion toward the Ni/solder interface as compared to EN-based joints. It is more favorable to employ Ni-12wt%P for the Sn-58Bi/Au/EN/Cu joint. Electroless nickel with a higher phosphorous content of 12wt%P is a better effective diffusion barrier during aging. In addition, P enrichment occurs near the interface of EN/solder and the degree of P enrichment is enhanced with the aging time.

10:55 AM
Solid-State Reactions Between the Au/Ni Surface Finish and the SnAgCu Lead-Free Solders with Different Cu Concentrations: L. C. Shiau[1]; Y. L. Lin[1]; W. C. Luo[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 32054 Taiwan

The SnAgCu series of solders are very promising lead-free replacements for the Sn-37Pb solder, especially for reflow soldering applications. For industrial uses, a 0.2% uncertainty in composition is generally considered acceptable. However, our recent study revealed that, during reflow, the Cu concentration had a very strong effect on the reactions between SnAgCu solders and Au/Ni surface finish. With increasing Cu concentration in solder, the reaction product at the interface after reflow changed from a Ni3Sn4-based compound to a Cu6Sn5-based compound. In this study, we would like to extend our earlier study to investigate whether this strong concentration dependency also occurs during the solid-state aging of the solder joints. We aged solder joints at the solid-state at several different temperatures for time as long as 1000 hours. The solder compositions studied include Sn3.5Ag0.2Cu, Sn3.5Ag0.3Cu, Sn3.5Ag0.4Cu, Sn3.5Ag0.5Cu, Sn3.5Ag0.6Cu, Sn3.5Ag0.7Cu, and Sn3.5Ag1.0Cu. Analysis techniques used include optical microscope, SEM, EPMA, and XRD. It turned out that after solid-state aging, the dependence in Cu concentration disappeared. Detailed mechanism will be presented for this interesting phenomenon.

11:15 AM
Study of the Reaction Mechanism Between Electroless Ni-P and Sn and its Effect on the Crystallization of Ni-P: *Yoon Chul Sohn*[1]; Yu Jin[1]; Sung K. Kang[2]; Won Kyoung Choi[2]; Da-Yuan Shih[2]; [1]KAIST, Matls. Sci. & Eng., 373-1 Kusong-dong, Yusong-gu, Taejon 305-701 Korea; [2]IBM T. J. Watson Research Center, PO Box 218, Rt. 134, Yorktown Heights, NY 10598 USA

The reaction mechanism between electroless Ni-P and Sn has been investigated to understand the effects of Sn on the solder reaction-assisted crystallization at low temperatures as well as the self-crystallization of Ni-P at high temperatures. Ni_3Sn_4 starts to form in a solid-state reaction before Sn melts. Heat of reaction for Ni_3Sn_4 is measured during the Ni-P and Sn reaction, 241.2 J/g. It is found that solder reaction not only promotes crystallization at low temperatures by forming Ni_3P in P-rich layer but also facilitates self-crystallization of Ni-P by reducing the transformation temperature and heat of crystallization. The presence of Sn reduces the self-crystallization temperature of Ni-P by about 10°C. The heat of reaction for the self-crystallization decreases with an increased Sn thickness.

11:35 AM
Development of Electroplate Under Bump Metallization for Flip Chip Lead-Free Solder Bumping: *Su-Hyeon Kim*[1]; Jong-Yeon Kim[1]; Jin Yu[1]; [1]Korea Advanced Institute of Science and Technology, Ctr. for Elect. Pkgg. Matls., Matls. Sci. & Eng., 373-1 Guseong-dong, Yuseong-gu, Daejon 305-701 Korea

The recent transition from lead-bearing to lead-free solder bumping in flip chip packages requires the replacement of the conventional under bump metallization (UBM). In the midst of recently suggested UBM schemes, electropated UBM is still commonly used for electroplate solder bumping. However, when we use electroplated thick UBM in the chip side, the stress problem should be taken into account as well as the interfacial reaction with solders. In the present research, Cu/Ni-Cu alloy/Cu multi-layered UBM was newly developed. The Cu bottom layer is designed as a cushion layer to reduce the stress, the Ni-Cu alloy layer acts as a diffusion barrier, and the Cu finish can promote the solderability. We utilized a simple process to deposit both Cu and Ni-Cu alloy layers by electroplating in a single bath. The plated Cu/Ni-Cu alloy/Cu UBMs were characterized in terms of stress, interfacial reaction with lead-free solder, and electroplating character.

11:55 AM
Metallurgical Reaction Between Ni/Cu UBM and Lead-Free Sn-Ag Flip Chip Solder Bump: *Chien Sheng Huang*[1]; Jenq Gong Duh[1]; Yen Ming Chen[2]; [1]National Tsing Hua University, Dept. of Matl. Sci. & Eng., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan; [2]National Chiao Tung University, Dept. of Matls. Sci. & Eng., 1001 Ta Hsueh Rd., Hsinchu 300 Taiwan

Recently, there are several international legislations to ban the use of Pb for the environmental concern. The eutectic Sn-Ag solder is one of strong candidates to replace the conventional Sn-Pb solder due to its excellent mechanical property. In this study, interfacial reaction between eutectic Sn-Ag and Ni/Cu under bump metallization (UBM) was investigated with a joint assembly of 96.5Sn-3.5Ag/Ni/Cu/Ti/Si3N4/Si multiplayer structure. After the first reflow, only one scallop-type (Ni1-x, Cux)3Sn4 IMC was found between solders and Ni. The thickness of IMC was about 1 mm. The concentration of Cu in (Ni1-x, Cux)3Sn4 was around 1.5 at.%. During multiple reflows, both thickness of IMC and Cu content in (Ni1-x, Cux)3Sn4 increased. In comparison, the thickness of (Ni1-x, Cux)3Sn4 formed in the Sn-Pb system remained almost the same despite the numbers of reflow. Correlations between the IMC morphology and Cu diffusion behavior in the joint assembly will be investigated with respect to the microstructural evolution due to interfacial reaction between the solder and the UBM.

Magnesium Technology 2003: Magnesium General Sessions

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, LMD-Magnesium Committee, International Magnesium Association, MPMD-Solidification Committee
Program Organizers: Howard I. Kaplan, US Magnesium LLC, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; John L. Mihelich, Metal Experts International, Winston, GA 30187 USA

Thursday AM — Room: 2
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: Eric Nyberg, Pacific Northwest National Laboratory, Matls. Procg. Grp., Richland, WA 99352 USA; Zi-Kui Liu, Pennsylvania State University, Matls. Sci. & Eng., State College, PA 16082-5006 USA

8:30 AM
New Refractory Lining Concept at Dead Sea Magnesium Electrolysis Cells: *Zeev Goshen*[1]; Edward Anton Snajdr[2]; Peter Nebgen[3]; Manfred Rosch[4]; Eli Aghion[5]; Zeev Rubinowitz[6]; [1]Minerals & Refractories, Ltd., PO Box 106, 43 Haíazmauth St., Yehud 56100 Israel; [2]Vesuvius USA, Bettsville Rsrch. Ctr., 495 Emma St., PO Box 392, Bettsville, OH 44815 USA; [3]IKB GmbH, Rennweg/Klingelswiese 2, Postfach 1417, Andernach D-56604 Germany; [4]Refraline (pty), Ltd., 82 Bell St., Meadowdale Ext. 1, PO Box 8393, Germiston Edenglen 1613 S. Africa; [5]Dead Sea Magnesium, Magnesium Rsrch. Div., Potash House, PO Box 75, Beer Sheva 84100 Israel

Refractory lining in primary magnesium electrolysis cell is exposed to aggressive environment, therefore, needs to be periodically replaced and re-built. This constant refurbishment is costly, time consuming and halts production for the turnaround duration. The project (called ìTAMARî) objective was to completely redesign the cell refractory lining concept, materials, structure, and installation method. During the project, many advanced (but not necessarily expensive) refractory products were tested and selected for better durability. The cell refractory lining structure was re-designed to incorporate mostly pre-fab units, make use of up-to-date monolithic products, and enable quicker installation. Thus, trying to achieve: lower refurbishment costs, longer life expectancy, and shorter cell turnaround (idle) time. ìTAMARî Project took about two years. The international team, headed by Minerals & Refractories, Ltd. from Israel, included Vesuvius USA, IKB - Germany, Dead Sea Magnesium - Israel, and Refraline - South Africa. The project was partially funded by the BIRD Foundation. Recently, a prototype cell was installed at Dead Sea Magnesium and now operating and monitored.

9:00 AM
Explosion Weld Clad for Magnesium Melting Furnace Crucibles: *George A. Young*[1]; John G. Banker[1]; [1]DMC Clad Metal Division, 5405 Spine Rd., Boulder, CO 80301 USA

Large magnesium melting and alloying crucibles, referred to as mag pots, are typically constructed of cast iron or low carbon steel. The steel interior is resistant to attack by the molten magnesium. However, the exterior of the steel pot is subject to high temperature oxidation. Cleanliness and control of iron oxide accumulation is critical for safe operations. Clad pots consisting of an exterior layer of high temperature stainless steel or nickel alloy and a low carbon steel interior provide a number of operational benefits. These include improved safety, increased dimensional stability, and considerably longer service life, especially for gas fired furnaces. The explosion cladding technology has been used for clad pot manufacture since the mid-1980ís. The cladís metallurgical bond is not deleteriously affected by long term operation at high temperatures or the stresses of cyclic temperature exposure typical during mag pot operation. Test data indicate long term stability at up to 1000∞C. The presentation d iscusses the various metal alloys which are being used in magnesium pot applications, their performance attributes and limitations, clad fabrication techniques, and inspection methods. Pot design issues as relates to both performance and economics are discussed.

9:30 AM
Scale-Up of Magnesium New Rheocasting from a Laboratory Level to an Industrial Process: *Werner Fragner*[1]; Christian Peterlechner[1]; Ralph Potzinger[2]; [1]ARC Leichtmetallkompetenzzentrum Ranshofen GmbH, Shaped Parts, AMAG-FVA-Gebaeude, Postfach 26, Ranshofen A-5282 Austria; [2]TCG Unitech AG, Lab. Mgr., Quality Mgr., Steiermaerker Strafle 49, Kirchdorf an der Krems A-4560 Austria

New Rheocasting (NRC) is a novel semi solid casting process for high quality castings. Originally designed for aluminum, laboratory trials on a 350 ton squeeze casting machine gave promise to introduce NRC to the magnesium market. However, scaling up to an industrial 800 ton plant demands consideration of several factors that differ from aluminum: Heat loss and alloy composition affect cycle time and process window. Process parameters such as casting speed, pressure, and temperature influence mechanical properties and microstructure. Varying composition in the Mg-Al-Zn system (AZ alloys) changes the NRC process window, which affects the ideal fraction liquid of the slug, i.e. castability. To provide the cast shop with the optimal casting temperature, a numerical approach was developed based on calculated phase diagrams. With proper machine and process modifications it is shown that Mg-NRC is a feasible way to produce sound Mg components on an industrial scale.

10:00 AM
Advances in Thixomolding Magnesium Part II: *Randy S. Beals*[1]; Steve LeBeau[2]; Yanzhong Wu[1]; Oscar E. Roberto[3]; P. Shashkov[1]; Simon Dewey[4]; [1]DaimlerChrysler Corporation, Matls. Eng. Dept. 5810, 800 Chrysler Dr., CIMS 481-01-41, Auburn Hills, MI 48326 USA; [2]Thixomat, 620 Technology Dr., Ann Arbor, MI 48108 USA; [3]Henkel Surface Technologies, Light Metals Grp./ANOMAG, 32100 Stephenson Hwy., Madison Heights, MI 48071 USA; [4]Keronite, Ltd., PO Box 700, Granta Park, Great Abington, Cambridge CB1 6ZY England

This report is a continuation of a previously published paper regarding ThixomoldingÆ magnesium alloys. The previous report discussed the characteristics of thixomolding magnesium alloys with low and high fraction percent solids. The effect of the amount of prior fraction solid on the physical properties of selected samples are further explored, with the percent solids in both AZ-91D and AM-60 B alloys evaluated over a range from 0% to 20%. The corrosion resistance of test plates that had been exposed to the Anomag and Keronite surface finishing technologies before and after 240 hour salt spray corrosion testing was examined in more detail than previously published. The surface features, cavitation and corrosion mechanics of the tested panels were analyzed and the results are discussed. The corrosion penetration at the interface between the scribe, coating and the material was also investigated. The corrosion behavior of the different coatings are discussed.

10:30 AM Break

10:45 AM
Low Cost SiCp/AZ91 Mg Composites Prepared by RCM: *Shae K. Kim*[1]; Hoon Cho[1]; Hyung-Ho Jo[1]; Young-Jig Kim[2]; [1]KITECH, Adv. Matl. R&D Ctr., 472, KaJwa 4 Dong, Seo-Ku, Incheon 404-254 Korea; [2]Sungkyunkwan University, Dept. of Adv. Matl. Eng., 300, ChunChun-Dong, JangAn-Ku, Suwon, GyeongGi-Do Korea

Magnesium metal matrix composites are gaining increased importance for transport and electronics applications where low inertia is required, with their improved stiffness, wear resistance, and elevated-temperature properties. Although the use of ceramic particle instead of fibrous reinforcements has help to reduce the over-all material cost, however, the processing-related cost should also be considered. This paper describes a novel low-cost molten metal mixing method, RCM (Rotation-Cylinder method), which has been developed for preparing SiC particle reinforced magnesium composites, with the aim of rapid incorporation and homogeneous distribution of reinforcement particles in an ambient atmosphere. RCM claims to significantly reduce the time required for incorporation and particle agglomerations by the U-shaped melt surface with the Rankine vortex. Sound magnesium composites can be produced in conjunction with subsequent investment casting or thixoforming processes. The development background, characteristic vortical motion, and on-going research strategy of RCM will be described.

11:15 AM
A Thermodynamic Database for Magnesium Alloys: *Marie Piche*[2]; Arthur Pelton[2]; Christine Brochu[1]; [1]Noranda, Noranda Tech. Ctr., 240 Hymus, Montreal, Quebec H9R1G5 Canada; [2]…cole Polytechnique de MontrÈal, Matls., MontrÈal H3C 3A7 Canada

A thermodynamic database for the Mg, Al, Mn, Fe, Be system as been developed at the CRCT (Centre for Research in Computational Thermochemistry), a research center based at the …cole Polytechnique de MontrÈal, in collaboration with Noranda Technology Centre for the MÈtallurgie Magnola, Inc. foundry. The goal of this work is to have a better understanding the chemistry of the alloying process of magnesium. The software used to make optimisations of the systems and to built the solutions and compound database is FACTSAGE 5.0. It permits to define the activity of each element of the system in the liquid and the solid phases and to calculate complex chemical equilibrium in a multicomponent system. The binary systems Al-Fe, Al-Mg, Al-Mn, Fe-Mg, Fe-Mn and Mg-Mn are taken from the COST 507 project, a thermochemical database for light metal alloys, and the binary systems with beryllium (Al-Be, Be-Fe, Be-Mg and Be-Mn) are evaluated in this work. The evaluation is made with literature data of phase equilibria, activitie s and thermodynamic properties of magnesium rich end data. For the multicomponent systems, they are interpolated from the binaries and optimised the same way. With the magnesium database, the software FACTSAGE 5.0 can plots phase diagrams, simulates the alloying process of magnesium for temperature between 500°C and 900°C and predicts the phase formation at each step. Magnesium producers can improved alloying method and increased the element recovery by calculating the distribution of the element between the magnesium liquid and the sludge, the heat required to maintain the furnace at constant temperature and the kind of intermediate phases formed during element addition.

11:45 AM
Orientation Dependence of Fatigue Crack Propagation in Magnesium Single Crystals: *Shinji Ando*[1]; Hideki Tonda[1]; [1]Kumamoto University, Dept. of Mechl. Eng. & Matls. Sci., Fac. of Eng., 2-39-1 Kurokami, Kumamoto 860-8555 Japan

The fatigue crack growth behavior of pure magnesium single crystals has been investigated in laboratory air at room temperature. Four type of compact specimens with different notch orientations were prepared from magnesium single crystal. Fatigue crack propagation behavior of each specimens were different related to notch orientation. In the case of A-specimen which notch plane and direction is (1210)[1010], a fatigue crack propagates parallel to notch plane. In B-specimen with (1010)[1210] notch, a fatigue crack propagates along to [1010] which inclined 30° to notch plane. In D-specimen with (1210)[0001] notch, a crack propagated to [0001] at low ΔK. The crack are inclined to [1210] gradually with increasing ΔK. In F-specimen with (0001)[1120] notch, a crack propagates parallel to basal plane with {1012} twin. Fatigue crack growth rate is lowest at B-specimen, and fastest at F-specimen.

Measurement and Interpretation of Internal/ Residual Stresses: SXRD on Composites

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Shaping and Forming Committee
Program Organizers: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Bimal K. Kad, University of California, Ames Laboratory, La Jolla, CA 92093-0085 USA

Thursday AM Room: 17B
March 6, 2003 Location: San Diego Convention Center

Session Chairs: Mike Prime, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Bjorn Clausen, Los Alamos National Laboratory, LANSCE-12, Los Alamos, NM 84545 USA

8:30 AM Invited
The Use of High Energy X-Rays from the Advanced Photon Source to Study Stresses in Materials: *Dean R. Haeffner*[1]; [1]Argonne National Laboratory, APS, 9700 S. Cass Ave., Bldg. 431-A008, Argonne, IL 60439 USA

High-energy, third-generation synchrotrons, such as the Advanced Photon Source (APS), are well suited to produce very high fluxes of x-rays with energies above 40 keV. These so-called ìhigh-energyî x-rays are particularly well suited for studies of internal stresses and texture in materials. The low absorption of high-energy x-rays by most materials allows probing to depths comparable to that done with neutrons, but with much better spatial resolution and with much more beam intensity. The SRI-CAT high-energy x-ray program at the APS 1-ID beamline has been developing optics and experimental techniques for high-energy x-rays since the onset of the APS operations in 1995. Stress studies have been carried out on a variety of systems, including duplex steels, thick coatings, bulk metallic glasses, and composites. Examples from this research will be shown and plans for a future dedicated high-energy beamline (HEX-CAT) at the APS will be presented. Use of the Advanced Photon Source was supported by the US Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

9:00 AM Invited

High Spatial Resolution Stress Measurements Using Synchrotron Based Scanning X-Ray Microdiffraction with White or Monochromatic Beam: *Nobumichi Tamura*[1]; Bryan C. Valek[2]; John C. Bravman[2]; Ralph Spolenak[3]; Wendel A. Caldwell[1]; Rich S. Celestre[1]; Alastair A. MacDowell[1]; Howard A. Padmore[1]; Jim R. Patel[1]; [1]Lawrence Berkeley National Laboratory, MS 2-400, 1 Cyclotron Rd., Berkeley, CA 94720 USA; [2]Stanford University, Dept. Matls. Sci. & Eng., Stanford, CA 94305 USA; [3]Max-Planck-Institut f¸r Metallforschung, Heisenbergstr. 3, Stuttgart D-70569 Germany

Scanning X-ray Microdiffraction (µ-SXRD) combines the use of high brilliance synchrotron sources with the state-of-the-art achromatic X-ray focusing optics and fast large area 2D detector technology. Using either white or monochromatic beams, it allows for orientation and strain/stress mapping of polycrystalline thin films with submicron spatial resolution. As an example of applications of the technique, the evolution of inter and intragranular stresses in micron size individual grains of a polycrystalline Al thin films deposited on Si, was monitored during an in-situ thermal cycling experiment. Strong stress and orientation variations were measured between grains and inside individual grains, which could be explained in terms of yield stress distribution and grain-to-grain interactions. If the µ-SXRD data are averaged over a macroscopic range, results show good agreement with macroscopic texture and stress measurements.

9:30 AM

Multi-Scale Characterization of Deformation Heterogeneities in Mo Single Crystal: *Erica Thea Lilleodden*[1]; Nobumichi Tamura[2]; David Lassila[3]; J. W. Morris[1]; [1]Lawrence Berkeley Laboratory, Matls. Sci., 1 Cyclotron Rd., MS 66-200, Berkeley, CA 94720 USA; [2]Lawrence Berkeley Laboratory, Adv. Light Source, 1 Cyclotron Rd., Berkeley, CA 94720 USA; [3]Lawrence Livermore National Laboratory, Eng., PO 808, L-113, Livermore, CA 94550 USA

High brilliance synchrotron sources used in conjunction with improved focusing optics have furthered the spatial resolution of x-ray diffraction measurements. A beamline at the Advanced Light Source at Lawrence Berkeley Laboratory, which is dedicated to microdiffraction experiments, is used in scanning mode to map out Laue diffraction patterns across the surface of a sample with a lateral resolution of 1 micron. These measurements are then analyzed in order to quantify the distribution of residual stress and strains in the sample. Furthermore, the distribution of dislocations in the sample can be elucidated from such measurements in conjunction with other scales of characterization. Here, we present preliminary findings on the spatial distribution of dislocations in a compression loaded Mo single crystal, and discuss the importance of X-ray microdiffraction in the hierarchy of multi-scale characterization of deformation behavior.

9:50 AM Break

10:05 AM

Microdiffraction Study of the Strains at Domain Walls in BaTiO3: *Robert C. Rogan*[1]; Ersan Ustundag[1]; Geoff Swift[1]; Nobumichi Tamura[1]; [1]Lawrence Berkeley National Laboratory, Adv. Light Source, Berkeley, CA 94720 USA; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA

The local strain fields around ferroelectric 90∫ domain walls in BaTiO3 have been investigated for the first time using the emerging technique of polychromatic scanning X-ray microdiffraction. A tetragonal BaTiO3 single crystal was scanned with a sub-micrometer X-ray beam to collect Laue patterns from regions around an individual domain. These patterns yielded information about orientation relationships across domain boundaries as well as the three-dimensional strain tensor associated with the domain. The results suggest the presence of significant residual elastic strain fields around domains in a nominally stress-free BaTiO3 crystal. The diffraction data will also be compared to and interpreted with micromechanical models.

10:25 AM

X-Ray Stress Analysis of Damage Evolution in Ti-SiC Composites: *Jay C. Hanan*[1]; Ersan Ustundag[1]; Irene J. Beyerlein[2]; Geoffrey A. Swift[1]; Jonathan D. Almer[3]; Ulrich Lienert[3]; Dean R. Haeffner[3]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Theoretl. Div., Los Alamos, NM 87545 USA; [3]Argonne National Laboratory, Adv. Photon Source, Argonne, IL 60439 USA

High energy X-ray microdiffraction was used to determine the elastic lattice strains of both phases in a Ti-SiC composite providing the in-situ load transfer under applied tensile stress at the scale of the microstructure. To understand the damage evolution, the measured strains were compared to those predicted by a modified shear lag model. Comparisons between the model and the data demonstrated the importance of accounting for the matrix axial and shear stiffness, provided an optimal stiffness ratio for load transfer and planar interpretation of the geometry in the composite, showed the matrix within and around the damage zone sustained axial load, and highlighted matrix yielding observed in the composite. It was also shown that an area detector is essential in such a study as it provides multiaxial strain data and helps eliminate the ìgraininessî problem.

10:45 AM

Internal Stresses and Orientation-Changes in Directional Solidification Microstructures: *Bernard Billia*[1]; Joseph Gastaldi[2]; Nathalie Bergeon[1]; Haik Jamgotchian[1]; Henri Nguyen Thi[1]; [1]L2MP, UMR CNRS 6137-University Aix-Marseille 3, Faculte des Sciences Saint-Jerome, Case 142, 13397 Marseille, Cedex 20 France; [2]CRMC2/CNRS, Campus de Luminy Case 913, 13288 Marseille, Cedex 9 France

In the processing of structural materials, all along alloy solidification from its melt internal stresses are generated in the solid by the composition variations that accompany microstructure formation. Directional solidification, which allows independent control of growth parameters, is used as model configuration. By means of in situ white-beam synchrotron X-ray topography, internal strains and stresses, and orientation-changes, created in the process of cellular/dendritic interface formation are followed in real time during directional solidification of thin Al-based binary alloys. With X-ray topography comprehensive information can be obtained by considering different reflecting planes. It follows that: -elastic strains giving specific contrasts precede visible interface corrugation, -cells are stressed due to non-uniform solute concentration in the solid, and -grain polygonisation results from changes in cell-body orientation, either suddenly or in a somewhat coherent manner. Based on mechanical shearing and bending, the phenomena can be analyzed, and modeling proposed.

11:05 AM

Distribution of Residual Microstresses: *Yu. Perlovich*[1]; M. Isaenkova[1]; H. J. Bunge[2]; [1]Moscow Engineering Physics Institute, Kashirskoe shosse 31, Moscow 115409 Russia; [2]TU Clausthal, Clausthal-Zellerfeld 38678 Germany

X-ray diffractometers of last generations allow to combine texture and microstress measurements. By use of the scanning regime or a position-sensitive detector, the X-ray line profile is registered for each point of the texture pole figure, so that additionally generalized pole figures of peak position 2q and line broadening b can be constructed. Though first attempts to obtain these pole figures were undertaken long ago, construction of distributions 2q(y,j) and b(y,j) became a routine procedure only recently, as both measurements and data treatment were completely automated. In the given work systematic studies of residual deformation effects in textured materials resulted in determination of main principles, controlling the above distributions. The distribution of elastic microstrains depending on grain orientation was studied in details as applied to various rolled metal materials (steels, Nb, Mo, Cu, Zr-alloys, Ti, single crystals Ti-Ni) having textures of different types. For the sake of the effective systematization of obtained experimental data, measured values of the Bragg angle 2qhkl(y,j) were recalculated into values of the interplanar spacing dhkl and then - into values [dhkl(y,j) - dav)/dav, that is the relative deviation of interplanar spacing along the direction (y,j) from the average weighted level dav. The average level is admitted to be conditioned by elastic macrostresses (stresses of 1st kind) acting within the irradiated volume, whereas local fluctuations about this level are connected with elastic microstresses (stresses of 2nd kind) of opposite signs. By the definition, residual stresses of the 2nd kind, or elastic microstresses, are equilibrated within a volume of the group of neighbouring grains, which to a first approximation corresponds to the volume participating in X-ray diffraction by the texture measurement. The analysis of experimental data shows, that the actual variant of stress equilibrium in rolled metals is connected with crystallographic orientations of grains and with their position relative to texture maxima and minima as well as to the planes of symmetry of the pole figure. Most often the distribution of elastic microstresses shows a cross-wise character, consisting in alteration of the predominant sign (≤+≤ or ≤-≤) of microstresses, when passing from one quadrant of the pole figure to another. As a result, elastic microstresses of opposite signs prove to be equilibrated about both longitudinal and transverse planes. In cases of simple rolling textures the distribution of residual microstrains acquires some additional features, which seem to be a remarkable manifestation of the tendency to stress equilibrium: - when the rolling texture contains a single component (for example, in the case of the rolled single crystal Ti-Ni), this component breaks in half, that is in two close subcomponents, which differ in signs of the elastic strain along the same direction; - when the rolling texture consists of two crystallographically equivalent components, the equilibrium of elastic microstresses is ensured by the fact, that deformation tensors for their crystallites prove to be mutually opposite; - in the case of the rolling texture with a significant axiality the stretched zones of elastic compression and extension are situated at opposite slopes of texture maxima.

Mercury Management: Remediation and Fundamentals

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizer: Larry Twidwell, Montana Tech of the University of Montana, Metallurgical and Materials Engineering, Butte, MT 59701 USA

Thursday AM — Room: 1B
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: Jerry Downey, Hazen Research Inc., Golden, CO 80403-1848 USA; Charles Gale, Summit Valley Engineering and Equipment, W. Bountiful, UT 84087 USA

8:30 AM Invited
Process Development Strategies for Mercury Remediation: *Jerome P. Downey*[1]; Guy L. Fredrickson[1]; [1]Hazen Research, Inc., 4601 Indiana St., Golden, CO 80403-1848 USA

The technologies developed within the field of extractive metallurgy to recover, concentrate, and purify metals and industrial minerals from ores, have served as the foundation for all of the subsequent environmental remediation, materials recycling, and waste management technologies. This paper discusses the application of classical extractive engineering principles to the development of a thermal process for the recovery of mercury from solid wastes. Process development is presented as a series of logical stages, which begin with a thorough characterization of the site-specific waste, move on to bench and pilot scale testing of the process options, and end with a functional process flowsheet. This paper also presents thermodynamic data on many mercury species of interest to the minerals, power, and chemical industries. These data can be used to model the speciation and recovery of the mercury as a function of various process parameters.

9:00 AM Invited
Selection and Demonstration of a Process for Mercury Remediation in Soils: A Case Study: *Charles W. Kenney*[1]; [1]Hazen Research, Inc., 4601 Indiana St., Golden, CO 80403-1848 USA

Mercury contamination of soil at an old chemical plant surrounded by a residential community and adjacent to a river has provided unique challenges for the current owner. Inorganic and organic mercury compounds accumulated over the past 50 years to levels that require remediation. Hazen Research, Inc. conducted a multi-phased program to characterize contaminants and their occurrences, evaluate various treatment technologies, and demonstrate the selected technology at bench and pilot scales. A combined physical cleaning and chemical treatment was demonstrated to meet treatment goals for the site. This paper discusses the methods of process selection, and results of the bench- and pilot-scale testing.

9:30 AM Invited
Treatment of Mercury-Contaminated Soil, Mine Waste and Sludge Using Silica Micro-Encapsulation: *Amy Anderson*[1]; [1]KEECO, Lynnwood, WA USA

The Silica Micro-Encapsulation (SME) Technology has been utilized on three separate projects focused upon the stabilization of mercury-contaminated materials. At the Mother Lode Mine in Oregon approximately 450 cubic yards of soil were amended with the chemical using a backhoe and nominal amounts of water to promote the reaction. Results collected via EPA Method 1311 TCLP, exhibited leachable mercury concentrations below the treatment goal of 200 parts per billion (ppb); actual concentrations ranged from 0.8 ppb to 2.6 ppb. In a laboratory study jointly sponsored by the EPA Mine Waste Technology Program and Superfund Innovative Technology Evaluation (SITE) Demonstration Program the SME Technology, an inorganic sulfide chemical and a generic phosphate treatment were tested for their ability to treat mine waste from the Sulfur Bank Mercury Mine. Technical and economic performance of the silica-based technology exceeded the two competing technologies, achieving an 88% reduction in total leachable mercury and a greater than 99% reduction in the particulate-associated mercury as compared to the control samples. Costs of treatment were estimated at $18.26 per metric ton. At a former chloralkali plant in British Columbia, dredged lagoon sludge was amended with the SME chemical to reduce leachable mercury concentrations to 0.150 ppm or less, based on EPA Method 1311 TCLP. Results from laboratory and field tests established an optimum chemical dosage rate of 3% chemical by dry weight of sludge. Samples were evaluated during field tests for weight, temperature, mercury vapor and leachable mercury. Results exhibited a 75% reduction in leachable mercury as compared to the control and satisfied the project treatment goals. Operating costs were estimated at $15.00 per metric ton.

10:00 AM Break

10:15 AM Invited
Utilization of an Equilibrium Calculational Program to Demonstrate the Hydrometallurgical Treatment of Mercury Wastes: *Hsin H. Huang*[1]; Larry G. Twidwell[1]; Courtney A. Young[1]; [1]Montana Tech of the University of Montana, Sch. of Mines & Eng., 1300 W. Park, Butte, MT 59701 USA

Equilibrium calculational software programs can be effectively utilized to model and to understand the behavior of mercury. The equilibrium calculation program STABCAL will be demonstrated. The illustrated diagrams include Eh-pH, activity or concentration as a function of pH, Eh or additional ligand concentration. The resulting diagrams and simulations will be presented to demonstrate the fundamentals of geological deposition processes, pyro- and hydrometallurgical and environmental treatments and recovery, transpositon between aqueous and gaseous phases and mercury dispersal in the environment.

10:45 AM Invited
Mercury, Lead and Cadmium Removal from Aqueous Streams Using Silica Polyamine Composites: *Edward Rosenberg*[1]; Robert Fischer[1]; Carolyn Hart[1]; Purity Systems, Inc.[2]; [1]University of Montana, Dept. of Chem., Missoula, MT 59812 USA; [2]3116 Old Pond Rd., Missoula, MT 59802 USA

New composite materials have been developed that are particularly effective for the removal of lead, cadmium and mercury from low levels (i.e. National Sanitary Foundation recommended challenges: 150, 30 and 6 micrograms/L respectively) to the allowable release levels (15, 6 and 2 micrograms/L respectively). The synthesis and physical characteristics of these materials will be described. Data on idle column leaching will be presented and the various methods attempted for regeneration of the columns will be discussed. Conditions for the application of these materials to the recovery and remediation of these metals will be recommended.

11:15 AM
Mercury Spill Response: *Lise Mercier*[2]; *Neal Langerman*[1]; [1]Advanced Chemical Safety, 7563 Convoy Ct., San Diego, CA 92111 USA; [2]EPS Chemicals, Inc., 161-145 Tyee Dr., Pt. Roberts, WA 98281 USA

Mercury use, in thermometers, switches, floating contacts, or other applications, results in spills. These spills pose a long-term toxicity hazard to employees, their families, and the environment. Prompt response and efficient clean-up is essential. Spills from thermometers and switches are generally small and can be efficiently handled by area workers using a MERCONô Spill Kit. As the amount of mercury spilled increases, the knowledge and protection for the responders increase and the use of more sophisticated clean-up and decontamination tools is required. Responders to a large mercury spill must be trained under the OSHA ìHazardous Waste Operations and Emergency Responseî (29 CFR 1910.1209q) standard. In addition to personal protective equipment, responders must have a mercury vacuum, a mercury vapor detector, and an adequate supply of MERCON-Xô. The safe and effective use of these procedures and tools will be illustrated with actual examples.

Microstructural Processes in Irradiated Materials: Fusion Reactor Materials

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

Thursday AM Room: 11A
March 6, 2003 Location: San Diego Convention Center

Session Chairs: David Gelles, Pacific Northwest National Laboratory, Richland, WA 99352 USA; Robin Schaeublin, EPFL, CRPP, Villigen AG 5232 Switzerland

8:30 AM
Irradiated Microstructrures in Fusion Reactor Materials: *Steven J. Zinkle*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA

Microstructural studies are indispensable for determining fundamental physical processes that occur in materials during energetic particle irradiation. The major materials systems of interest for fusion reactor applications include body centered cubic metallic alloys (ferritic/martensitic steels, vanadium alloys, Mo alloys), SiC/SiC and carbon composites, and ceramic insulators. Cu alloys and austenitic stainless steel are also of interest, in particular for near-term proposed next-step burning plasma experiments such as ITER. This presentation will review recent electron microscopy results on irradiated materials, with an emphasis on the fluence and temperature-dependent accumulation of defect clusters, and in-situ and post-deformation observations of irradiated and deformed metals. Intrinsic differences between the defect accumulation behavior of BCC and FCC metals will be highlighted. Recent experimental observations on flow localization due to plastic deformation of quenched and irradiated metals (in particular, twinning and dislocation channeling), and studies of the detailed dislocation-defect cluster interaction mechanisms will be summarized.

9:15 AM
A Comparison of Helium Implanted Iron and EUROFER97: Effects of He Dose: *Morten M. Eldrup*[1]; Bachu N. Singh[1]; Peter Jung[2]; [1]Risoe National Laboratory, Matls. Rsrch., Roskilde DK-4000 Denmark; [2]Forschungszentrum J¸lich, Association EURATOM-FZJ, Institut f¸r Festkˆrperforschung, J¸lich D-52425 Germany

This work is part of an effort to investigate, experimentally as well as theoretically, the role of He in cavity nucleation in neutron irradiated iron and steel. We have already reported results on cavity formation at low temperatures and at low neutron doses in iron and steel without He-implantation. Pure iron and EUROFER97 steel will be homogeneously implanted with He at 50C and 350C to doses of 1, 10 and 100 atppm and subsequently neutron irradiated at the implantation temperatures. The microstructure after He implantation and neutron irradiation will be investigated, as well as the annealing behaviour of some selected specimens. Positron annihilation spectroscopy (PAS), which is sensitive to cavities in the size range from single vacancies to several nano-metres, will be used as the main characterisation technique. The present contribution will describe and discuss the results of the studies of the as-implanted samples.

9:35 AM
Post-Irradiation Deformation of Ferritic Steels: *David S. Gelles*[1]; [1]Pacific Northwest National Laboratory, P8-15, PO Box 999, Richland, WA 99352 USA

In order to understand the processes of post-irradiation deformation in ferritic steels, the microstructures of deformed tensile specimens of Fe-9Cr and F82H have been examined by transmission electron microscopy in order to study dislocation behavior during and after channel deformation. It is found that channel deformation occurs in Fe-9Cr after irradiation to between 10 and 40 dpa at ~400∫C, whereas no channel deformation is found in F82H (Fe-7.5Cr-2W-0.1C) following irradiation to 2.6 dpa at 330∫C. Microstructural studies reveal that a<100> loops formed by irradiation in Fe-9Cr can easily be removed by intersection with moving individual a/2 <111> dislocations, whereas irradiation produced loop damage in F82H is very limited and has little effect on moving slip dislocations. The dislocation interaction mechanisms will be carefully described in order to provide a basis for modeling post-irradiation deformation behavior.

9:55 AM Break

10:25 AM
Dislocation-Defect Interactions in Materials: *Joshua S. Robach*[1]; Athanasios Arsenlis[2]; Ian M. Robertson[1]; Brian D. Wirth[2]; [1]University of Illinois, Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA; [2]Lawrence Livermore National Laboratory, Matls. Sci. & Tech. Div., PO Box 808, L-353, Livermore, CA 94551 USA

In order to develop predictive models of the mechanical response of irradiated materials it is necessary to understand the fundamental physical processes controlling the deformation. This is particularly important near yielding where local defect interactions, which cannot be averaged, may dominate the behavior. Samples of a range of materials containing various densities and distributions of stacking-fault tetrahedra and small dislocation loops were examined using the in-situ TEM straining technique. In this paper, the dynamics of the interaction of dislocations with these defects are presented. Defect annihilation mechanisms as well as the conditions required to produce defect-free channels are proposed. The experimental results are compared to atomistic simulations of unit interactions. Based on these new observations an improved continuum model is presented.

10:45 AM
Irradiation Induced Stacking Fault Tetrahedra in fcc Metals: *Robin Sch‰ublin*[1]; Nadine Baluc[1]; Maximo Victoria[1]; [1]Swiss Federal Institute of Technology Lausanne, CRPP Fusion Tech.-Matls., Villigen PSI 5232 Switzerland

Irradiation can induce the formation of stacking fault tetrahedra (SFTs) in a number of fcc metals, such as stainless steel and pure copper. In order to understand the role of the materialís parameters on this formation, pure Cu, Ni, Pd and Al, having a stacking fault energy of about 70, 140, 180 and 200 mJ/m2, have been irradiated with high energy protons to doses ranging from 10-4 to 10-1 dpa at room temperature. The irradiation induced microstructure has been investigated using transmission electron microscopy. It appears that all irradiated metals but Al present SFTs. Their formation energy as a function of size has been calculated using elasticity of the continuum, with respect to the formation of a number of other possible defect configurations. It appears that the key parameters are the stacking fault energy and the shear modulus. Their implication on the formation of the SFTs under irradiation is discussed.

11:05 AM
Effects of Irradiation on the EUROFER 97 RAFM Steel: *Nadine L. Baluc*[1]; Robin E. Sch‰ublin[1]; Philippe Sp‰tig[1]; Max Victoria[1]; [1]Centre of Research in Plasma Physics, Fusion Tech. Matls., Swiss Federal Inst. of Tech.-Lausanne, Assoc. EURATOM-Swiss Confederation, Villigen-PSI, AG 5232 Switzerland

Series of Charpy and tensile flat specimens have been prepared from a plate of the reduced activation ferritic/martensitic (RAFM) steel EUROFER 97 (Fe-9CrWVTa). A part of them were irradiated with 590 MeV protons in the Proton Irradiation Experiment (PIREX) facility at the Paul Scherrer Institute. The irradiations were performed at ambient temperature (300-320 K), 523 K and 623 K to doses ranging between 0.3 and 2 dpa. Charpy impact tests, tensile deformation experiments were performed over a wide range of temperatures on the unirradiated and irradiated specimens. The defects associated with irradiation were imaged in transmission electron microscopy by using the bright/dark field and weak beam techniques. The whole set of results will be presented in comparison with those previously obtained for the F82H and OPTIMAX series of RAFM steels.

11:25 AM
Characterization and Modeling of Helium Bubbles in Self-Irradiated Plutonium Alloys: *Adam J. Schwartz*[1]; Mark A. Wall[1]; Charlene M. Schaldach[1]; Wilhelm G. Wolfer[1]; [1]Lawrence Livermore National Laboratory, CMS, L-355, 7000 East Ave., Livermore, CA 94550 USA

The characterization of microstructural changes in Pu-Ga alloys resulting from aging phenomena is an important technical challenge to the nuclear Stockpile Stewardship program. We have identified at least two age-related phenomena that may occur in Pu alloys, dimensional changes due to the initial transient, helium accumulation, and void swelling, and phase instability. The initial transient is a well-known effect that results from the initial cascade damage. This form of dimensional change tends to saturate within approximately two years. A second contributor to dimensional change is the build-up of helium as a result of the alpha decay. Helium is generated at a rate of approximately 40 parts per million per year as a result of alpha decay.

Transmission electron microscopy observations indicate a high density of small nanometer sized bubbles. We will describe a model for the nucleation and growth of the He bubbles with simple rate equations. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

Science and Technology of Magnetic and Electronic Nanostructures: Nanoscale Probes

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Ramamoorthy Ramesh, University of Maryland, Department of Materials and Nuclear Engineering, College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Thursday AM Room: 15A
March 6, 2003 Location: San Diego Convention Center

Session Chair: ChangBeom Eom, University of Wisconsin, Dept. of Matls. Sci. & Eng., Madison, WI 27708-0300 USA

8:30 AM
Control of Carbon Nanotube Configuration for Nano-Device Applications: *Sungho Jin*[1]; [1]University of California-San Diego, La Jolla, CA 92130-0411 USA

Carbon nanotubes are fascinating new nano materials with many interesting physical, chemical and electronic properties and potential new applications. They have a graphitic tubular structure with nanometer-scale diameter and very large aspect ratio. For most of the possible technical applications, a well-aligned growth structure, rather than a random, tangled configuration, is desired. While a dense, forest-like alignment is relatively easily obtained, the growth of low-density yet aligned carbon nanotubes with a specific diameter and length at specific nano-scale locations is not easy. Various possible approaches of nucleation control for growth of such desired configuration of carbon nanotubes will be discussed in relation to some potential electronic applications.

9:15 AM
Opportunities in Nanomagnetism: *Samuel D. Bader*[1]; [1]Argonne National Laboratory, Matls. Sci. Div., Argonne, IL 60439 USA

Magnetism is one of the oldest fields in science but it is also at the forefront of the nanotechnology revolution. This talk draws on historical roots to provide a modern experimental perspective on recent issues and challenges in the field. Highlighted areas include the quest for magneto-electronics and the importance of novel interfacial magnetic coupling phenomena. Examples of how nanotechnology provides new strategies to realize composite magnetic materials with exceptional properties and interesting physics will be presented.

10:00 AM
Nanoscale Piezoelectric Imaging and Piezoelectric Nanostructures: *Marin Alexe*[1]; [1]Max Planck Institute of Microstructure Physics, Halle D-06120 Germany

One of the challenging target of the ferroelectric community is to demonstrate that high density devices based on ferroelectric oxides are feasible and there are no technological or basic physical phenomena which will restrict the downscaling of the ferroelectric oxides toward the nanoscale level, i.e. dimensions in the range of 20-100 nm. The present talk addresses simultaneously both issues of patterning of crystalline oxide structures and characterization of ferroelectric at nanoscale levels. Patterning of ferroelectric structures down to several tens of nanometers using both ìtop-downî and ìbottom-upî approaches based on electron-beam lithography and self-assembly methods, respectively will be presented. Ferroelectric, piezoelectric and switching measurements on nanoscale structures and thin films using scanning probe microscope (SPM) working in piezoresponse mode will be also broadly discussed.

10:45 AM
Magnetic Force Microscopy of Nanostructured Materials: *R. D. Gomez*[1]; [1]University of Maryland, Dept. of Electl. & Compu. Eng., College Park, MD 20742 USA

In its invention in 1987, magnetic force microscopy (MFM) has become a dominant tool for magnetic characterization at the ten-nanometer length scales. It is a robust instrument, operable in air, and uses very little surface preparation. Additionally, MFM images obey a straightforward instrument response function, from which the field producing component of the magnetization can be readily extracted. In comparison with other high-resolution imaging techniques, MFM has had the highest impact towards understanding magnetism at the local regime. Magnetic nanostructured materials, which I define as artificially synthesized particles or particle arrays under hundred nm in lateral dimensions, is a field that is greatly influenced by MFM. Among the many conceived applications of these nanoparticles, the technologically vigorous areas include ultrahigh density data storage, magnetic random access memory devices, and biological tagging and detection. In these applications, the keys to successful device development are the understanding of magnetic domains and the behavior with applied fields, the distribution of switching fields, the influence of particle-particle interactions, and spin polarized electron transport. In this talk, I will review the basics of MFM as well as discuss some of the recent advances we developed to enhance its functionality. I will present some of our work on imaging in in-situ applied fields, in ultra-high vacuum, at low temperatures, as well as, driving domain walls using high speed electronic pulses. I will present specific examples and highlight some new insights and processes on small magnetic particles.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Materials Processing Technologies III: Casting and Aluminium Processing

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors
Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Thursday AM Room: Point Loma
March 6, 2003 Location: San Diego Marriott Hotel

Session Chairs: W. D. Cho, University of Utah, Dept. of Metallurgl. Eng., Salt Lake City, UT 84112 USA; V. M. Kevorkijan, Independent Researching Slovenia

8:30 AM
Modeling of Melting Behavior of Aluminium Metal in Molten Salt and Metal Bath: *B. Zhou*[1]; Y. Yang[1]; M. A. Reuter[1]; [1]Delft University of Technology, Dept. of Appl. Earth Scis.,, Mijnbouwstraat 120, Delft 2628 RX The Netherlands

During secondary aluminium recovery, complex aluminium scraps are melted and refined often in a rotary melting furnace, and the aluminium metal has to pass through a molten salt layer and melt down in a bottom aluminium bath. An experimental study and industrial observations have indicated that salt shell formation and re-melting on the metal solids in the early stage plays a critical role in the overall melting process of the scrap. In the present paper, mathematical models are developed to simulate the process of salt shell formation and its re-melting, as well as aluminium melting in the molten melt. Influence of the salt layer properties, the residence time of the metal in the salt layer, the particle size and shape, and the temperature of melt were investigated. Reasonable agreement with the measurement data was reached. The developed melting model will be used to construct a population balance model of the scrap melting behaviour for a rotary melting furnace, taking into account the distributed characteristics of the scrap. Finally, an overall process model based on Computational Fluid Dynamics (CFD) for the rotary furnace will be constructed, coupling with the population balance model for the scrap melting.

8:55 AM
Electrical Noise Analysis of Industrial Aluminium Smelting Cells: *A. Agnihotri*[1]; [1]Jawaharlal Nehru Aluminium Research Development and Design Centre, Wadi, Nagpur 440023 India

One of the interesting research endeavours in the aluminium reduction industry has been resistance/voltage variation in aluminium reduction pots. Measurement of cell voltage and current simultaneously were conducted in actual plant with high-speed data acquisition system. It was found that the amplitude, frequency, etc. of cell resistance provided valuable information of anode bottom, metal rolling, cell temperature, etc. Special measurements were made in cells at various states of disturbances viz. uneven anode bottom, before anode effect, during anode effect, after anode effect. The resistance/voltage data was analysed at these cell states and electrical noise patterns were studied. The study provided valuable information on cell state by means of data, which is generally averaged out in normal plant operation. Statistical analysis of data obtained provides further scope in evaluating the status of process and detecting operational problems like uneven anode bottom, hydro-dynamic instability, etc. Data analysis deserves more attention as they can be used in predicting the cell stability and operating parameters at very early stage. Details of these are presented in this paper.

9:20 AM
Theoretical Bases of High-Efficient Purification of Molten Aluminum and Purification Practice: *Gaosheng Fu*[1]; Jixing Kang[1]; Wenzhe Chen[1]; Kuangwu Qian[1]; [1]Fuzhou University, Dept. of Mechl. Eng., Fuzhou 350002 China

In order to obtain aluminum products with higher quality, the harm of metallurgical defects such as inclusions (Al2O3) and hydrogen must be eliminated or weakened before pouring molten aluminum into mold, because their presence will have direct or indirect influences on strengths, deformation properties and performances of aluminum products. The effective purification treatment of molten aluminum is the key to improving melt cleanness of aluminum. In this paper, based on the analyses of the essences of behavior of inclusions and hydrogen in molten aluminum and their interactive relationship, which are the theoretical bases of high-efficient purification of molten aluminum and are of the utmost importance in determining the technology of purification of molten aluminum, the interactive ìparasitic mechanismî between inclusions and hydrogen in molten aluminum is put forward. At the same time, aimed at the currently existing problems in the methods of purification, the principle for purification of molten aluminum, that is, ìremoving inclusions is the basis for eliminating hydrogen and, therefore, more attention should be firstly paid on removing inclusionsî is firstly put forward, thus breaking through the trammel of traditional ideas of purification i.e., more attention is firstly paid on eliminating hydrogen. The principle has been tested and verified in authorsí researches and practices. According to this principle above, a new method of purification by filtrating with flux and a corresponding high-efficient flux for removing inclusions have been developed, which has been successfully applied to productions of some high-performance aluminum sheets such as aluminum sheet used for pressure can, etc. It is found that with special flux(CJ-5) for removing inclusions and filtration technology, and proper addition amount and melting temperature, the rate of removing inclusions and the extent of lowering porosity for different grades commercial purity aluminum could amount to about 70-82% and 60-88% respectively, and mechanical properties of this material were improved remarkably, especially the relative increase of elongation(d) was about 70% in comparison to that of no filtration.

9:45 AM
Original Industrial Application of Two Numerical Models in Concasting Technology: Frantisek Kavicka[1]; *Josef Stetina*[1]; [1]Brno University of Technology, Fac. of Mechl. Eng., Technicka 2, Brno 616 69 Czech Republic

Solidification and cooling of a continuously cast slab and simultaneous heating of a crystallizer is a very complicated problem of transient heat and mass transfer. Nowadays, the solving of such a problem is impossible without numerical models of the temperature field not only of the slab itself, while it is being processed through the whole concasting machine (CCM), but of the crystallizer as well. Two original numerical models have been developed and used in the investigation of a continuously cast steel slab. The first (one of two) 3D model of the temperature field of a concasting is capable of simulating the temperature field of a caster. Experimental research and data acquisition have to be conducted simultaneously with the numerical computation-not only to confront it with the actual numerical model, but also to make it more accurate throughout the process. After computation, it is possible to obtain the temperatures at each node of the network, and at each time of the process. The utilization of the numerical model of solidification and cooling of a concasting plays an indispensable role in practice. The potential change of technology-on the basis of computation-is constantly guided by the effort to optimize, i.e. to maximize the quality of the process. The user can therefore choose any appropriate longitudinal or cross-section of a slab and display or print the temperature field in a 3D or 2D graph whenever necessary. The second numerical model of dendritic segregation of elements assesses critical points of slabs from the viewpoint of their increased susceptibility to crack and fissure. In order to apply this model, it is necessary to analyse the heterogeneity of samples of the constituent elements (Mn, Si and others) and impurities (P, S and others) in characteristic places of the solidifying slab. The numerical model, based on measurement results obtained by an electron micro-probe, generates distribution curves showing the dendritic segregation of the analysed element, together with the distribution coefficients of the elements between the liquid and solid states. The combination of both models enables the prediction of cracks and fissures in critical points of the continuously cast carbon-steel slab. Both models had been applied in the industrial investigation of a cast low-carbon-steel slab.

10:10 AM Break

10:30 AM
Study on the Energy Absorption Property of Foaming Alminium: *Yihan Liu*[1]; Guangchuen Yao[1]; Xiaoming Zhang[1]; [1]Northeastern University, Sch. of Matls. & Metall., Shenyang 110004 China

Some different density foaming aluminium samples were prepared in the semi-industrial scale experiment system. Their properties such as energy absorption characteristics, mechanical properties, heat conductivity and acoustical properties were detected. The results showed that the foaming aluminium density from 0.5 to 0.6 g/cm3 have the better energy absorption, suit to be the ensuring bar of the car, and also suit to be the cushion materials for the engine. The foaming aluminium density from 0.25 to 0.4g/cm3 have the better acoustical properties and heat conductivity resistance, suit to be the filling material for the door, the ceiling and the partition of the car engine.

10:55 AM
Study on Formation and Magnetic Susceptibility of Intermetallic Compound Containing Iron, Manganese in the Aluminum Melts: *Guangchun Yao*[1]; Linli Wu[1]; Lei Zhang[1]; [1]Northeastern University, The Key Lab. of Electromagnetic Procg. of Matls., Shenyang 110004 China

Iron and silicon impurities would form needle and branch shape intermetallic compounds in the molten aluminum when there was no manganese. It is difficult to separate them from molten aluminum by electromagnetic separation. The Fe-Si-Al and Fe-Al intermetallic compounds were transform into Mn-Fe-Si-Al or Mn-Fe-Al compounds when the manganese was added into the molten aluminum. The metallograph show that the shape of these intermetallic compound particles is lumpish and spherical shape. These impurity particles will suffer less resistance when they were migrated in the molten aluminum and it is benefit to be separated. Some $FeSiMnAl_4$ and $FeMnAl_6$ were paramagnetic materials. Their impurity particles could be migrated by the magnetic force and could be separated from the molten aluminum.

11:20 AM
Study on the Factors Affecting Separation of Iron Impurity from Molten Aluminum by Altering Induced-Magnetic Field: *Guangchun Yao*[1]; Lei Zhang[1]; Linli Wu[1]; [1]Northeastern University, The Key Lab. of Electromagnetic Procg. of Matls., Shenyang 110004 China

There are branching and needle-shape intermetallic compounds composed of iron, silicon and aluminum in the molten aluminum. When manganese metal was adding into molten aluminum in a suitable proportion, the shape of the intermallic compound particles may become lumpish and spherical. Depend on the difference of these impurity particles and molten aluminum in electric and magnetic properties, these impurity intermetallic compound particles can be separated by altering magnetic field. In this experiment the altering magnetic field was formed by a ìCî type loop which AC (500Hz) current went through. The inner vacum of the separator made of fireclay refractory materials was 25◊15◊130mm^3 with a lateral baffle inside. The separator was put into the interspace of the ìCî type loop. The experiments of separating iron-rich impurity particles were conduced in different separate time, magnetic intensity and adding manganese respectively. It was found that the longer the separating time was the larger transfe rred amount of impurities would be within three minutes, the transferred amount of impurities increased with the magnetic field intensity strengthened, the separating impurities effect from molten aluminum was increased obviously by adding manganese metal.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Experimental Techniques

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Thursday AM — Room: Solana
March 6, 2003 — Location: San Diego Marriott Hotel

Session Chairs: Roberto Parra, Universidad de ConcepciÛn, Dept. of Metallurgl. Eng., ConcepciÛn Chile; Takeshi Azakami, Saitama Institute of Technology, Mechl. Eng., Saitama Japan

8:30 AM
Recent Progress in Thermal Diffusivity Measurement of Molten Oxides by the Laser Flash Method: *Hiromichi Ohta*[1]; Shibata Hiroyuki[2]; Yoshio Waseda[2]; [1]Ibaraki University, Dept. of Matls. Sci., Hitachi 316-8511 Japan; [2]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls. (IMRAM), Sendai 980-8577 Japan

Precise thermal properties of molten materials are essentially required for analyzing the process control in metal production. These properties are also important as a clue to consider the dynamic structure of liquid materials. However, it is difficult to obtain the precise thermal diffusivity values of molten materials at high temperature because of the effect of bubble formation and the severe separation of radiative and conductive heat components. We have developed many types of laser flash technique to measure thermal diffusivity of molten oxide such as three layered cell system composed of metal plate/liquid phase/metal crucible, two layered cell system of liquid phase/metal crucible and differential data analysis to estimate the sample thickness as well as radiative heat flow. The main purpose of this paper is to present a novel experimental technique recently developed for measuring thermal diffusivity of molten oxides at high temperatures. In this new technique, the sample is melted in a metal crucibl e and heated to the desired temperature. A single laser pulse is flashed on the bottom of the crucible and infrared ray irradiated from the same bottom surface is measured for obtaining the temperature decay from which thermal diffusivity of the liquid sample can be estimated. The validity of this new laser flash technique has been confirmed by obtaining the thermal diffusivity values of silicate melts in the temperature range between 773 and 1673 K.

8:55 AM
Two Methods for the Continuous Measurement of Slag Viscosity from the Glassy to the Liquid States: *Yutaka Shiraishi*[1]; Yutaka Sakurai[1]; [1]AGNE Gijutsu Center, Ltd., Minami-Aoyama 5-1-25, Kitamura Bldg., Minato-ku, Tokyo 107-0062 Japan

Two methods were developed to measure the viscosity of slag from the glassy to the liquid states in an ascending temperature process. The first method, referred to as the sp method, employs rotating parallel plates within which is placed a solid cylindrical sample in line with a sphere. The viscosity of the glass sample is first determined from the rate of penetration of the sphere into the sample. After the sphere is fully immersed within the sample, the viscosity is determined from the creep rate of the sample between the parallel plates. In the liquid state, the viscosity is determined from the torque generated by the rotation of the plates. The second method, referred as the tp method, uses a tube which is pushed into a sample held in a rotating crucible. In this case, the viscosity is determined from the rate of penetration of the tube into the glass sample and from the torque exerted on the tube by the rotation of the liquid sample. Both methods were tested using standard glasses, including boron trioxide and NBS standard glass #711. The main difference between these two methods is in sample preparation. Comparative measurements between the sp and the tp methods were performed using a sodium boro-di-silicate glass containing 5%mol of alumina. The results showed good agreement in the mid-range of glassy viscosities, 105 to 107 Pa.s. In the liquid state, there were no significant differences between the two methods. A discussion is presented about the observed discrepancies in the results.

9:20 AM
Advanced Image Analysis of Molten Slag for Process Control: *Subagyo*[1]; G. A. Brooks[1]; [1]McMaster University, Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

The condition of slag in a metallurgical reactor is vital to ensuring control of the process. A new technique based on multivariate image analysis (MIA) of digital images processing has been developed to provide an online measurement of important slag characteristics, such as the presence of exposed metal, the presence of solid phases, and temperature. This paper shall describe the technique and provide some results from plant trials.

9:45 AM
Impurity Removal from Carbon Saturated Liquid Iron Using Lead Solvent: *Katsunori Yamaguchi*[1]; Yoichi Takeda[1]; [1]Iwate University, Fac. of Eng., Ueda 4-3-5, Morioka, Iwate 020-8551 Japan

A new technique for the removal of tramp-elements from steel scrap has been required from the viewpoint of saving energy and protection of environment. In order to obtain fundamental information on the removal of copper, tin and zinc from molten iron, the extraction method by using of phase separation in the liquid Fe-C-Pb system was investigated at 1453K. A mixture of iron, lead and carbon melted in a carbon crucible separates into two phases of lead and iron containing carbon. The compositions on the miscibility gap in the Fe-C-Pb system are 95.4%Fe-4.5%C-0.1%Pb and 99.9%Pb-0.1%Fe. The distribution ratios of copper, tin and zinc between the lead and the iron phases, LX=[mass%X]Pb/(mass%X)Fe, are 2.2, 2.1 and 1.4, respectively. Seventy percent of copper and tin from iron scrap can be eliminated with comparable lead addition.

10:10 AM Break

10:30 AM
The Criteria of Hydrogen and Oxygen Potentials in Copper Melt for Making a Sound Casting of Bar and RodñThe Guide of the Effective Use of an Electrochemical Gas Monitoring System: *Norihiko Fukatsu*[1]; Noriaki Kurita[1]; Daisuke Yamamoto[2]; [1]Nagoya Institute of Technology, Dept. Matls. Sci. & Eng., Gokiso-cho, Showa-ku, Nagoya 466-8555 Japan; [2]Mizuno Corporation, Miyahara-cho, Omiya 330-0038 Japan

The in-line monitoring of the hydrogen and oxygen potentials in molten copper has been realized by using the electrochemical sensors based on the proton and the oxide ion conducting solid electrolyte. In order to use this sensing system effectively, the criteria of the potentials for making sound casting must be known. In the present work, the copper melt equilibrated with a given hydrogen and oxygen potentials was quenched in the crucible. The relations between the holding potentials and the gas porosity in the solidified sample were investigated by measuring the density of the sample and microscopic observations. The criteria thus determined were examined with reference to the measured potentials in the practical casting machine. In tough-pitch copper casting, the problem due to gas porosity was found to be serious when the hydrogen potential increased. In the oxygen-free copper casting, the evolution of hydrogen was found not to be avoided. The gas porosity in the casting, however, was found to be eliminated when the oxygen potential was kept at a low value.

10:55 AM
Viscosities of FeO-MgO-SiO2 and FeO-MgO-CaO-SiO2 Slags: Xi Dai[1]; Xueping Gan[1]; Chuanfu Zhang[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

In the present work,the viscosities of molten FeO-MgO-SiO2 and feO-MgO-CaO-SiO2 semi-synthetic slags at nickel flash smelting conditions were measured in the temperature range from 1523 to 1723 K using a rotational viscometer. The Fe/SiO2 ratio was maintained constant at 1.2 and the calcium and the magnesium oxide contents were varied in the range from 2 to 8 wt-% and 9 to 12 wt-%, respectively. The viscosity values are presented as a function of temperature and composition.

11:20 AM
Measurement of the Oxygen Potential of Non-Ferrous Slags with an Ex-Situ Electrochemical Device: *Nele Moelans*[1]; Bert Coletti[1]; Bart Blanpain[1]; Patrick Wollants[1]; Jaak Plessers[2]; Marc

THURSDAY AM

Straetemans[2]; [1]Katholieke Universiteit Leuven, Dept. of Metall. & Matls. Eng., Kasteelpark Arenberg 44, Leuven B-3001 Belgium; [2]Heraeus Electro-Nite Int. N.V., Centrum Zuid 1105, Houthalen B-3530 Belgium

A measurement method using an ex-situ electrochemical measurement device was developed to determine the oxygen potential of non-ferrous metallurgical slags. The electrochemical cell consists of the molten slag sample, an inert Ir electrode, a stabilized ZrO2 solid electrolyte, and a Ni/NiO reference electrode. Different slags obtained from non-ferrous pyrometallurgical processes were analysed at 1100 and 1200∞C and $p(O_{/2})$ values between 2 10^{-3} and 1 10^{-12} were obtained. The measurement values were evaluated with thermodynamic calculations and using processing considerations. It was found that the reproducibility and stability of the measurement was significantly improved with the addition of carbon to the slag, especially for slags with a low oxygen potential.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: Copper IV

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Thursday AM — Room: Santa Rosa
March 6, 2003 — Location: San Diego Marriott Hotel

Session Chairs: Susumu Okabe, Mitsubishi Materials Corporation, Tokyo 100-8117 Japan; Florian Kongoli, FLOGEN Technologies Inc., Matls. Tech. Dept, Montreal, Quebec H3S 2C3 Canada

8:30 AM

Application of Porous Plug System in Anode Furnace, Onsan Smelter: *Sang-Su Lee*[1]; Baek-Sang Kim[1]; Sei-Phim Choi[1]; [1]LG-Nikko Copper, Inc., Daejung-Ri 70, Ulju-Gun, Onsan-Eup, Ulsan City 689-892 Korea

This paper shows the application of porous plug system in the Mitsubishi continuous copper smelting and converting process. This system has already adopted and utilized in the steel industry to enhance productivity. Recently, some copper smelter has adopted this system but the result has not been satisfactory. Porous plug system was installed at the Anode furnace in December 2001 to reduce oxidation time and decrease the oil consumption. Some good effect is that nitrogen stirring the melt through porous plugs, improved the heat transfer of the melt. As the result of enhancing heat transfer, oil consumption and inside build-up was actually decreased. But the oxidation time has not sufficiently been reduced until now. In the future, further research and local test is needed for productivity increase and cost saving.

8:55 AM

Behavior of Silver in the Electric Furnace for Copper Dross Treatment: *Takao Yasugi*[1]; [1]Toho Zinc Company, Ltd., Production Dept., 5562-1 Higashino-cho, Toyota-gun, Hiroshima-Pref. 725-0222 Japan

The majority of copper in the raw material migrates to the decopperized dross formed when crude lead produced from the blast furnace is treated by the decopperizing process. Decopperized dross is treated in the electric furnace, producing crude lead, matte, and speiss. Decopperized dross contains large amounts of silver. Therefore, it is important to improve the rate of silver migration to crude lead. For this reason, we examined the behavior of silver between the crude lead, speiss, and matte phases. At the same time, we conducted several tests to improve the percentage of silver migrating to crude lead.

9:20 AM Invited

A Process Designed for the Ancient Copper Smelting Slags: *Bora Derin*[1]; *Onuralp Y¸cel*[1]; Ercan Acma[1]; Okan Addemir[1]; [1]Istanbul Technical University, Metallurgl. & Matls. Dept., Fac. of Cheml. & Metallurgl. Eng., Maslak, Istanbul 80626 Turkey

This work carried out on the ancient copper smelting slags located in K¸re-Turkey for recovery of valuable metals. In this process, copper and cobalt were recovered as metallic and their components, where as iron was recovered as magnetic oxide or pigment from the slag. The process stages were involved carbothermal reduction in DC-Arc Furnace, leaching, chemical precipitation, selective roasting and product preparation. In this paper, the process stages were described as theoretically and experimentally where as slag and products were characterized in detail.

9:45 AM

Present and Future of Caletones Smelter: *Patricio Burchard Chacana*[1]; *Gerardo Hern·n Achurra*[1]; Julio Gatica Buchi[1]; Fernando Andres Condore[1]; [1]CODELCO, El Teniente, Caletones Smelter, Millan 1020, Rancagua, OíHiggins VI 000 Chile

During the last years, Caletones Smelter like others world copper smelter, has evolved from conventional, inefficient, pollutant and expensive technologies, to modern, environmentally friendly and efficient ones. For it and besides for improving its competitive position, in the last period this Smelter shut down their reverberatory furnace, started up two big acid plants and has developed some pirometallurgical technologies. The main facilities of Caletones Smelting and Converting Process, in the present and future, are the Teniente Converters, reactors with a high operational availability that have reached concentrate top process capacities near to 2,400 T/D. These reactors operate in most of the Chilean smelters as well as in other world smelters: Zambia, Peru and Mexico. Since 2000, the Caletones Smelting and Converting Process, has been performed in two Teniente Converters, four Slag Cleaning Furnaces and four Pierce-Smith Converters. These facilities, together with the operation of two Fluidized Bed Drye rs and two Oxygen Plants have allowed to reach concentrate smelting capacities and copper productions of 1,250 KDT/year and 380 KFT/year, respectively. Associated to El Teniente Division Expansion Plan, in a first stage, Caletones Smelter will increase its capacity to 1,440 KT/year the 2004. In the long term, Caletones has different options, from 1,600 to 2,000 KT/year, producing anodes, white metal or a mixture of them. This options are connected with CODELCOís Smelters & Refineries Strategic Plan, including Mejillones Project. The main challenges of Caletones Smelter are associated to develop and consolidate Continuos and Environmental Friendly Processes, automation and control of different processes and improve its leadership between World Copper Smelters.

10:10 AM Break

10:25 AM Invited

Thermodynamic Fundamentals of Calcium Ferrite Slag and their Application to Mitsubishi Continuous Copper Converter: *Fumito Tanaka*[1]; Osamu Iida[2]; Yoichi Takeda[3]; [1]Mitsubishi Materials Corporation, Central Rsrch. Inst., 1-297 Kitabukuro-cho, Saitama 330-8508 Japan; [2]Mitsubishi Materials Corporation Metals Company, Process & Tech. Dept., 1-5-1 Ohtemachi, Chiyoda-ku, Tokyo 100-8117 Japan; [3]Iwate University, Dept. of Matls. Sci. & Tech., 4-3-5 Ueda, Morioka, Iwate 020-8551 Japan

The Mitsubishi process is the sole pyrometallurgical process for continuous production of blister copper from copper concentrates and has been successfully operated around the world. The development of calcium ferrite slag (CaO-FeOX-Cu2O), so called ìC-slagî, has enabled continuous converting from matte to blister copper in commercial scale operation. Commercial interests in the slag led to many papers over CaO-FeOX slag. However, Cu2O-bearing slag has not been studied well due to experimental difficulties. The authors have quantified thermodynamic properties of CaO-FeOX-Cu2O system. The results have been utilized to analyze various issues related to the converting furnace, such as magnetite precipitation from slag or from blister copper, and to develop better control criteria for the continuous converting operation.

10:50 AM

Process Development, Optimization and Automation through Appropriate Thermophysicochemical Modeling and Simulation Software: *F. Kongoli*[1]; I. McBow[1]; S. Llubani[1]; [1]FLOGEN Technologies, Inc., 5757 Decelles Ave., Ste. 511, Montreal, Quebec H3S 2C3 Canada

Nonferrous and ferrous smelting industries have recently faced the unavoidable necessity of changing and/or improving the smelting technologies as a result of the use of new raw materials which are becoming available from different geographical areas. These new feed materials usually contain different ore composition and higher level of minor components, which adversely affect the smelting process. Due to the problems encountered in several processes as a result of this feed diversification some work has been undertaken in order to make uniform the feed and avoid later surprises in the smelting process. However this has proven to be very difficult and sometimes almost impossible. In this work a more viable approach is undertaken to deal with this problem. It consists of controlling not the cause of the problem i.e. the feed composition but instead the results of this feed change, i.e. the end-point of smelting technologies. It is shown that this approach, when carried out through appropriate thermophysico-chemical modeling and simulation software is not only easier and less costly but it also helps the automation of the smelting process. Several examples are given and future work is underlined.

11:15 AM

Control of Magnetite Behavior in the Mitsubishi Process at Naoshima: *Nozomu Hasegawa*[1]; Hideya Sato[1]; [1]Mitsubishi Materials Company, Ltd., Naoshima Smelter & Refinery, 4049-1 Naoshima-Cho, Kagawa-Gun, Kagawa 761-3110 Japan

The phase diagrams and microstructures of slags in the Mitsubishi Continuous Copper Smelting and Converting Process were studied for the prevention of magnetite troubles such as accretion build-up on furnace hearths and launders, melt outlet blockages, slag viscosity increases and so on. According to the result of those studies, the silica and lime content in the silicate slag at the smelting furnace and the lime content in the calcium-ferrite slag at the converting furnace were changed, and then the slag loss of copper in the discard slag has been stabilized lower than before and the cleaning- frequency of melt outlets and launders has been significantly reduced. However, those procedures may decrease the desirable amount of accretion-coating inside the furnaces and thereby reduce the furnace campaign lives. Therefore, an estimating method of those accretion amounts was developed, and slag compositions and temperatures have been controlled within the appropriate ranges to prolong furnace lives and also prevent magnetite troubles.

11:40 AM

The Behavior of Impurities at Kosaka Smelter: *Satoshi Nakagawara*[1]; Kenji Watanabe[2]; [1]Kosaka Smelting & Refining Company, Ltd., Production Mgmt. Sec., 60-1 Otarube, Kosaka, Kazuno, Akita 017-0202 Japan; [2]Kosaka Smelting & Refining Company, Ltd., Techl. Dept., Kosaka, Kazuno, Akita 017-0202 Japan

Kosaka Smelter has been changed to a custom smelter and devoting to treat the complex sulfide copper concentrates in the world. Since the mid-80s, a progressive diversification of the smelter input has taking place, impure and complex sulfide concentrate being introduced in increasing proportions compared with the period of the ìBlack Oreî treatment. And recently, Kosaka is trying to add value to its copper smelting by recovering valuable metals from non-concentrate materials (e.g.?\@recycled materials and the residue generated in the zinc smelter), and by increasing the volume of recovery. In this process, precious metals are mostly recovered and unnecessary impurities are efficiently removed. Thus, it can be said that recent operation of Kosaka Smelter depends on the treatment of impurities. This is a report on the behavior of impurities at Kosaka Smelter which performs outstanding smelting operation.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Iron and Steel Making Fundamentals III and Applications

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Thursday AM — Room: Leucadia
March 6, 2003 — Location: San Diego Marriott Hotel

Session Chairs: H. G. Kim, POSCO Research Institute Korea; Varadarajan Seshadri, Universidade Federal de Minas Gerais, Dept. of Metallurgl. Eng. & Matls., Belo Horizonte Brazil

8:30 AM Invited

Operational Improvements for RH-KTB Degasser of Companhia Siderurgica De Tubarao (CST) Steel Plant Through a Physical Modeling Study: *Varadarajan Seshadri*[1]; Itavahn Alves da Silva[2]; Carlos Antonio da Silva[2]; Roberto Parreiras Tavares[1]; Ernane M·rcio de Castro Martins[3]; Fernando Demuner da Silva[1]; Geraldo Alves Vargas Filho[4]; Paulo SÈrgio Bringhenti Lascosqui[4]; [1]Universidade Federal de Minas Gerais, Dept. of Metallurgl. Eng. & Matls., Belo Horizonte Brazil; [2]Universidade Federal de Ouro Preto, Dept. of Metallurgl. & Matls. Eng., Ouro Preto Brazil; [3]Universidade Federal de Ouro Preto, REDEMAT, Ouro Preto Brazil; [4]Companhia Sider˙rgica de Tubar„o, Vitoria Brazil

Improvements in the efficiency of the metallurgical performance of refining reactors in the metallurgical industry can be achieved through physical modeling of the unit and experimentation in the laboratory scale. In the Rh process, macroscopic parameters such as circulation time and mass transfer coefficient for the degassing step are important parameters which decide improved operational practice. In this study the effect of some operational parameters, such as diameter of snorkels, gas injection flow rate through bottom of the ladle, gas flow rate through nozzles on the circulation rate and decarburization of steel are investigated using a physical model. The object of the investigation was to improve the performance characteristics of the RH-KTB degasser of Companhia Sider˙rgica de Tubar„o (CST) steel plant.

9:00 AM

Optimization of the Injection Refining-Up Temperature Process (IR-UT) of the Companhia Siderurgica de Tubarao Steel Plant (CST) Using Physical Modeling: *Carlos Antonio da Silva*[1]; Itavahn Alves da Silva[1]; Varadarajan Seshadri[2]; Cristiano Magson de Oliveira Genelhu Silva[3]; Marcos de Paula Alves[1]; Carlos Alberto Perim[4]; [1]Universidade Federal de Ouro Preto, Dept. of Metallurgl. & Matls. Eng., Ouro Preto Brazil; [2]Universidade Federal de Minas Gerais, Dept. of Metallurgl. Eng. & Matls., Belo Horizonte Brazil; [3]Universidade Federal de Ouro Preto, REDEMAT, Ouro Preto Brazil; [4]Companhia Sider˙rgica de Tubar„o (CST), Vitoria Brazil

Steel refining reactors based on chemical heating are being used as an alternative to ladle furnaces. They show a high degree of flexibility in respect of heating rate in addition to the ease in carrying out refining reactions such as desulphurization, inclusion removal, alloying etc. IR-UT and CAS-OB are examples of such reactors. Their performance is dependent upon geometrical characteristics as well as operational conditions which determine the flow field inside the vessel. A physical model of the IR-UT process of the Companhia Sider˙rgica de Tubar‚o steel plant situated in Vitoria, Brazil was developed to assess the features of the flow field and its influence on the metallurgical behavior. Results of the physical model were used to optimize the process parameters.

9:25 AM
Effect of TiC Addition on Corrosion Resistance of MgO-C Based Refractories to Smelting Reduction Slag: *Qingcai Liu*[1]; Jing Lin[1]; Dengfu Chen[1]; Joseph W. Newkirk[2]; [1]Chongqing University, 174 Shapinba St., Chongqing 400044 China; [2]University of Missouri-Rolla, 1870 Miner Cir., Rolla, MO 65409 USA

The interaction between MgO-C-TiC refractories and slag of smelting reduction with and without iron bath was studied by rotary immersion and stationary immersion test. The effects of the TiC addition to the corrosion behavior of the MgO-C based refractories were investigated in detail. The present work highlights significant improvements in the corrosion resistance of the refractories to slag from addition TiC in the MgO-C based refractories. Petrographic and SEM analysis of the refractories after the slag test show that TiC increases the viscosity of both the glassy phase of the refractory and the slag film. This effect retards the slag penetration into refractory and the interaction between the slag film and the deterioration layer of refractory.

9:50 AM
Assessment of the Performance of the Tundish of a Six Strand Continuous Casting Unit of Companhian Siderurgica de Belco-Mineira(CSBM) Using Physical Modeling: *MÙnica Suede Santos Silva*[1]; Elvis GonÁalves da Mota[2]; Edilson CaniÁali Fracalossi[2]; Carlos AntÙnio da Silva[2]; Itavahn Alves da Silva[2]; Joaquim GonÁalves da Costa Neto[3]; Varadarajan Seshadri[4]; [1]Universidade Federal de Ouro Preto, REDEMAT, Ouro Preto Brazil; [2]Universidade Federal de Ouro Preto, Dept. of Metallurgl. & Matls. Eng., Ouro Preto Brazil; [3]Companha Siderurgica de Belgo Mineira, Jo„o Monevalde Brazil; [4]Universidade Federal de Minas Gerais, Dept. of Metallurgl. Eng. & Matls., Belo Horizonte Brazil

This work describes characterization of the performance of the tundish of a six strand continuous casting unit of the Companhia siderurgica Belgo Mineira, Jo„o Monlevade, Brazil using physical modeling. Permanent and transient conditions, due to ladle metal composition changes are considered. Pulse and step tracer addition techniques are employed to assess the control variables. For transient conditions the degree of inter-mixing and the minimum residence time are determined with a view to optimizing the process especially during the grade changes of steel.

10:15 AM Break

10:25 AM
Interfacial Area in Pyrometallurgical Reactor Design: *Geoffrey Alan Brooks*[1]; Subagyo[1]; [1]McMaster University, Dept. of Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S4L7 Canada

Increasing interfacial area through gas injection is one of the main methods for accelerating reaction between slag, metals and gases in modern metallurgical reactors. This paper will examine the limitations of top lance and bottom tuyere blowing in terms of interfacial area generation and examine alternative reactor designs that may overcome these limitations. The effect of the quantity and size of droplets generated through gas injection on overall reactor design performance, especially relating to settling rates, will be examined in this paper.

10:50 AM
Software for Selection and Analysis of Mould Fluxes for Continuous Casting of Slabs: *Fabricio Batista Vieira*[1]; Varadarajan Seshadri[2]; Roberto Parreiras Tavares[2]; [1]Vallourec & Mannesmann Tubes, Belo Horizonte, MG 30640-010 Brazil; [2]Federal University of Minas Gerais, Metallurgl. & Matls. Eng. Dept., Belo Horizonte, MG Brazil

Mould fluxes are synthetic slags normally used in continuous casting of steels, particularly in slab casting. They should satisfy several requirements, which include: - reducing the friction between the strand and the mould walls; - controlling the heat transfer between the solidified shell and the mould. The performance of mould fluxes in satisfying the requirements above can have a significant effect on the efficiency of the continuous casting process and on the surface quality of the products. This performance is affected by various parameters including the characteristics of the mould fluxes, the quality of the steel being cast and the operational conditions of the caster. The extremely complex relationships involving the parameters mentioned above make it very difficult to have simple criteria for selecting mould fluxes for casting a certain grade of steel in a continuous casting machine. These difficulties led steelmakers and mould flux suppliers to select mould fluxes based mainly on plant trials. In t he present work, a software to help in the analysis and development of mould fluxes for continuous casting was developed. This software includes several models for prediction of mould flux viscosity and consumption. A mathematical model was also used to predict the heat flux through the mould flux. The proposed methodology was applied to continuous casting of crack sensitive steels. Some of the results given by this software are presented, discussed and compared to plant data.

11:15 AM Invited
Modeling of EAF Slag Chemistry for Optimal Slag Foaming and Refractory Service Life: *James P. Bennett*[1]; Kyei-Sing Kwong[1]; [1]USDOE, Albany Rsrch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

EAF slag chemistry and its control have been recognized as important in producing quality steel at a low price for a number of decades. EAF steel producers make lime and dolomite additions to furnaces, controlling the C/S ratios and sulfur pickup from the steel. Existing phase diagram data at 1600°C for CaO, MgO, FeO, SiO2, and Al2O3 was used to write a computer model optimizing slag chemistry for slag foaming and reduced refractory wear. The model, its development, and its use in an industrial steelmaking environment will be discussed along with how the model can improve energy efficiency. The importance of EAF mass balance to account for critical materials like Si in scrap and refractory wear using the model will also be emphasized.

General Poster Session

Program Organizers: TMS, Warrendale, PA 15086 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Mon PM - Tues PM - Wed PM Room: Upper Level Ballroom 6 Lobby
March 3-5, 2003 Location: San Diego Convention Center

Effects of Changes in Test Temperature and Notch Root Radius on Fracture Toughness of Fully Pearlitic Eutectoid Steel: *Adel B. El-Shabasy*[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., Case Sch. of Eng., Cleveland, OH 44106 USA

The effects of changes in test temperature and notch root radius on the fracture toughness of fully pearlitic eutectoid steel have been investigated. Notched and fatigue precracked specimens have been tested at 148 K as well as room temperature. The effects of changing the notch root radius from fatigue precracked to that of blunt notch on the fracture toughness of fully pearlitic eutectoid steel are observable. Reducing the test temperature from the room temperature to 148 K reduces the precracked specimenís fracture toughness by nearly 50%, while the toughness values of the notched specimens are approximately double the toughness values of the fatigue precracked specimens. In addition, the effects of changes in microstructural features (e.g. prior austenite grain size, pearlitic inter lamellar spacing) on the fracture toughness are presented.

Effects of Mixed Mode Loading on Fracture Toughness of Fully Pearlitic Eutectoid Steel at Low Temperature: *Adel B. El-Shabasy*[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., Case Sch. of Eng., Cleveland, OH 44106 USA

The fracture toughness of fully pearlitic eutectoid steel under mixed mode I/II loading has been investigated using notched bend bar specimens. In this method, notched specimens of fully pearlitic eutectoid steel are subjected to combined mode I/II fracture under asymmetric three-point bend loading and to pure mode I under symmetric bend loading. All the tests were performed at 148 K on four different microstructural conditions for a fully pearlitic eutectoid steel. Increasing the eccentric distance between the notch and the loading point produces an increase in the ratio mode II/mode I, and a change in the fracture angle. The effects of changes in the ratio mode II/mode I on the fracture load, fracture angle, and resulting toughness are presented.

Electrochemical Properties of the Vacuum Arc Deposited WC-Cr1-xAlxN Multilayer Coatings in a 3.5%NaCl Solution: *Seung-Ho Ahn*[1]; Ji-Hong Yoo[1]; Jeong-Ho Lee[1]; Jung-Gu Kim[1]; Ho-Young Lee[1]; Jeon-Gun Han[1]; [1]SungKyunKwan University, Dept. of Adv. Matls. & Eng., 300, Chunchun-Dong, Jangan-Gu, Suwon, Gyounggi-Do 440-746 S. Korea

Multilayered WC-Cr1-xAlxN coatings of stepwise Al concentration were deposited on AISI D2 steel by cathodic arc deposition(CAD). The Al concentration could be controlled by using evaporation source for Al and fixing the evaporation rate of other metals (WC alloy and Cr). WC-Cr1-xAlxN coatings are performed by their periodically repeated structures of lamellae of WC-Cr/WC-Cr1-xAlxN materials. The corrosion behavior of WC-Cr1-xAlxN coatings in deaerated 3.5%NaCl solution was investigated by electrochemical tests (galvanic corrosion test, potentiodynamic polarization test, electrochemical impedance spectroscopy) and surface analyses (glow discharge optical emission spectroscopy, X-ray diffraction, atomic force microscopy, scanning electron microscopy). The galvanic current density of WC-Cr0.5Al0.5N coating showed that the current density was decreased gradually with exposure time from a low initial value of approximately 2 to 1.2ßÀ/ß≤ at the end of the exposure time. The results of potentiodynamic polarization tests showed that the WC-Cr0.5Al0.5N coating revealed higher corrosion resistance. In EIS, the WC-Cr0.5Al0.5N coating showed an increased charge transfer resistance (Rct) relative to other specimens with increasing the immersion time. The SEM observation of the cross-sectional view and sample surfaces revealed a homogeneous structure and typical corroded surface.

Characterization of Mechanical Properties of Ni-Graphite Composite Materials: *Ai Liu*[1]; Gerhard E. Welsch[1]; Robert L. Mullen[2]; Dov Hazony[3]; [1]Case Western Reserve University, Matls. Sci. & Eng., Rm. 438 White Bldg., 10900 Euclid Ave., Cleveland, OH 44106 USA; [2]Case Western Reserve University, Dept. of Civil Eng., Cleveland, OH 44106 USA; [3]Case Western Reserve University, Dept. of Electl. Eng. & Compu. Sci., 10900 Euclid Ave., Cleveland, OH 44106 USA

Closed and open cell materials have been made and evaluated by people to get lightweight materials with good mechanical properties. In these materials, cell wall buckling and crashes during loading process are the main reasons of deformation. In order to prevent these phenomena, Ni-coated graphite powders are sintered, which make an interior pressurized close-cell composite. Mechanical and physical properties including density, yield strength, fracture toughness and damping coefficient, are characterized by bending, compression and ultrasonic propagation testing methods. Results show a density of 3.3g/cc and a young?s modulus of around 28Gpa. Also deformation procedure has been studied. Comparison result(mainly based on Gibson and Ashby's book and publications), with solid Ni or other foam materials are gained.

Properties of Plasma Spray Formed Alumina-Titania Ceramic Composite: *C. V. Gokularathnam*[1]; R. Krishnamurthy[2]; M. V. Gopalakrishnan[1]; T. Sornakumar[3]; [1]Indian Institute of Technology, Dept. of Metallurgl. Eng., Madras-600036 India; [2]Indian Institute of Technology, Dept. of Mechl. Eng., Madras-600036 India; [3]Thiagarajar College of Engineering, Dept. of Mechl. Eng., Maduraiñ625015 India

Advances in ceramic processing technology have resulted in a new generation of high-performance structural ceramics exhibiting improved properties through microstructural engineering. The advanced structural ceramics have very good corrosion resistance, wear resistance, high refractoriness, good mechanical strength, high fracture toughness and hardness, ionic conduction, high melting point, low thermal conductivity at high temperature and thermal stability and resistance to thermal shock. In the present study, freestanding bodies of ceramic composites of the alumina-titania (60% wt alumina and 40% wt titania) have been developed by plasma spray technique using a plasma-spraying machine. The XRD analysis indicated that the spray formed samples contain -Al2O3, rutile TiO2 and traces of -Al2O3; and the post heat treatment samples contain -Al2O3, rutile TiO2 and traces of Al2TiO5. The density, hardness, youngís modulus and strength improved by post heat treatment. The SEM observations of the fractured surfaces of the as sprayed samples exhibited quasi plastic mode of failure and the post heat treated samples exhibited more ductile mode of failure.

The Interfacial Reaction of Sn-Ag-Cu/Electroless Ni-P UBM for Flip Chip: *Chang Youl Lee*[1]; Won Jong Cho[1]; Seung Boo Jung[1]; Chang Chae Shur[1]; [1]SungKyunKwan University, Sch. of Metallurgl. & Matls. Eng., 300 Chaunchun-dong, Jangan-ku, Suwon, KyungGi-Do 440-746 Korea

The interfacial reaction between electroless Ni-P UBM and Sn-4Ag-0.5Cu solder was investigated. While intermetallic phase formed in the interface An-3.5Ag solder/Ni UBM was Ni3Sn4, in the case of Sn-4.0Ag-0.5Cu, intermetallic phase formed was Ni-Cu-Sn ternary composition. The Cu atoms in the solder diffused in the solder/UBM interface and formed intermetallic phase. During aging, the thickness of the intermetallic layer was increased and the deposited Ni-P UBM layer (amorphous structure) was crystallized partially. it is considered that P was accumulated because of consumption of Ni according to the interfacial reaction.

Fabrication and Characterization of Pt Ionic Polymer Metal Composite Actuators: *Corey Thomas Love*[1]; Jason Richard Knechel[1]; Bryce Matthew Whited[1]; Donald J. Leo[2]; [1]Virginia Polytechnic Institute and State University, Dept. of Matls. Sci. & Eng., 213 Holden Hall, Blacksburg, VA 24061-0237 USA; [2]Virginia Tech, Ctr. for Intelligent Matls. Sys. & Struct., Dept. of Mechl. Eng., 307 Durham Hall, Blacksburg, VA 24061-0261 USA

Ionic polymer metal composite (IPMC) actuators consisting of an ion exchange membrane (NafionÆ) and embedded Pt electrodes were fabricated using an impregnation/reduction technique. The IPMC actuators are electromechanical devices that produce a large deformation in response to a low applied voltage. Actuators were characterized using scanning electron microscopy, energy dispersive x-ray spectroscopy, and x-ray photoelectron spectroscopy. Blocked force mechanical characterization techniques were employed to analyze the electromechanical properties of the actuators. Results indicate that increased Pt concentration in the impregnation solution increased Pt uptake by the polymer, increased electrical conductivity, and improved deflection. Mechanical response was hindered however by a larger modulus attributed to a larger amount of Pt deposited within the NafionÆ. A correlation was drawn between the Pt uptake and its effect on modulus and mechanical response.

Interfacial Reactions Between In-48Sn Solder and Cu Substrates: *Dae-Gon Kim*[1]; Chang-Youl Lee[1]; Seung-Boo Jung[1]; [1]Sungkyunkwan University, Adv. Matls. & Process Rsrch. Ctr. for IT, 300 Chunchun-Dong, Jangan-Gu, Suwon, Kyonggi-Do 440-746 Korea

The growth kinetics of intermetallic compound layers formed between eutectic In-48Sn solder and bare Cu substrates by solid state isothermal aging were investigated. The interfacial reaction between eutectic In-48Sn solder and bare Cu substrate was investigated at 343, 353, 363 and 373 K for reaction times ranging from 0 to 60 days were used. For the identification of intermetallic compounds, both Scanning Electron Microscopy (SEM) and Electron Microprobe Analysis (EPMA) were employed. The intermetallic compound layer was composed of two phase: Cu18In23Sn9 adjacent to the solder and Cu29In9Sn12 adjacent to the copper. A quantitative analysis of the intermetallic compound layer thickness as a function of time and temperature was performed. The total intermetallic layer exhibited a parabolic growth at given temperature range. Because the values of time exponent(n) have approximately 0.5, the layer growth of the intermetallic compound was mainly controlled by volume diffusion over the temperature range studied.

Recovering Metallic Zinc from Hot-Dip Galvanization Skimmings: *G. ÷zbayoglu*[1]; K. Dikbiyik[2]; [1]Middle East Technical University, Mining Eng. Dept., Ankara Turkey; [2]Turkish Labour Association, Ankara Turkey

The objective of this research is to investigate the concentration possibilities of metallic zinc from the galvanization skimmings (or dry ash) formed during the hot-dip galvanizing process at the Kismet Galvanizing Plant, and the zinc oxide content of pneumatic cyclone underflow product produced at the Mutlu-Metal Plant. X-ray diffraction analysis showed that the galvanization skimmings were mostly composed of metallic zinc and zinc oxide. Zinc hydroxide chloride and carbonate were found in minor amounts. On the other hand, the dominant compound in the pneumatic cyclone underflow product was zinc oxide; other zinc complexes were in the minority. The zinc grades of the galvanization skimmings and pneumatic cyclone underflow product were determined as 76.56% and 71.55%, respectively, by chemical analysis. Wet screen analysis of the galvanization skimmings showed that the zinc grade of the screen fractions decrease with the particle size. A similar behaviour was also observed in the pneumatic cyclone underflow product whose size distribution was determined by Coulter Counter Analysis. Three different procedures were followed in the concentration of galvanization skimmings. In these methods, jig, shaking table, hydrocyclone and flotation were used in different combinations. By the application of the third method the highest zinc grade was obtained as 91.83% with a recovery of 70.89%. As the particle size in cyclone underflow product was fine, tabling, cycloning, and indirect flotation were applied to its concentration. As a result, the concentrate, containing up to 98.40% zinc oxide, could be produced by the highest recovery attainable was 60.69% against 94.70% zinc oxide.

Effects of Changes in Test Temperature on Fatigue Crack Propagation of Laminated Metal Composites (LMCs): *Hala A. Hassan*[1]; J. J. Lewandowski[1]; M. H. Abd El-latif[2]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Eng., Cleveland, OH 44106 USA; [2]Ain Shams University, Dept. of Design & Production Eng., Cairo Egypt

The effects of changes in test temperature from 298 K to 148 K on the fatigue crack propagation of 6090/SiC/20p-6013 and 6090/SiC/25p-6013 laminates tested in the crack arrestor orientation were investigated. The fatigue crack propagation behavior of the individual monolithic and DRA layers was additionally compared to that of the laminates. The fatigue behavior of the laminates was significantly different than that of the individual laminae and was affected by changes in the test temperature as well as by a thermal cycle to low temperatures (e.g. 77 K or 148 K) prior to testing at either 213 or 298 K. In-situ monitoring of fatigue crack growth and post mortem analyses were used to determine the likely source(s) of the effects of changes in test temperature and differences between the LMCs and its constituents.

Thermal Contraction and Expansion Behaviors During Solidification in Continuous Casting Carbon Steels: H. C. Kim[1]; J. S. Lee[1]; *J. H. Lee*[1]; O. D. Kwon[2]; C. H. Yim[2]; [1]Changwon National University, Dept. of Metall., 9 Sarim-dong, Changwon, Kyungnam 641-773 S. Korea; [2]Posco, Iron & Steel Making Rsrch. Grp., 5 Dongchon-dong, Pohang, Kyungbuk 790-360 S. Korea

Continuous casting process has been widely used in steel industries due to its advantages of the improved yield, low cost, and high thermal efficiency. Upon the request of increasing product quality, the mold design to enhance heat transfer from the metal to mold has been an important technology. The air gap between the metal and mold causes subsurface cracks. In order to compensate the air gap in mold design, the thermal contraction is an essential factor. In this study, the thermal contraction/expansion behaviors were examined upon phase transformation between the liquid and delta/gamma phases by a floating plate technique, measuring the levels of the floating plate on the molten liquid during melting and solidifying. It was found that the thermal contraction and expansion behaviors were very dependant on phase transformation of the delta/gamm phases near the melting temperature.

Small-Angle Scattering Using High-Energy X-Rays: *Jonathan D. Almer*[1]; Ulrich Lienert[1]; Dean Haeffner[1]; [1]Argonne National Laboratory, Adv. Photon Source, 9700 S. Cass Ave., Bldg. 431A, Argonne, IL 60439 USA

Acquisition of microstructural information during in-situ (service) conditions is an ongoing need for fundamental materials insight and computational input. One of the primary tools for studying nano-scale inhomogeneities, such as pores and precipitates, is small-angle x-ray scattering (SAXS). We present a novel SAXS camera utilizing high-energy x-rays (HEX) from the Sector 1-ID Advanced Photon Source beamline. Key camera features include an intense HEX brilliance which enables spatial and temporal resolution, and the high penetrating power of HEX (several mm in most materials at 80 keV) which enables in-situ experiments and samples bulk behavior. Furthermore, the SAXS camera can be combined with wide-angle scattering to investigate amorphous and/or crystalline phases. We illustrate these capabilities with in-situ studies of bulk-metallic glasses and coatings at elevated temperatures. Use of the Advanced Photon Source was supported by the United States Department of Energy, Office of Science, Office of Basic Energy Science, under contract number W-31-109-Eng-38.

High Energy Particle Irradiation of YSZ: *Jill N. Johnsen*[1]; Fritz B. Prinz[2]; Joanna R. Groza[1]; [1]University of California-Davis, Cheml. Eng. & Matls. Sci., 1 Shields Ave., Davis, CA 95616 USA; [2]Stanford University, Mechl. Eng., Stanford, CA 94305 USA

High energy particle irradiation was used to introduce dislocations into Yttria Stabilized Zirconia (YSZ) for use as an electrolyte in solid oxide fuel cells. Irradiation with 800 keV electrons was used to introduce vacancies into single crystal YSZ (8 mol% Y_2O_3). Thermal annealing was performed to study the dynamic recovery behavior of the dislocations introduced by irradiation and to determine the edge character of the dislocations. The vacancies coalesced to form vacancy clusters and edge type dislocations upon thermal annealing at 1000°C. The YSZ specimens with irradiation induced dislocations will be studied by Atomic Force Microscopy (AFM) in conjuction with impedance spectroscopy. The goal is to study the surface topography and measure conductivity at the dislocation steps on the specimen surface. The final result will be a measure of oxygen ion conductivity as a function of dislocation density.

Characteristics of Cellular Solidification in Fe-18Cr Ferritic Stainless Steels: *J. H. Lee*[1]; S. K. Kim[2]; J. W. Kim[2]; Y. D. Lee[2]; *S. Liu*[3]; *R. Trivedi*[3]; [1]Changwon National University, Dept. of Metall., 9 Sarim-dong, Changwon, Kyungnam 641-773 S. Korea; [2]Posco, Stainless Rsrch. Grp., 5 Dongchon-dong, Pohang, Kyungbuk 790-360 S. Korea; [3]Iowa State University, Ames Lab., Ames, IA 50011 USA

Fe-Cr alloys have been important structural materials in industry as ferritic stainless steels because of its good corrosion resistance and high strength at high temperatures. Most of studies have been focused on corrosion and mechanical properties in this system, however, very little work has been conducted in solidification behavior. Recently, there has been a discussion on the absolute stability in undercooled melts of Fe-Cr alloys due to its very narrow solid/liquid mushy zone in the phase diagram. In this study, the solid/liquid interface stability was examined by independent control of solidification rates and thermal gradients in 18Cr stainless steels using directional solidification technique. Cellular and planar interfaces could be found easily, rather than dendritic. It was interesting to find out that the dendritic interface changed to cellular as increasing solidification rates. A possibility of the high rate cell, forming at high solidification rates, will be discussed.

Aluminum Oxide Coating on Nickel Substrate by Metal-Organic Chemical Vapor Deposition: *Jun Co Nable*[1]; Malgorzata Gulbinska[1]; Steven L. Suib[1]; Francis Galasso[1]; [1]University of Connecticut, Chem. Dept., 55 N. Eagleville Rd., Storrs, CT 06269-3060 USA

Aluminum oxide thin films were coated onto nickel substrates via chemical vapor deposition (CVD). The aluminum oxide was produced by pyrolysis of a metal acetylacetonate precursor. This reaction was done at relatively low temperatures, from 435∞C to 550∞C to deposit a thin film of aluminum oxide. The coating provided protection against isothermal oxidation at elevated temperatures. Resistance to oxidation was obtained for all of the coated substrates. Surface morphology, however, changed when heated for a prolonged time. Samples coated at 500∞C and 550∞C are effective up to 900∞C whereas samples coated at 435∞C were good up to 800∞C. The metal oxide compositions and phases were studied by X-ray diffraction and EDAX. The surface properties of the deposited thin films were investigated with scanning electron microscopy (SEM).

Primary Carbides in Tool Steel and High Speed Steel: *Karin Frisk*[1]; [1]Swedish Institute for Metals Research, Drottning Kristinas v‰g 48, Stockholm SE_11428 Sweden

Primary carbides formed in tool steel due to micro-segregation have bee studied. In commercial materials the primary carbides are dissolved by heat treatments. To predict time and temperatures needed for the homogenising heat treatments, the size and composition of the carbides is needed. The composition of the steel, but also the solidification rate, will influence the type and composition of carbides. Experimental investigations of primary carbides in high-speed steel samples solidified with varying rates are presented. The solidification rate influences the size of the carbides and also the composition and the type of carbide. The influence on carbide type is especially pronounced for very high solidification rates obtained in gas atomised powder. The results are interpreted and using equilibrium calculations and diffusion calculations, with Thermo-Calc and DICTRA. Full diffusion calculations and approximations are tested. It was found that if back diffusion of C is taken into account, a Scheil simulation for all other elements successfully predicts a realistic micro-segregation in temperature and composition.

Theoretical Simulation and Experimental Investigations of the Surge Response of a Tower Model of Vertical Conductor: *Md. Osman Goni*[1]; Hideomi Takahashi[1]; [1]University of the Ryukyus, Matl. Structl. & Energy Eng., 1 Senbaru, Nishihara, Okinawa 903-0213 Japan

The steel tower surge impedance is one of the basic parameters for the anti-lightning design. Therefore, since Jordan, a lot of experiments and theories are proposed, however, there are no established theories. This is the stumper which is known as ìthe vertical conductor problemîî in the present-day electric engineering. Therefore, it is given up to make the problem clear, and the present-day situation is about to adopt the numerical analysis which can explain the phenomenon comparatively well. As for surge impedance on a tower model of a vertical conductor, we have a theoretical formula by Lundholm. The theory of Lundholm looks like to be a perfect theory, however, this formula does not coincide with the experimental results. Thus, there is the strange situation that the Jordanís formula of the wrong theory agrees with the experiments more correctly. Hara et al derived experimental formula. Moreover, one of the authors proposed a theoretical formula of surge impedance; considering the existence of ground su rface, and without ground surface. The former formula is very similar to the experimental formula of Hara et al. In this research, these theoretical formulas of surge impedance are examined by the simulation analysis of vertical conductor with the help of Numerical Electromagnetic Code (NEC-2) and the experimental results of that. In the measurement at an actual tower, however, it is difficult to stretch a current lead wire vertically from the tower top, where the current lead wire acts as a vertical lightning channel. Measurements on reduced-scale models are more economical than those on full-sized towers, and are flexible in setting up various experimental arrangements. It is, however, not easy to maintain the accuracy of the measurement, since the geometrical size of the measuring devices is large relative to the measured system. The simulation and experimental analysis of surge response are carried out in the several arrangements of the current lead wire and the current source: (i) vertical and at the top of vertical conductor, (ii) vertical and a little far from the top of vertical conductor, and (iii) horizontal and far from the top of vertical conductor. In all the cases, the voltage measuring wire is placed at the perpendicular to the current lead wire. Each of the arrangement of the current lead wire affects the measured surge impedance of the vertical conductor and these will be explained in this research in detail. If a travelling wave propagates along the vertical conductor at the velocity of light, the reflected wave from the ground should return to the top of the vertical conductor just after the round-trip time of the travelling wave in the vertical conductor. We ascertained these phenomenon in the both measured and computed results. The agreement between the measured and computed results is also quite well with minor difference. The experimental set up of the model to be analyzed in this paper is verified with the simulation result of the equivalent circuit model by the EMTP.

On the Effect of Indentersí Shape in Nanoindentation: *Reza A. Mirshams*[1]; Padma Parakala[1]; Seifollah Nasrazadani[1]; Kun Lian[2]; [1]University of North Texas, Dept. of Eng. Tech., Denton, TX 76303 USA; [2]Center for Advanced Microstructure and Devices (CAMD), Baton Rouge, LA 70803 USA

Nanoindentation tests have been routinely used to evaluate the mechanical properties of thin films; coatings, and micro phases. Berkovich tip has been used extensively in the investigations. We report the results of a study on the effect of geometry of nanoindenter tips on the deformation mechanisms and the characterization of mechanical properties of gold and nickel. Three different tips have been used in this study: Berkovich, cube corner, and spherical end cone. Sputtered thin gold films on Si substrates and electrodeposited nickel, as thin films on pure Cu substrates and as thin sheets, were used in this investigation. Microstructure studies were performed by using SEM, and XRD. The results indicate strong effect of the geometry of indenters on the evaluation of mechanical properties. Different mechanisms of deformation and methods for correction of results are discussed.

Materials Selection for Hydrochloric Acid Storage Tanks: *Rafic Moubarac*[1]; [1]Experco Composites Inc., 4946 Ste Suzanne St., Pierrefonds, Quebec H8Y 1Z9 Canada

This paper will review the choice of materials of construction for large hydrochloric acid storage tanks. It will compare corrosion and high temperature resistance of these materials to concentrated hydrochloric acid, as well as life cycle and cost. It will explain the permeation and blistering mechanisms in fiberglass reinforced plastic tanks, and compare resins and veils. Global case histories in the mining, smelting, refining and hydrometallurgy industries will be presented.

Synthesis and Study of NiTi-Ta Alloys by Mechanical Alloying: *Srikanth Kanchibhotla*[1]; [1]Florida International University, Mech. Eng., 10555 W. Flagler St., Miami, FL 33174 USA

Shape Memory Alloys (SMAs) are a unique set of alloys that display recoverable stress-induced and temperature-induced deformations while undergoing reversible phase transformation. The use of NiTi shape memory alloys has been proposed and demonstrated for many medical applications. However, due to poor X-Ray visibility and narrow transformation temperature hysteresis, tantalum is added as a ternary element which causes significant changes in the properties of the alloy. By starting with pure powders of Ni,Ti and Ta, NiTi-Ta alloys were synthesized with nanolayered structure and refined grain size using mechanical alloying technique. The effect of Ta addition on the microstructure and phase transformation temperature of Ni50Ti50-xTax and (Ni51Ti49)1-xTax (x= 2~20%) have been studied by means of optical microscopy, SEM and DSC. The change of microstructure and the distribution of the constitutional phases under various heat-treatment processes are being investigated and will be reported.

Solidification Behaviors with N Contents and Solidification Rates in Duplex Stainless Steels: *Hyunchul Kim*[2]; Jaehyun Lee[2]; Jinil Son[1]; Sangshik Kim[1]; [1]Gyeongsang National University, Matls. Sci. & Eng., 900 Gazwa-dong, Chinju, Kyongnam 660-701 Korea

Duplex stainless steel has been a considerable research subject due to its good corrosion resistance by the austenite phase and high strength by the ferrite phase. These properties could be also enhanced in addition of N in duplex stainless steels. Most of researches have been mainly concentrated on corrosion properties, mechanical properties with heat-treatment, and alloying element effect. As castings increase for applications in industry, such as pumps and valves in petro-chemical plants, solidification behaviors with solidification rate and important alloying element of N content have been studied in the 25Cr-5Ni-2Mo-3Cu system alloys by directional solidification technique. Directional solidification experiments were carried out with solidification rate, N content, and under air to 1 N atmospheric pressure. As N content and N pressure increased, the volume of austenite phase increased and its shape went to be round. As solidification rate increased, the austenite phase became finer and randomly distributed.

Passivation of Pyrite: The Relation of the Hydrophobic Double Layer to Magnetic Phases on the Mineral Surface: *Charles I. Richman*[1]; [1]University of Nevada at Reno, Dept. of Metallurgl. & Matls. Eng., MS 388, Reno, NV 89557 USA

Passivation of exposed pyrite formations and gangue is an economic operation, useful for preservation of water quality and wildlife. The study of induced film formation on pyrite is important theoretically to the understanding of why passivation in sulfide minerals occurs. Magnetometer, and x-ray diffraction measurements show that leached CP develops a higher magnetic saturation point after the formation of pyrrhotite. Pyrrhotite is not formed when photo-catalytic additives are used with sulfuric acid. Our research shows that magnetic minerals and hydroxides are formed on the pyrite surface with the addition of passivation agents. Without the addition of passivation agents, a runaway sulfide oxidation reaction can occur on pyrite. Under runaway conditions, more sulfate is produced, and the system becomes more acidic, further causing the breakdown of the sulfide mineral. The formation of magnetic minerals as shown by magnetic susceptibility and x-ray diffraction data prevents further oxidation. Electrochemical Impedance Spectroscopy (EIS) is used to model the passivated double layer on the pyrite surface. The results show that in the passive state the relation the water molecules in the electrolyte are non-aligned and the surface is hydrophobic. A theory is presented to show the relationship to the hydrophobic state and the presence of magnetic phases on the pyrite surface.

Improving High Resolution TEM Images via Low Energy Ion Milling Specimen Preparation: *Shane P. Roberts*[1]; [1]South Bay Technology, Inc., 1120 Via Callejon, San Clemente, CA 92673 USA

The investigation of semiconductor materials using high resolution transmission electron microscopy (HRTEM) is a necessity in todayís manufacturing environment. The high resolution and combined analytical capabilities make TEM an important tool used for semiconductor device fabrication. As a result, TEM sample preparation plays an important role in todayís manufacturing and research environment. For semiconductor materials the use of the focused ion beam (FIB) has become a common method used in the preparation of TEM samples. Although the FIB is extremely useful, it does have some drawbacks, especially in high resolution TEM applications. The highly energetic, tightly focused ion beam creates amorphous damage in the crystalline sample, limiting the information that can be obtained from the sample.This problem can be alleviated by reducing the incident ion energy used during the milling process. A novel technique of using low energy argon ion milling to remove amorphous damage is presented.

Applications of Low Energy Ion Milling Technology for TEM Specimen Preparation: *Shane P. Roberts*[1]; [1]South Bay Technology, Inc., 1120 Via Callejon, San Clemente, CA 92673 USA

The investigation of semiconductor materials using high resolution transmission electron microscopy (HRTEM) is a necessity in todayís manufacturing environment. The high resolution and combined analytical capabilities make TEM an important tool used for semiconductor device fabrication. As a result, TEM sample preparation plays an important role in todayís manufacturing and research environment. For semiconductor materials the use of the focused ion beam (FIB) has become a common method used in the preparation of TEM samples. Although the FIB is extremely useful, it does have some drawbacks, especially in high resolution TEM applications. The highly energetic, tightly focused ion beam creates amorphous damage in the crystalline sample, limiting the information that can be obtained from the sample. This problem can be alleviated by reducing the incident ion energy used during the milling process. A novel technique of using low energy argon ion milling to remove amorphous damage is presented.

The Use Phase in the Aluminum Mass FlowñAn Approach for Integration: *Traute Koether*[1]; Bernd Friedrich[1]; [1]Aachen University of Technology-Germany, IME Process Metall. & Metal Recyling, Intzestr. 3, Aachen 52056 Germany

In mass flow analyses the use phase is often not adequately integrated. Above all user-related effects, life-time of products or product-systems are frequently unconsidered. Looking at the entire process chain the total demand for aluminum is important. But balancing the environmental impacts of the use phase of aluminum-bearing products such as cars, trains, window frames, or beverage cans three other targets win importance. These are the identification of environmental impacts during the use phase itself and its integration in the process chain. Using this data the third target aims at case studies which point out various user- related effects. For this purpose mass flows and induced environmental impacts of the particular product use, startup, maintenance and repair have to be quantified separately using LCA. For this targets the consideration of the multifunctional material properties is important which requires the choice of a suitable functional unit in order to reproduce all parameters. Furthermore, those mass flows have to be considered which are influenced by the user. Besides socio-economic factors the individual user behavior is determined by exogenous parameters such as distribution and availability of product information. Additionally, it is important to include the life-time of products, because the effects can only be shown after a certain time of usage or, due to the high life time of some products, by summation. On the basis of examples from the aluminum sector particular spectra of the usage are represented with focus either on user behavior, technological potential, recycling ability or life span.

Fabrication of Bi-Te Thermoelectric Devices using RF Sputtering Target prepared by MA-PCS Process: *Toshiyuki Nishio*[1]; Keizo Kobayashi[1]; Akihiro Matsumoto[1]; Kimihiro Ozaki[1]; Mitsuyoshi Sakai[2]; Akio Yamaguchi[1]; [1]National Institute of Advanced Industrial Science and Technology, Inst. for Structl. & Eng. Matls., 2266-98 Anagahora Shimoshidami, Moriyama-ku, Nagoya 463-8560 Japan; [2]Kitagawa Industries Co., Ltd., 1-27 Hikisawa kamiya-cho, Kasugai Japan

A p-type thermoelectric film of (Bi2Te3)0.25(Sb2Te3)0.75 and an n-type thermoelectric film of Bi2Te2.7Se0.3 were fabricated by radio frequency RF sputtering. The target for the sputtering were prepared by mechanical alloying(MA) and pulsed current sintering(PCS) process. (Bi2Te3)0.25(Sb2Te3)0.75 and Bi2Te2.7Se0.3 were synthesized by MA of Bi, Te, Sb and Se powders as starting materials for 10 hours or less. The obtained powders were consolidated by PCS process at 653K under the pressure application of 13MPa. After the compacts were connected to copper backing plates to prepare sputtering targets, thermoelectric films were succesfully fabricated by RF sputtering process on a polyimide substrate. The eight pairs of p-n elemental device with the size of about 30mm in thickness and 20mm in length prepared by this process generated an electromotive force of 56mV, when the temperature difference 30K were given to the devices.

Residual Stress User Center Facilities at ORNL: *Thomas R. Watkins*[1]; Camden R. Hubbard[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., High Temp. Matls. Lab., MS 6064, Bldg. 4515, Oak Ridge, TN 37830-6064 USA

The Residual Stress User Center assists academic and industrial users to characterize the macro and micro residual stresses in materials and systems. The facilities primarily utilize laboratory x-ray, synchrotron x-ray, and neutron diffraction methods and are complemented by strain gauging, electropolishing, coordinate measurement machine, x-ray furnaces, and micro Raman systems. Recent upgrades of the large specimen x-ray and neutron diffraction mapping systems aim to achieve an order of magnitude improvement in capabilities and speed. These systems permit automated surface and through thickness residual stress mapping on small research and large industrial specimens. The high-flux, parallel beam synchrotron beam line X14A at NSLS is particularly valuable for measurement of stress on highly curved specimens and for grazing incidence studies. Two 4-axis diffractometer, one coupled with a rotating anode generator and multilayer x-ray optics, provide additional capabilities for laboratory based measurements. Access to these facilities is via the HTML User Program (www.ms.ornl.gov/htmlhome/rsuc).

Joints Characteristic of Friction Welded Copper/Medium Carbon Steel: *Won Bae Lee*[1]; Seong Yeon Kim[1]; Dae Up Kim[2]; *Yun Mo Yeon*[3]; *Seung Boo Jung*[1]; [1]SungKyunKwan University, Adv. Matls. & Process Rsrch. Ctr. for IT, 300 Cheoncheon-dong, Jangan-gu, Suwon, Gyounggi-do 440-746 Korea; [2]Hyundai Mobis, Rsrch. Inst., Youngin, Gyounggi-do 449-910 Korea; [3]Suwon-Science College, Automatic-Welding Eng., Whasung, Gyounggi-do 445-742 Korea

The metallurgical and mechanical properties of friction welded joint copper(OFC)/medium carbon steel has been studied in the work. The joint strength increased with increasing upset pressure and decreased with increasing friction time because thermal softened region existed in copper part. This region was changable with friction welding conditions. The fracture was occurred at heat affected zone and copper base metal. Hardness near the interface was slightly lower than that of base metal at copper side because of dynamic recrystallization. But the steel part showed the slighly higher value than that of base metal. This reason caused by phase transformation from mixed ferrite and pearlite structure to martensite and fine pearlite in the interface. This stucture hardened the steel part of interface. The maximum joints strength, 330MPa, appeared at the condition of upset pressure, 325MPa and frcition time 0.1 sec.

Effect of Cr, CO, W, Cu, Ni and Ca on the Corrosion Behavior of Low Carbon Steel in Synthetic Ground Water: *Yoon Seok Choi*[1]; Seung Jae You[1]; Jae Joo Shim[1]; Jung Gu Kim[1]; [1]SungKyunKwan University, Adv. Matls. Eng., 300 Chunchun-dong, Jangan-Gu, Suwon 440-746 S. Korea

The aqueous corrosion characteristics of 1% Cr-steel alloyed with small amounts of Co, W, Cu, Ni and Ca in synthetic ground water was studied by electrochemical corrosion tests (potentiodynamic test and electrochemical impedance spectroscopy (EIS) measurements) and analytical techniques. Neither carbon steel nor new-alloyed steels showed passive behavior in synthetic ground water. Steel containing Co, W, Cu, Ni and Ca showed higher corrosion resistance than 1% Cr-steel in the potentiodynamic tests. EIS measurements showed that the Nyquist plot presented initially one time constants, and then changed to two time constants as growing the rust layer. Furthermore, the resistance of the rust (Rrust) increased with adding the alloying elements, such as Co, W, Cu, Ni and Ca, and that the structural factor of the rust became predominant in Rrust. The better corrosion resistance of the new-alloyed steel is attributed to protectiveness of the surface layer. The corrosion products were examined using scanning electron m icroscopy

(SEM), electroprobe x-ray microanalysis (EPMA) and X-ray photoelectron spectroscopy (XPS). The results of EPMA indicated that Cr, Cu, Ni and Ca were concentrated in the inner region of the rust layer, while Co and W were distributed all over the rust layer. XPS results showed that Co existed as a trivalent oxide in the rust layer and W in the rust appeared in the form of a WO4 compound. Consequently, these compounds act as a factor for corrosion resistance in aqueous solutions, and Cu, Ni and Ca containing specimen revealed the highest corrosion resistance due to the contribution to inner rust layer formation at the initial state of corrosion.

International Symposium on Gamma Titanium Aluminides: Poster Session

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee

Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

Monday 7:00-9:00 PM	Room: Upper Level City-Side Corridor Lobby
March 3, 2003	Location: San Diego Convention Center

Session Chairs: Andrew H. Rosenberger, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA; Helmut Clemens, GKSS, Inst. for Matls. Rsrch., Geesthacht D-21502 Germany; Young-Won Kim, UES Inc., Matls. & Processes Div., Dayton, OH 45432 USA

TEM and HRTEM Investigation of Intragranular Lamellar Transformations in the Ti-48 at. % Al Alloy: *Williams Lefebvre*[1]; Annick Loiseau[2]; Alain Menand[1]; [1]UniversitÈ de Rouen, Site Universitaire du Madrillet, FacultÈ des Sciences et des Techniques, Ave. de líUniversitÈ, BP 12, Saint Etienne du Rouvray, Cedex 76801 France; [2]Onera, LEM CNRS/Onera, BP 72, 29 Ave. de la Division Leclerc, Chatillon 92322 France

The aim of this study is to investigate two types of lamellar transformations involved in the Ti-48 at. % Al model alloy. The first one is designated as a classical lamellar transformation. It is involved for a low cooling rate from the single alpha-phase field. The second one is the ultrafine lamellar transformation, which occurs when the alloy is quenched from the single alpha-phase field. According to original step heat treatments, new features of these two transformations have been revealed. We have established that the ultrafine transformation starts with the intragranular precipitation of ìGP-zones likeî gamma plates in the alpha2-phase and is followed by a growth according to a ledge mechanism. As for the classical lamellar transformation, our observations revealed the interfacial precipitation and growth of gamma lamellae at lamellar interfaces, outside grain boundaries.

Thermo-Physical Properties of a Ti-44Al-8Nb-1B Alloy in the Solid and Molten Conditions: *Richard A. Harding*[1]; Robert F. Brooks[2]; Gernot Pottlacher[3]; J. Brillo[4]; [1]The University of Birmingham, IRC in Matls. Procg., Edgbaston, Birmingham B15 2TT UK; [2]National Physical Laboratory, Queens Rd., Teddington, Middlesex TW11 0LW UK; [3]Technische Universit‰t Graz, Institut f¸r Experimentalphysik, Graz A-8010 Austria; [4]DLR-German Aerospace Center, Institut f¸r Raumsimulation, Porz-Wahnheide, Linder Hˆhe, Kˆln 51147 Germany

The commercial implementation of TiAl alloys is currently hampered by difficulties in melting and casting them. Computer modelling has much to offer for helping to understand and optimise the widely-used Induction Skull Melting and investment casting techniques, but its application is limited by the lack of information of the thermo-physical properties of TiAl alloys. Obtaining data in the molten condition is particularly difficult due to the high reactivity of these alloys, and special containment techniques are required. This paper will briefly describe the methods used by a number of European institutes including the pulsed heating (or exploding wire) technique, differential scanning calorimetry, levitation drop calorimetry, a laser flash technique and a miniature electro-thermomechanical test system. Results will be presented for the properties of a Ti-44Al-8Nb-1B alloy in the solid and molten conditions, including the density, electrical resistivity, enthalpy and thermal diffusivity.

Near-Gamma TiAl Precision Casting and its Optimization: *Antonin Dlouhy*[1]; Ladislav Zemcik[2]; Robert Valek[1]; [1]Institute of Physics of Materials/AS CR, Mechl. Properties, Zizkova 22, Brno 616 62 Czech Republic; [2]Brno University of Technology, Dept. of Castg., Technicka 2, Brno 616 69 Czech Republic

Two drawbacks are associated with the Induction Skull Melting process often used in the near-gamma TiAl precision casting technology: (i) a relatively small achievable superheat which frequently causes misruns in thin-wall castings and (ii) a high investment cost necessary to built up the melting unit. Therefore, a more common melting route based on the ceramic crucible has been reconsidered to overcome both disadvantages mentioned. The paper reports on results of trial casting experiments performed using this common melting route. A preliminary metallographic study indicated that, while the cast volume is free of the contamination, which would result from the reaction between the melt and the crucible, there still are internal cracks generated during the cast solidification and cooling. Modelling and experimental effort is made to optimise the key parameters of the process (the amount of superheat, the mould temperature and the cooling kinetics) to improve the quality of casts.

Joining Ti-48Al-2Cr-2Nb by Diffusion Brazing: *AnÌbal Reis Guedes*[1]; Ana Maria Pinto[1]; Manuel Fernando Vieira[2]; Filomena Viana[2]; [1]Universidade do Minho, Dept. de Engenharia Mec‚nica, Campus de AzurÈm 4800-058 Guimar„es Portugal; [2]Faculudade de Engenharia da Universidade do Porto, Dept. de Engenharia Metal˙rgica e Materiais, GMM/IMAT, Rua Dr. Roberto Frias, Porto 4200-465 Portugal

Ti-48Al-2Cr-2Nb was joined by diffusion brazing using a Ti/Ni/Ti clad-laminated filler alloy. Experiments were conducted in the temperature range of 600 to 1050∫C in order to assess the chemical and microstructural evolution (1) within the filler, up to 900∫C, prior to the reaction with the γ-TiAl alloy, (2) across the interface for joining at 1000 and 1050∫C under a 10 min holding stage. The microstructure and the chemical composition of the interfaces and of the filler alloy were studied by scanning electron microscopy (SEM) and by energy dispersive X-ray scans (EDS), respectively. In the course of the heating stage, several reaction layers were formed within the filler due to the interdiffusion of Ti and Ni atoms. Ultimately, the interdiffusion within the filler alloy led to the formation of liquid phase that wets and reacts with the γ-TiAl alloy. The reaction between the liquid and the γ-TiAl alloy produced layered interfaces that are essentially composed of α2-Ti3Al, TiNiAl and TiNi2Al intermetallic compounds.

Determination of Partition Coefficient of Chromium in $\alpha+\gamma$ and $\alpha 2+\gamma$ Ti-47Al-2Cr and Ti-48Al-2Cr-2Nb Alloys: *Anne Ricaud*[1]; Alain Menand[1]; Thomas Marc[2]; [1]Universite de Rouen, GPM-UMR CNRS 6634, Ave. de líUniversite BP 12, Saint Etienne du Rouvray 76801 France; [2]ONERA, DMMP, BP 72, Chatillon 92322 France

Segregation of Cr has been reported to appear to α2-γ and γ-γ interfaces in engineering heat treated γ-based TiAl alloys. In this paper, the partition behaviour of chromium was investigated by tomographic atom probe (TAP) in isothermal aged Ti-47Al-2Cr alloy (homogenized 4h at 1400∞C, heat treated 2h at 1270∞C, 16h at 1200∞C and 168h at 1000∞C and then oil quenched) and in aged GE type alloy Ti-48Al-2Cr-2Nb. The Ti-47Al-2Cr alloy structure is fully lamellar whereas GE alloy shows duplex microstructure. The Cr distribution between α2 and γ phases has been examined. Several α2-γ interfaces have been analyzed. In alloys aged in ($\alpha+\gamma$) field, the partition coefficient of chromium between α and γ phases has been determined to be approximatively the same for ternary and GE alloys. Conversely, for alloys heat treated in ($\alpha 2+\gamma$) field, chromium is nearly equally distributed in both phases. Besides, chromium segregation has been observed at α2-γ interface. This chromium coverage was observed to be discontinuous.

Comparative Role of Dislocation Blocking Mechanisms (Linear and Point) in the Temperature Anomaly of the Yield Stress of Intermetallics: *Bella A. Greenberg*[1]; Mikhail A. Ivanov[2]; [1]Russian Academy of Sciences, Ural Div., Inst. of Met. Phys., 18 S. Kovalevskaya Str., Ekaterinburg GSP-170, Sverdlovsk Region 620219 Russia; [2]National Academy of Sciences of Ukraine, Inst. of Met. Phys., 36 Vernadsky Av., Kiev 03680 Ukraine

Characteristic elements of the dislocation structure in intermetallics are presented by both segments blocked along some lines and pinning points of a dislocation line. The latter are associated with kinks (macrokinks and microkinks) and are observed in Ni3Al (on superdislocations) and TiAl (on superdislocations and single dislocations). Let it be emphasized that similar pinning points are found in semiconductors too. The interaction of kinks was considered when segments having a certain orientation were blocked. Thermally activated blocking of dislocation segments was shown to be the dominant factor determining the yield-stress anomaly. Oppositely, the appearance of pinning points was an accompanying feature only. It was established why pinning points and the temperature anomaly of yield stress were observed simultaneously in intermetallics, while the said anomaly was not observed in semiconductors. Blocking mechanisms of different types of dislocations were determined. Plastic deformation equations were derived taking into account the aforementioned mechanisms. Conditions, under which a deviation from the indestructible character of dislocation barriers still enabled observing the temperature anomaly of yield stress, were specified.

Joining of Sheet Based Titanium Aluminide Products: *Bernhard Tabernig*[1]; Heinrich Ewald Kestler[1]; [1]PLANSEE AG, Tech. Ctr., Reutte 6600 Austria

Due to material properties like high specific stiffness and strength combined with good oxidation behaviour titanium aluminide alloys have a great potential for high temperature applications in aerospace and automotive industries. For introducing this material class in practical service, however, amongst other issues reliable joining techniques have to be established. This work reviews the research activities and progress in the development of joining techniques for gamma TiAl, e.g. beam welding techniques, solid state joining as well as brazing, at Plansee AG. In our paper we present mainly results gained on sheet based gamma TiAl products. Emphasis will be put on integrity and the resulting mechanical properties of the joints. For example crack free welds in 1mm thick sheet material have been achieved by an advanced EB-welding technique. Laser spot welding of thin gamma TiAl foils was successfully performed in a specially designed shielding gas chamber. Brazing and diffusion bonding of gamma TiAl honeycomb structures was investigated and first test samples were produced. Finally an outlook is given on the industrial feasibility, upscaling considerations as well as possibilities to transfer these technologies to specific applications.

Transient Liquid Phase Joining of Gamma Met PX: *Daniel Alan Butts*[1]; William F. Gale[1]; Tao Zhou[1]; [1]Auburn University, Matls. Rsrch. & Edu. Ctr., 201 Ross Hall, Auburn, AL 36849 USA

The well-known process of Transient Liquid Phase (TLP) bonding has been widely employed in primary fabrication, casting repair, and post-service refurbishment operations for numerous applications, including gas turbine engine components. TLP joining offers the advantage of producing bonds with microstructures and mechanical properties similar to those of the substrate materials. Hence, TLP bonding is very well suited to joining intermetallics with carefully tailored microstructures, such as Gamma Titanium Aluminides. The research presented in this paper contrasts current work on joining of Gamma Met PX with earlier studies by the authors of 48-2-2 (Ti-48 at.% Al-2 at.% Cr-2 at.% Nb). Although these two systems behaved very differently, high quality bonds were produced in both cases using a suitable composite interlayer (consisting of a liquid former and non-melting phase). This paper also discusses a simple method by which the composite interlayer can be mechanically deposited on the substrate, thus simplifying the process for industry.

Synthesis of Gamma-TiAl/Al2O3 In-Situ Composites: *Deliang Zhang*[1]; Zhihong Cai[1]; [1]University of Waikato, Matls. & Process Eng., PB 3105, Hamilton New Zealand

Gamma titanium aluminide intermetallic compound has been the centre of attention of many materials researchers due to its promising potential of being applied widely as a high performance light material. In contrast, the interest in titanium aluminide based intermetallic-ceramic composites has not yet been very strong. This may change in the future, as development of titanium aluminide based composites will offer opportunities to achieve better properties for particular applications and/or cut down the cost of producing final components. At University of Waikato, we have developed a process which can be used to synthesize low cost gamma-TiAl/Al2O3 in-situ composites using Al and TiO2 powders as raw materials. This process utilizes the fact that Al can react with TiO2 forming Ti3Al or TiAl and Al2O3, and involves a combination of high energy mechanical milling, thermal treatment and sintering. Even though the TiAl matrix of the composites contains a substantial amount of oxygen (up to 5at%) which makes the TiAl phase more brittle, this process is attractive due to its great potential in producing relatively low cost TiAl/Al2O3 composite materials and near net shaped components. This paper will describe the microstructures obtained at different stages of the process and the mechanical properties (bending strength, hardness and fracture toughness) of the bulk TiAl/Al2O3 composites with two different volume fractions of Al2O3 produced by pressureless sintering and hot isostatic pressing of the composite powders. The effects of different factors on microstructures and mechanical properties of the composites will also be discussed.

Milling, Consolidation and Mechanical Properties of Ti-48at%Al-2at%Nb/SiC Nanocomposites: *Deliang Zhang*[1]; Jeremy P. Wu[1]; Jing Liang[1]; [1]University of Waikato, Dept. of Matls. & Process Eng., PB 3105, Hamilton New Zealand

As part of our research on titanium based high performance materials, we produced Ti-48at%Al-2at%Nb/SiC (TiAl-2Nb/SiC) composites containing 10vol.% of nanometer scale SiC particles. The process used involves high energy mechanical milling of a mixture of titanium, aluminium, niobium and silicon carbide powders to produce TiAl-2Nb/SiC nanocomposite powder, and consolidating the composite powder by using pressureless sintering and/or hot isostatic pressing (hipping). The study focuses on the effect of milling conditions on the microstructure of the composite powder particles, the effect of sintering and hipping conditions on the microstructures of the bulk composite produced and the effect of microstructure on mechanical properties including high temperature mechanical properties. This paper is to describe and discuss the results of this study. In particular, the role that the SiC particle size and the interfacial reaction between SiC and the TiAl-2Nb matrix play in determining the mechanical properties of the composite will be discussed.

The Effect of Long Time Elevated Temperature Exposure on the Microstructure and Creep Behavior of Ti-48Al-2Cr-2Nb-xMo Titanium Aluminides: *Eric A. Ott*[1]; Tresa M. Pollock[2]; [1]GE Aircraft Engines, Matls. & Process Eng., 1 Neumann Way, MD M89, Cincinnati, OH 45215 USA; [2]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., H. H. Dow 2042, Ann Arbor, MI 48109 USA

The creep behavior of gamma-based titanium aluminide alloys is dependent upon bulk alloy composition, microstructure, phase morphology, and stability. This study examines Ti-48Al-2Cr-2Nb-xMo alloys to assess the effect of 760C thermal exposures for up to 2000 hours on creep behavior. Creep testing indicates heat treatments which involve a low temperature age or a slow final cooling rate result in minimal changes in minimum creep rate with exposure while other heat treatments result in up to one order of magnitude increase in the minimum creep rate after long time exposure. Electron microscopy and image analysis indicates that some microstructural changes occur during exposure but these are unlikely to result in the minimum creep rate changes observed. Analysis of the gamma phase lattice parameter and composition indicates that some changes occur during exposure and these changes are likely to have a significant effect on the minimum creep rate.

Cost-Effective Production of Powder Metallurgy Gamma Titanium Aluminide Sheet: Vladimir S. Moxson[1]; *F. H. (Sam) Froes*[2]; Susan L. Draper[3]; [1]ADMA Products, Inc., 8180 Boyle Pkwy., Twinsburg, OH 44087 USA; [2]University of Idaho, IMAP, Mines Bldg. Rm. 321, Moscow, ID 83844-3026 USA; [3]NASA Glenn Research Center, 21000 Brookpark Rd., MS 49-1, Cleveland, OH 44135 USA

It is extremely difficult to fabricate thin gage Titanium Gamma Aluminide (TiAl) sheet and foil products because of their inherent low ductility. A cost-effective powder metallurgy approach to produce thin gage TiAl and composite Ti-6Al-4V/TiAl/Ti-6Al-4V flat products by using an innovative process will be reported in this paper. The microstructures and properties of thin gage TiAl will be presented along with the microstructures of composite Ti-6Al-4V/TiAl/Ti-6Al-4V material. This work demonstrates a cost-effective method for producing gamma titanium aluminides and composite materials for various advanced applications including heat-shields and structural honeycomb structures.

Preliminary Results on Damage Tolerance in Fatigue Tests of γ-TiAl Valves: *Francesco Marino*[1]; Marco Guerra[2]; Andrea Rebuffo[3]; Massimo Rossetto[4]; [1]Politecnico di Torino, Scienza dei Materiali e Ingegneria Chimica, 24 corso Duca degli Abruzzi, Torino 10129 Italy; [2]C.I.T.T., Reactive Metals Technologies, 33/H, via Aquileia, Baranzate di Bollate, Milano 20021 Italy; [3]Phitec Ingegneria srl, Business Palace, Rivoli, Torino 10098 Italy; [4]Politecnico di Torino, Meccanica, 24 corso Duca degli Abruzzi, Torino 10129 Italy

Internal combustion engine valves produced via induction skull melting and centrifugal casting in a permanent mold were submitted to mechanical fatigue tests. These valves, with nominal chemical composition Ti-47Al-2Nb-2Cr, have fully lamellar microstructure with a mean grain size of 300 mm and differ each other for the presence of internal defects: shrinkages and pores. A soundness hierarchy of the valves was established on the basis of defects in term of morphology, size and distribution shown by radiographic examinations performed according to ASTM E 1742-95 standard. To evaluate the damage tolerance of the valves some fatigue tests were performed using a resonant machine Amsler type 100 kN 10HFP 422 properly equipped to simulate the working conditions with exception for the impact on the valve seat and the compression step. The test was performed in

POSTERS

tension-tension mode, the applied stress range was 140 MPa under a stress ratio R of 0.026.

The Precipitation of Discrete α_2 Particles Within the γ Phase of TiAl Based Alloys: *F. Meisenkothen*[1]; R. Banerjee[1]; G. B. Viswanathan[1]; H. L. Fraser[1]; [1]Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43016 USA

The gettering of interstitial contaminants, by the α_2 laths in fully lamellar TiAl based alloys, has been shown to be responsible for the enhanced slip activity that is observed in the γ laths of these alloys. However, absorption of the interstitial impurities from the γ, limits the number of active slip systems that can operate in the α_2 phase. The result is that the α_2, an already brittle phase, becomes even less likely to undergo plastic deformation.The brittle α_2 laths constrain the γ phase, and the overall ductility of the fully lamellar material is thus reduced. However, by suitably engineering the sequence of phase transformations leading to microstructural development in TiAl based alloys, it is possible to employ the gettering benefits provided by the α_2 phase, while eliminating the constraints imposed by it on the γ phase. The addition of small amounts of the ternary alloying element, Ta, makes it possible to subject the material to a true γ solution heat treatment by adequately modifying the region of γ phase stability in the Ti-Al-Ta phase diagram. Upon cooling from the single phase γ region, multiple variants of the α_2 phase precipitate directly from the γ phase allowing for the formation of discrete α_2 precipitates in a γ matrix.

Effect of Rolling Strain on the Processing of Gamma Titanium Aluminides by Roll Bonding and Reaction Annealing: *Gajanan P. Chaudhari*[1]; Viola L. Acoff[1]; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., Box 870202, Tuscaloosa, AL 35487-0202 USA

Elemental aluminum and titanium foils were roll bonded at room temperature with different rolling strains, as the first step in the processing of gamma titanium aluminides. Subsequent two-stage reaction annealing treatment of the cold rolled laminates yielded gamma titanium aluminide sheet material. The effect of rolling strain on the interface development was examined. Increased rolling strain was found to influence the diffusion rates of the metallic elements. Higher stain levels accelerated the diffusion, which reduced the sheet processing time. The thickness, morphology and composition of the intermetallic compounds that formed at the interface of the elemental aluminum and titanium layers were studied using light microscopy and scanning electron microscopy equipped with energy dispersive spectrometry. The phases formed were verified using x-ray diffraction.

Phase Selection in Gamma TiAl Intermetallic Alloys: *Guosheng Shao*[1]; *Panos Tsakiropoulos*[1]; [1]University of Surrey, Sch. of Eng. (H6), Mechl., Matls. & Aeros. Eng., Guildford, Surrey GU2 7XH England

The metallurgy of gamma TiAl base alloys presents numerous challenges to the development and use of these materials in structural engineering applications. A key challenge is to understand phase competition and phase selection in all stages of processing, to allow the selection of suitable alloy compositions and processing routes. A distinctive insight of the physical metallurgy of these materials has been possible at Surrey by combining experimental studies of phase transformations and of alloying behaviour at the electronic level with first principal calculations, thermodynamic modelling and time dependent and steady state nucleation theory in materials processed by near equilibrium and non-equilibrium methods. The paper will discuss the dependence of phase competition between the alpha, beta and gamma phases on cooling rate and undercooling, the A2 Æ B2 phase transformation and omega phase formation in Ti-Al-TM systems. Alloy compositions and processing that lead to omega phase formation in the B2 matrix of TiAl-TM will be presented.

The Effect of Copper on the Cyclic Oxidation of Ti-48Al-2Cr-2Nb: *Jeffrey W. Fergus*[1]; Nicole L. Harris[1]; Christopher J. Long[1]; Victoria L. Salazar[1]; Tao Zhou[1]; William F. Gale[1]; [1]Auburn University, Matls. Rsrch. & Educ. Ctr., 201 Ross Hall, Auburn, AL 36849 USA

One of the challenges in the application of gamma titanium aluminide alloys is the development of suitable joining techniques for fabrication and repair. Transient Liquid Phase (TLP) bonding with a copper-containing composite interlayer has been used successfully to join titanium aluminide alloys. The TLP bonding process introduces a small amount of copper into the alloy. Although the oxidation resistance of titanium aluminide alloys can be strongly affected by alloying additions, the amount of copper present in the TLP bond has been shown to be neutral or beneficial to the isothermal oxidation resistance of the alloy. In this paper, results on the cyclic oxidation of Ti-48Al-2Cr-2Nb with 0-2% copper will be presented.

The Effect of Hafnium Additions on the Microstructure and Mechanical Properties of Gamma-Based Titanium Aluminide Alloys: *Johann Muellauer*[1]; Fritz Appel[1]; [1]GKSS-Research Centre, Inst. for Matls. Rsrch., Max-Plank-Strasse 1, Geesthacht D-21502 Germany

Solid solution hardening is a well established metallurgical technique for improving the strength properties of engineering materials. Solutes can affect phase equilibria, phase distribution, and can precipitate new phases, which may significantly alter strength and ductility. With this perspective in mind the effect of Hf additions on the microstructure and mechanical properties of two-phase titanium aluminide alloys were investigated. Hf is know to exclusively occupy the Ti sublattice of gamma-TiAl and exhibits a significant size misfit with the Ti atoms. Thus, one has a relatively clear situation, and can expect a relatively large hardening effect. The resulting strengthening effect will be investigated by mechanical testing involving thermodynamic glide parameters and microstructural characterisation.

Self-Oriented Lamellar Microstructure in Cast Platy TiAl: *Ji Zhang*[1]; Mingzhi Jiang[1]; Erik Strˆm[2]; Changhai Li[2]; Zengyong Zhong[1]; [1]Central Iron and Steel Research Institute, High-Temp. Matls. Rsrch. Div., No. 76 Xueyuan Nanlu, Beijing 100081 China; [2]Chalmers University of Technology, Dept. of Materials Science & Eng., Gothenburg SE-41296 Sweden

The good balance of strength and ductility can be achieved in the TiAl/Ti3Al lamellar structures when the angle between their boundaries and loading axis approaches to 0°. However, it is still difficult to utilize this mechanical anisotropy in the platy TiAl components through the longitudinal directional solidification since the preferred growth direction of primary alpha dendrites is normally parallel to its [0001] direction. Simple casting operations with proper radiating conditions can produce regular columnar grains in a platy material, resulting in the lamellar microstructure inside being aligned parallel to the surfaces. However, this casting configuration contains dense casting flaws and segregations in the place where the columnar crystals growing from the opposite surfaces meet for final solidification. This paper intends to eliminate the porosity and segregation in the cast Ti-46.5Al-2.5V-1.0Cr plates by employing centrifugal casting, HIP and heat treatment. The usability of the improved self-oriented lamellar microstructure is then evaluated by its ambient ductility, notch sensitivity and high temperature strength.

A Realistic Model for Studying the Effect of Welding Parameters on GTA Welding of Gamma Titanium Aluminide: *Kirtikumar B. Bisen*[1]; Viola L. Acoff[1]; Mario F. Arenas[1]; Nagy El-Kaddah[1]; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., Box 870202, Tuscaloosa, AL 35487-0202 USA

Limited research has been done to date to study the effect of various welding parameters on the weld pool dimensions of gas tungsten arc (GTA) welded gamma titanium aluminide. Previous research by the authors shows that welding current has a strong effect on the weld pool properties and dimensions. Other welding parameters such as arc length can also have a considerable effect on the shape of the weld pool and eventually on the microstructure and mechanical properties of the weld. A realistic model has been developed to predict the weld pool shape and temperature profiles as a function of welding current and arc length The results revealed that welding parameters govern the weld pool dimensions and temperature profiles significantly. The experimental results were in good agreement with the results predicted by the model.

Microstructural Stability of Fully Lamellar Gamma TiAl Alloys Under Creep: *Karthikeyan Subramanian*[1]; Young Won Kim[2]; Michael J. Mills[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA; [2]UES, Inc., Matls. & Proc. Div., Dayton, OH 45432 USA

Microstructural changes during creep were investigated in a fully lamellar titanium aluminide alloy containing carbon and silicon. A detailed investigation of the creep behavior of the aged (stabilized) and unaged (unstabilized) alloys was carried out and subsequent TEM studies were performed to characterize the microstructural changes during creep. Precipitation strengthening due to carbide and silicide formation leads to significantly reduced primary creep strain and secondary creep rates in the aged alloy. Intimately related to the process of precipitation is the dissolution of metastable alpha-2, which takes place during aging prior to creep. Dissolution of metastable alpha-2 is proposed as the reason for the larger strain rates in all stages of creep for the alloy in the unaged condition. Despite alpha-2 being the stronger phase, removal of alpha-2 prior to creep may actually be beneficial. These results suggest that microstructural stability is critica in order to achieve the highest possible creep strengths.

Development of a Cast Gamma TiAl Alloy with Near-Lamellea Microstrucure: *Li Shi Qiong*[1]; Jun Tao Li[1]; Yun Jun Cheng[1]; [1]Central Iron and Steel Research Institute, High Temp. Matls. Rsrch., No. 76 Xue Yuan Nan Lu, Beijing 100081 China

Recent study further indicated that the a cast gamma TiAl alloy, Ti-46.5Al-2.5V-1.0Cr(at%), developed in CISRI, not only has good combination of ductility and strength but also has higher creep and fatigue resistance at both room and elevated temperatures especially for the near-lamllea(NL) microstructure obtained after HIPing. Several structural components,such as swirlers, blades and rotors used for the aircraft and vehicle engines were fabricated by means of induction skull melting(ISM) and centrifugal casting with improved ceramic mold and optimized casting technique. A solidification simulation of the centrifugal investment casting for TiAL turbocharger rotors was also performed by a modified FT-Star software in order to predict the effects of the cast parameters on distribution of internal defects and solidification process.

Effect of Microstructure on Thermal Stability and Mechancial Properties of an Engineering Gamma-TiAl Based Alloy During Long-Term Exposure at 700∞C and 800∞C in Air: *Manuel Beschliesser*[1]; Gerhard Dehm[2]; Heinrich Kestler[3]; Helmut Clemens[4]; [1]Materials Center Leoben (MCL), Physl. Metall. & Matls. Testing, Franz-Josef-Str. 18, Leoben 8700 Austria; [2]Max-Planck-Institut fuer Metallforschung, Heisenbergstr. 3, Stuttgart 70569 Germany; [3]PLANSEE Aktiengesellschaft, Tech. Ctr., Reutte 6600 Austria; [4]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str., Geesthacht 21502 Germany

A gamma-TiAl based alloy with the nominal composition of Ti-46.5at.%Al-4at.%(Cr,Nb,Ta,B) has been exposed to air at 700∞C and 800∞C for up to 10,000 hours. The sheet material exhibits different microstructures prior to static annealing: a primary annealed microstructure which consists of fine grained gamma-TiAl phase with some amount of the alpha2-Ti3Al and beta/B2 phase at grain boundaries and triple junctions and two fully lamellar microstructures with parallel aligned gamma and alpha2 lamellae which exhibit different interface spacing. Evolution of type and composition of constitutional phases in the primary annealed microstructure has been recorded as a function of annealing time and temperature whereas the modification of microstructure due to thermal instability and the impact on mechanical properties has been investigated in the fully lamellar microstructures. The paper summarizes a systematic approach towards understanding of modifications in microstructure and phase stability as well as the mechanisms which cause these changes in gamma-TiAl based alloys during service operations.

The Effect of Processing and Porosity on the Elastic Moduli of Gamma-TiAl: *Moshe P. Dariel*[1]; Ori Yehezkel[1]; [1]Ben-Gurion University, Dept. Matls. Eng., Beer-Sheva 84105 Israel

The effect of porosity on the dynamic elastic moduli of gamma-TiAl samples that had been consolidated by cold isostatic pressing (CIP), pressureless sintering or hot isostatically pressing (HIP) was studied by ultrasonic sound velocity measurements. The results indicate that the sound wave velocities and the elastic moduli are affected by the processing route and depend not only on the density that was attained but also on the temperature at which the consolidation was performed. The effect of pressure on both the density and elastic moduli of samples that had undergone CIP or low temperature HIP (T~700∞C) is limited. Pressureless sintering in the 700 to 1100∞C temperature range barely affects the density but substantially the elastic moduli. In the 1150 to 1420∞C temperature range, both density and elastic moduli increase. High temperature HIP (T~1150∞C) yields nearly full density compacts with Youngís modulus of 197 GPa and Poissonís ratio of 0.22. The elastic moduli follow a linear dependence on porosity in the 0 to 0.4 range.

Segregation and Precipitation Patterns in Gamma Titanium Aluminides Alloyed with Carbon and Silicon: *Mallikarjun B. Karadge*[1]; Perena Gouma[1]; [1]State University of New York, Matls. Sci. & Eng., 314, Old Engineering Bldg., Stony Brook, NY 11794-2275 USA

K5 (Ti-46Al-2Cr-3Nb-0.2W) based gamma titanium aluminides alloyed with carbon and silicon have shown significantly enhanced high temperature creep resistance through the presence of fine H-type carbides and silicides along the a2/g and g/g interfaces. Although the advantages of such precipitates are known, the solute segregation and precipitation patterns are not well established and further studies are needed for improvement of this alloy. Differential scanning calorimetric studies have been carried out on pure K5, K5 alloyed with silicon (0.2 at. %) and K5 alloyed with carbon (0.1 at. %) and silicon (0.2 at. %) to clearly differentiate between the carbide and silicide precipitation processes involved. In situ TEM and HREM coupled with EELS were used to study solute segregation, nucleation and growth of the carbide and silicide precipitates at an atomistic level. Preliminary in situ heating studies indicated that carbides preferentially nucleate inside the a2 lath while silicides nucleate at the a2/g interface. The information obtained through this study can be applied for the development of similar such precipitation strengthened intermetallic alloys.

Effects of Added Interstitial Elements in Quaternary Ti-Al-Cr-Nb: *MÈlanie Lamirand*[1]; Jean-Louis Bonnentien[1]; Jean-Pierre Chevalier[1]; [1]CECM-CNRS, 15 Rue Georges Urbain, Vitry, Cedex 94407 France

Microstructural modifications in Ti-Al-Cr-Nb with controlled additions of interstitial elements, notably O, are presented. Trends observed are compared to those reported in binary and ternary alloys. Previous work shows that these interstitials affect the proportion of alpha2 and gamma phases in fully lamellar alloys, for a given cooling rate. The alpha2 volume fraction increases with increasing interstitial content, yielding a smaller interlamellar spacing, resulting in higher hardness. Quaternary Ti48AlCr2Nb2 alloy, using both ultra high purity and commercial purity Ti, have been prepared with clean processing and heat treatment. Interstitial elements have been added, and the microstructure analysed for given heat treatments and cooling rates. The aim is to ascertain whether the effects observed in binary and ternary alloys are transposable to quaternary alloys, i.e. increase in the alpha2 volume fraction, increased hardness and decreased ductility. The control of interstitials, especially O, is essential to ensure reproduceable microstructures and properties.

Evolution of the Microstructure of Two Nb-Containing Gamma-TiAl Alloys with Different Heat Treatments: *Maria Perez*[2]; IÒaki Madariaga[1]; IÒigo Hernandez[2]; Maria Luisa NÛ[3]; Jose San Juan[1]; Koldo Ostolaza[2]; [1]Industria de Turbo Propulsores ITP S.A., Materiales y Procesos, Parque Tecnologico n∫ 300, Zamudio, Vizcaya 48170 Spain; [2]Universidad del Pais Vasco UPV/EHU, Fisica de la Materia Condensada, Facultad de Ciencias Apdo. 644, Bilbao, Vizcaya 48080 Spain; [3]Universidad del Pais Vasco, Fisica Aplicada II, Facultad de Ciencias, Apdo. 644, Bilbao, Vizcaya 48080 Spain

Microstructures developed in two Nb-containing gamma-TiAl based alloys after heat treatments in the alpha and alpha+gamma regions of the Ti-Al phase diagram have been studied. The sheet material employed had an starting microstructure consisting of equiaxed gamma grains with an small volume fraction of alpha2 phase mainly located in gamma grain boundaries and triple points. Samples were heat treated at different temperatures ranging from 1200∫C up to 1400∫C. The hold times at these temperatures were varied from 1 minute up to 1 hour. Several cooling rates were also used to determine the influence of the cooling rate on the final microstructure. The different microstructures thus developed were analysed by means of optical and scanning electron microscopy. The identification of the various phases present in the microstructures was made with the help of X-ray difractometry, which also helped to understand the nature of the alpha to alpha+gamma transformation in these alloys.

High Temperature Internal Friction on Nb-Containing Gamma-TiAl Alloys: Maria Perez[1]; *Maria Luisa NÛ*[2]; IÒaki Madariaga[3]; Koldo Ostolaza[3]; Jose San Juan[1]; [1]Universidad del Pais Vasco (UPV/EHU), Fisica de la Materia Condensada, Facultad de Ciencias, Apdo. 644, Bilbao, Vizcaya 48080 Spain; [2]Universidad del Pais Vasco (UPV/EHU), Fisica Aplicada II, Facultad de Ciencias, Apdo. 644, Bilbao, Vizcaya 48080 Spain; [3]Industria de Turbo Propulsores ITP S.A., Materiales y Procesos, Parque Tecnologico n∫ 300, Zamudio, Vizcaya 48170 Spain

The aim of this work is to analyze the microscopic mechanisms controlling the plastic deformation at medium and high temperature on TiAl alloys by the technique of mechanical spectroscopy and to study the influence of the different microstructures of this material. Internal friction of the two phase alloy consisting in gamma-TiAl and alpha2-Ti3Al, with nominal composition: Ti-45Al-(5-10)Nb (in at%), has been studied on samples with different microstructures varying from duplex to fully lamellar ones. The internal friction measurements has been made in a subresonant torsion pendulus by two different methods; changing the temperature between 300K and 1200K at a constant frequency, and varying the frequency between 0.001Hz and 10Hz at a fixed temperature. The resulting mechanical spectra, have been analysed in order to obtain the activation parameters that identify the mechanisms responsible of the observed relaxations. These parameters have also been compared with those obtained from creep tests.

Development of Cost-Effective Technique for Sheet Rolling of Gamma-TiAl Based Alloys: *M. R. Shagiev*[1]; G. A. Salishchev[1]; [1]Institute for Metals Superplasticity Problems, 39 Khalturin Str., Ufa 450001 Russia

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The sheets of γ-TiAl based alloys produced by existed rolling techniques are very expensive because of high rolling temperatures. Besides these sheets exhibit low superplastic properties at temperatures below 1000°C that makes difficult their superplastic forming/diffusion bonding. In the current study the considerable decrease in the rolling temperature and improvement of superplastic properties of γ-TiAl based sheets were achieved through the grain refinement. So, formation in the rolling preforms of homogeneous micro- and submicrocrystalline structure made possible to perform the sheet rolling of γ alloys within the $(\gamma+\alpha_2)$-phase field, i.e. at 800-1100°C depending on alloy composition and grain size. Along with retaining the fine-grained microstructure in γ sheets, which provides superior superplastic elongations, the decrease in rolling temperature leads to substantial decrease in their cost because of energy savings, applicability of cheaper can materials than those being used now, less oxidation during preheating and better surface finish on γ sheets.

Non-Destructive Evaluation (NDE) of the Elastic Moduli of Near γ-TiAl Porous Compacts: *Ori Yeheskel*[1]; Menashe Shokhat[1]; [1]NRCN, PO Box 9001, Beer Sheva 84190 Israel

Sintering or HIP were employed to consolidate various powders sifted from a commercial pre-alloyed g-TiAl. The various particle sizes contain unequal amounts of a2-Ti3Al, and are regarded as near g-TiAl alloy. The elastic moduli were determined by dynamic technique. It is shown that both density and particle size affect the elastic moduli. The evaluation of the Youngís or bulk moduli from the porosity alone, yield large uncertainty (~100%) at high porosity (P>0.25). A linear correlation between the shear and Youngís moduli is demonstrated. This correlation enables the evaluation of the various elastic moduli from a single known elastic modulus or a single measured SWV. Plotting the elastic moduli against the longitudinal sound velocity yields a single and monotonous line along which all the data points lie. The uncertainty of such evaluated elastic moduli is small (< 5%) which open the way to use that technique for quantitative NDE of the elastic moduli of porous intermetallics.

Thermomechanical Processing and Tensile Mechanical Properties of Beta-Solidifying Gamma-TiAl+Alpha2-Ti3Al Alloys: *Valery Imayev*[1]; *Renat Imayev*[1]; *Andrey Kuznetsov*[1]; [1]Institute for Metals Superplasticity Problems, Khalturin Str. 39, Ufa 450001 Russia

Thermomechanical processing consisting of quasi-isothermal forging in the (alpha+gamma) phase field followed by isothermal forging in the (alpha2+gamma) phase field has been developed for difficult-to-work ingot-metallurgy gamma+alpha2 alloys. This processing was applied to beta-solidifying alloys Ti-45.2Al-3.5(Nb,Cr,B) and Ti-44.2Al-3.5(Nb,Cr,B) that resulted in a fine-grained microstructure with lamellae remnants. The use of beta-solidifying alloys allowed attaining high ductile/superplastic properties in the temperature range of 800-1100∞C and strain rate range of 3x10-4-10-1 s-1. Tensile tests were also performed for thermomechanically treated lamellar conditions produced in these alloys. The results obtained showed that due to good microstructural controllability during thermomechanical and heat treatments, boron containing beta-solidification alloys have a great potential for developing secondary hot working processes at relatively low temperatures and attaining the good balance of mechanical properties in lamellar conditions.

Modeling and Measurement of the Elastic-Plastic Behavior of Gamma Titanium Aluminide Colonies: *Reji John*[1]; W. John Porter[2]; Steven E. Olson[2]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct. (AFRL/MLLMN), WPAFB, OH 45433 USA; [2]University of Dayton Research Institute, 300 College Park, Dayton, OH 45469 USA

Colony-level finite element models, which are being developed to predict damage initiation and progression in Gamma TiAl, require knowledge of the orthotropic nature of the deformation and fracture behavior of individual colonies. Hence, an integrated experimental and analytical study was conducted to understand the effect of lamellar colony orientation on the elastic-plastic response of the colonies. The material used was Ti-46.5Al-3Nb-2Cr-0.2W (at%), which was heat treated to achieve a microstructure containing large (>7 mm), fully-lamellar colonies. Tensile specimens incorporating a single colony in the gage section were used to determine the deformation response. Three-dimensional finite element models, incorporating the actual orientations of all colonies, were developed for each specimen to deduce the colony properties from the measured stress-strain data. The predicted colony properties were compared to data available for PST and polycrystal gamma TiAl. Use of the elastic-plastic models to understand the fatigue initiation process will also be discussed.

Creep in Gamma-TiAl Sheet Material with a High Niobium Content: *Slawomir Bystrzanowski*[1]; A. Bartels[1]; R. Gerling[2]; F. P. Schimansky[2]; H. Kestler[3]; M. Weller[4]; H. Clemens[2]; [1]Technical University of Hamburg-Harburg, Dept. Matls. Sci. & Tech., Eissendorfer Str. 42, Hamburg D-21071 Germany; [2]GKSS-Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str., Geesthacht D-21502 Germany; [3]Plansee AG, Tech. Ctr., Reutte A-6600 Austria; [4]Max-Planck-Institut f¸r Metallforschung, Heisenbergstr. 3, Stuttgart D-70569 Germany

Creep properties were determined in Ti-46Al-9Nb (at%) sheet material which was processed by a powder metallurgical route. Fine-grained near gamma and fully lamellar microstructures were adjusted. The tensile 100 hours creep tests were carried out for under constant load at 700∞C. With changes of load between 150-250 MPa the stress exponent was evaluated and with temperature changes in the range of 973-1048K the activation energy was determined. Additionally, internal friction experiments were conducted giving a second independent access to the high-temperature deformation mechanisms. The results of the stress exponents and activation energies are in accordance with dislocation movement and thermally activated dislocation climb as predominant creep mechanism. A comparison with the creep behavior of previously investigated alloys, e.g. Ti-47Al-4(Cr,Mn,Nb,Si,B) (at%) and Ti-46.5Al-4(Cr,Nb,Ta,B) (at%), indicates a significantly better creep resistance of the high Nb containing alloy. The higher activation energy causes a slowing down of the climb.

Atomic Scale Chemistry of Carbide/Matrix Interfaces in Gamma TiAl Alloys: Stephan S.A. Gerstl[1]; David N. Seidman[1]; [1]Northwestern University, Matls. Sci., 2225 N. Campus Dr., Evanston, IL 60208 USA

Carbide precipitation is known to strengthen TiAl alloys at high temperatures. The needle-like structures of Ti2AlC have been reported to act as dislocation barriers, causing Orowan dislocation looping at low temperatures and dislocation climb at higher temperatures [Tian and Nemoto, 1995]. Transmission electron (TEM) and 3-dimensional atom-probe (3DAP) microscopies are utilized for characterizing the nanometer scale precipitates, their interfaces, and the effect of various microalloying elements (Zn, Mn, and Cr), which are added to the melt prior to carbide precipitation. The precipitate structure and number density, as a function of the microalloying additions, are investigated by TEM, whereas the chemical composition at the carbide/metal interface is studied by 3DAP. Partitioning and segregation behaviors of the alloying elements are discussed in terms of interfacial energies between the carbide and intermetallic phases. This research is supported by the DOE.

Synthesis of Nano-Crystalline Gamma-TiAl Materials: *Stephen John Hales*[1]; Terryl Anne Wallace[1]; Peter Vasquez[2]; [1]NASA Langley Research Center, Struct. & Matls., MS 188A, 2 W. Reid St., Hampton, VA 23681 USA; [2]NASA Langley Research Center, Sys. Eng., MS 392, 1A S. Marvin St., Hampton, VA 23681 USA

One of the principal problems with nano-crystalline materials is producing them in quantities and sizes large enough for valid mechanical property evaluation. The purpose of this study is to explore an innovative method for producing nano-crystalline gamma-TiAl bulk materials using high energy ball milling and brief secondary processes. Clean, nano-crystalline feedstock is being produced using a Fritsch P4 planetary mill recently installed at NASA-LaRC. The mill employs tungsten carbide tooling (vials and balls) and no process control agents to minimize contamination. Two approaches are being investigated, namely mechanical alloying of elemental powders and attrition milling of pre-alloyed powders. The objective is to subsequently use RF plasma spray deposition and short cycle-time vacuum hot pressing in order to effect consolidation while retaining nano-crystalline structure in bulk material. Results and discussion of the work performed to-date will be presented.

Effect of Ta and Zr Additions on the Oxidation Characteristics of Ti-44Al-xNb Alloys: John Woo[1]; *Rabindra N. Mahapatra*[2]; Shailendra K. Varma[1]; [1]The University of Texas at El Paso, Dept. of Metallurgl. & Matls. Eng., El Paso, TX 79968-0520 USA; [2]Naval Air Warfare Center, Aircraft Div., Patuxent River, MD 20650 USA

Quaternary alloys of Ti-44Al-x(9 or 11)Nb-2(Ta,Zr) have been subjected to cyclic oxidation in air for a period of one week at 900 and 1000∞C. The oxidation behavior has been characterized by thermogravemetric analysis, optical microscopy, SEM, and TEM. Zr alloys show a few orders of magnitude higher oxidation rate compared to alloys containing Ta. It is attributed to the formation of a spongy like structure which has been characterized as the alpha 2 phase formed as a result of disordered to ordered phase transformation during the oxidation process. The extent of this transformation is much higher in Zr alloys than in Ta alloys, and is observed to be accelerated at 1000∞C. The oxidation morphology and chemical compositions of oxides formed after the exposures were determined by utilizing SEM/EDAX. A layer rich of Al2O3 oxide formed next to the matrix followed by a layer mixed with Al2O3 and TiO2 while the topmost layer, directly facing air, was rich in TiO2. Change in the value of rate constant from initial oxidation stage to that observed at longer periods of time will be discussed.

Temperature Dependence of Compressive Behavior in Aluminum-Titanium-Vanadium Ternary Alloys Containing Gamma and Beta Grains: *Tohru Takahashi*[1]; Koshiro Otsuka[2]; Takahiro Murakoshi[2]; Tadashi Hasegawa[1]; [1]Tokyo University of Agriculture and Technology, Dept. of Mechl. Sys. Eng., Naka-cho 2-24-16, Koganei, Tokyo 184-8588 Japan; [2]Tokyo University of Agriculture and Technology Japan

Compressive behavior was studied on aluminum-titanium-vanadium ternary alloys. Yield strength and its temperature dependence was compared among materials whose microstructures comprised gamma grains, beta grains, or both. The Al40Ti30V30 (numbers in atomic %) alloy showed microduplex structure of about 3 microns grain diameters. This material showed limited deformability at temperatures below 900K. The 0.2% proof stress was about 800MPa at 900K, but it sharply decreased with increasing temperature. The Al35Ti25V40 alloy showed coarse grained microstructure of beta phase. This material was almost brittle up to 800K, but became deformable at and above 900K. At higher temperatures a considerable strain softening occurred, and it was followed by quasi-steady state flow. The beta phase was stronger than the gamma phase below 900K but it became softer than the gamma phase above 1000K. This can find possible application in high temperature forming of gamma+beta dual phase microstructure with enhanced strength at medium temperature.

Strain Ageing in Alpha2 and Gamma-Based Titanium Aluminides: *Ulrich Froebel*[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Strasse 1, Geesthacht D-21502 Germany

Deformation of alpha2 and gamma-based TiAl between 450-850K is characterized by negative strain-rate sensitivity and strain ageing effects. These phenomena are usually associated with dislocation locking according to the Portevin-LeChatelier effect. The pinning processes occurring at the dislocations and associated with these phenomena were studied by static strain ageing experiments utilizing alloys with many different aluminium concentrations. Accordingly, the strain ageing phenomena for stoichiometric compositions of the alpha2 and gamma phases are much less pronounced when compared with Ti-rich gamma alloys indicating that off-stoichiometric deviations might be most important for strain ageing. In off-stoichiometric alpha2 and gamma the dominant point defects are antisite atoms on the relevant sub-lattice. Association of the antisite atoms with vacancies leads to the formation of anti-structural bridges. The activation energies of strain ageing can be attributed to the diffusion of antisite atoms along an ti-structural bridges indicating that antisite atoms are involved in the pinning processes.

Processing of Gamma-Based TiAl Alloys by Torsional Deformation: *Uwe Lorenz*[1]; Stefan Eggert[1]; Michael Oehring[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str. 1, Geesthacht D-21502 Germany

Thermomechanical processing offers probably the most effective way for controlling the microstructure of gamma-based titanium aluminide alloys. Until now the traditional metal-forming operations, such as extrusion, forging and rolling, have been used. Although the feasibility of these methods has been demonstrated on an industrial scale, hot-worked products often suffer from insufficient consolidation, that is manifested by incomplete recrystallization and significant chemical inhomogeneity. In an attempt to overcome these problems, hot-working of TiAl alloys was performed utilising torsional deformation superimposed with compression. The advantage of the method is that much higher strain and mechanical work can be imparted, which trigger more intensive dynamic recrystallisation. The hot-working procedure leads to a significant structural refinement, which will be assessed by standard metallography, chemical microanalysis and transmission electron microscopy. The potential of the method for industrial application will be discussed.

Analysis of the Solidification Microstructure of Multi-Component Gamma-TiAl Alloys: *Viola Kuestner*[1]; Michael Oehring[1]; Anita Chatterjee[2]; Helmut Clemens[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Strasse 1, Geesthacht D-21502 Germany; [2]GfE Metalle und Materialien GmbH, Nuernberg D-90431 Germany

Gamma titanium aluminide alloys used for high temperature applications typically have an aluminium content in the range of 45-48 at.%. They solidify peritectically and show a coarse, dendritic microstructure, which leads to unacceptable mechanical properties in the as-cast condition. In view of the further development of cast alloys, the dependence of the solidifying microstructure on the aluminium content and other alloying elements was investigated. The activities were focused on the solidification path of technically interesting alloys, which depends largely on their composition. Furthermore, the role of grain refining agents and their interactions with other alloy elements were studied. The investigation has centered on scanning electron microscopy of arc melted buttons including quantitative EDS linescans.

Cost-Effective TiAl-Based Materials: Vladimir S. Moxson[1]; *F. H. (Sam) Froes*[2]; [1]ADMA Products, Inc., 8180 Boyle Pkwy., Twinsburg, OH 44087 USA; [2]University of Idaho, IMAP, Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Because of their inherent lack of ductility, TiAl-based materials are difficult to fabricate. In this paper, a powder metallurgy approach to production of both flat products (sheet/foil) and ìchunkyî components will be presented. Flat products as wide as 9" can be produced by this technique. Very complex ìchunkyî parts such as automobile connecting rods can be fabricated by this approach. Microstructures and mechanical properties will be presented.

Texture Formation During Thermomechanical Processing of Titanium Aluminides: *Werner Skrotzki*[1]; Roland Tamm[1]; Heinz-G¸nter Brokmeier[2]; Michael Oehring[2]; Fritz Appel[2]; Helmut Clemens[2]; [1]TU Dresden, IKFP, Dresden 01328 Germany; [2]GKSS Research Centre, Geesthacht 21502 Germany

The texture development has been studied in cast titanium aluminides (alloy A: Ti-47Al-4(Cr, Nb, Mn, Si, B) and alloy B: Ti-45Al-10Nb) deformed by forging and extrusion at different temperatures corresponding to different fractions of the alpha- (A3) or alpha2- (D019) and gamma- (L10) phase. The textures of the alpha-phase have been measured by neutron diffraction. Forging alloy B at 1100∞C in the alpha2 + gamma phase field leads to a <032] + <110] double fibre texture. Deforming the alloys at temperatures between 1250∞C and 1380∞C, with increasing temperature the gamma-fraction decreases and becomes zero above the alpha-transus temperature. Correspondingly, the fibre textures obtained by extrusion through a round die change from [001] + <111> via <111> to <011] + <110] + <112]. Extrusion through a rectangular die of alloy B at 1295∞C yields a dominant {111}<112> component. The type and strength of texture will be discussed with regard to starting texture, phase mixture, deformation mode, slip and twinning activity, dynamic recrystallization and phase transformations.

Crystallographic Considerations for Lamellar Orientation Control of Ti-48Al PST Crystal: *Yukinori Yamamoto*[1]; Masato Nagaki[1]; Masao Takeyama[1]; Takashi Matsuo[1]; [1]Tokyo Institute of Technology, Dept. of Metall. & Ceram. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Unidirectionally solidified Ti-48Al PST crystal grown by using a seed of γ single-phase single crystal (Ti-57Al) follows an orientation of the seed with its lamellar orientation parallel to one of the four $\{111\}_\gamma$ planes of the seed. However, in order to control the lamellar orientation of the PST crystal, we have to figure out the mechanism of which plane is to be chosen. There exist several lamellar grains following the seed orientation at the contact interface between the seed and grown crystal, and one of them is selectively grown. The crystallographic analysis of these grains reveals that the grain to be selected is somehow related to the preferred growth orientations of the primary α phase, $[0001]_\alpha$ and $\langle 10\text{-}10 \rangle_\alpha$, with respect to the growth direction. The details of the analysis as well as the experimental evidence to control the lamellar orientation will be presented.

The Effect of Water Vapor on the Oxidation Behavior of Gamma-TiAl-Ag Coatings at 800C in Air: *Zhenyu Liu*[1]; Toshio Narita[1]; [1]Hokkaido University, Grad. Sch. of Eng., Kita-ku, Kita 13 Nishi 8, Sapporo, Hokkaido 060-8628 Japan

Oxidation exposures were carried out to assess the oxidation resistance of Ti-48Al-xAg (x=1, 2, 3 at.%) coatings at 800∞C in air with different H2O contents for 100 hours. The coatings containing more than 1.0 at.% Ag have been proved to be resistant against oxidation attacks in wet air. It has been found that both Ag content in the coating and H2O content in the atmosphere have substantial influence on the oxidation behavior. For coatings with 1.0at.% Ag, TiO2 and the mixture of TiO2+Al2O3 were formed in 15.5 and 30.8 vol.%H2O, while TiO2, the mixture of TiO2+Al2O3 and continuous Al2O3 layers were formed in 9.0 vol.%H2O. For the coatings containing more than 1.0at.% Ag, higher H2O contents (15.5 and 30.8 vol.%) resulted in the formation of duplex oxide scale with a mixture of TiO2+Al2O3 on top of continuous Al2O3, while continuous single Al2O3 layer was formed in lower H2O content (9.0 vol.%). The effect of H2O vapor on the oxidation behavior was explained by the interaction of H2O and TiO2, or the rapid and anisotropic diffusion of hydrogen in TiO2 during

POSTERS

oxidation. After long-term oxidation exposures (1,000 hours) in 30.8% H2O, the coatings also showed high oxidation resistance and good compatibility with the TiAl-10Cr substrates, with no hard brittle compounds having been observed at the coating/substrate interface.

Processing and Properties of TiAl Alloy Manufactured with MA-PDS Process: *ZhengMing Sun*[1]; Hitoshi Hashimoto[1]; Toshihiko Abe[1]; [1]National Institute of Advanced Industrial Science and Technology, AIST-Tohoku, 4-2-1 Nigatake, Miyagino, Sendai 983-8551 Japan

TiAl powder was prepared with mechanical alloying (MA) process by using a nitrogen-shock technique, which improved the powder recovery rate to almost 100%. The MA powder was sintered and concurrently consolidated to full density by employing a pulse discharge sintering (PDS) process, which requires short-time and low-temperature sintering. The TiAl alloy obtained form this process showed a fine microstructure with equiaxed submicron grain size. The TiAl alloy demonstrated a high strength compared with those fabricated with conventional processes and the fracture strength increases with an increase in testing temperature below 873 K. The effect of alloying element such as Cr, Mn, Mg, Zr and sintering parameters as well as heat treatment on the microstructure and properties of the alloy were also investigated. Application of the TiAl alloys to automobile engine parts, sputtering target, diamond polishing will be introduced.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C. T. Liu: Intermetallics VIII–Poster Session

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

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Monday 7:00-9:00 PM Room: Upper Level City-Side Corridor Lobby
March 3, 2003 Location: San Diego Convention Center

Session Chairs: Seetharama C. Deevi, Philip Morris USA, Rsrch. Ctr., Richmond, VA 23234 USA; Yip-Wah Chung, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208-3108 USA; Peter K. Liaw, University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA

Creep Behavior of Nb_3Al-Based, Mechanically Alloyed Materials: *Anna Doll·r*[1]; Stanislaw Dymek[2]; [1]Miami University, Mfg. & Mechl. Eng. Dept., Kreger Hall, Oxford, OH 45056 USA; [2]University of Mining and Metallurgy, Metallurgl. & Matls. Eng. Dept., Krakow Poland

The creep resistance of Nb_3Al is comparable to some nickel based superalloys, making this high melting temperature ordered compound a candidate for next generation gas turbine engines. However, Nb_3Al is brittle at temperatures below 1475K. Mechanical alloying proved to be a viable route for processing Nb_3Al - based alloys with ductility improved as a result of grain refinement and introduction of the ductile Nb solid solution (Nb_{ss}). In this study, creep behavior of several Nb_3Al /Nb_{ss} materials, containing also oxide dispersoids and a nonequilibrium ordered phase, Nb_2Al has been investigated. It has been established that the Nb_2Al phase leads to the deterioration of creep properties. To improve creep resistance ternary additions of V have been used. This attempt leads to the processing of an alloy, Nb15at.%Al20at.%V, which, when consolidated in 1773K, does not contain the highly undesirable Nb_2Al phase and exhibits creep rates significantly lower than other materials investigated in the present research.

Creep and Microstructure of Near-Gamma TiAl Alloys: *Antonin Dlouhy*[1]; Kveta Kucharova[1]; [1]Institute of Physics of Materials/AS CR, Mechl. Properties, Zizkova 22, Brno 616 62 Czech Republic

Creep strength and microstructure of Ti-48Al-2Cr-2Ni and Ti-46Al-2W-0.5Si base alloys are compared. The influence of the prior to creep heat treatment on the initial microstructure is considered. In the case of the Ti-48Al-2Cr-2Ni alloy, nearly lamellar and equiaxed modifications were tested while the Ti-46Al-2W-0.5Si alloy was either nearly lamellar or duplex before creep. All these microstructural modifications were crept in tension at 750∞C and in the range of the applied stress. The obtained experimental results suggest that, in spite of variations based on the different alloy states, the Ti-46Al-2W-0.5Si alloy performs generally better than the Ti-48Al-2Cr-2Ni alloy. Transmission electron microscopy analysis provided evidence that W- and Si-rich phases which precipitate on both, the lamellar boundaries and inside the gamma grains, contribute to the higher creep strength of the Ti-46Al-2W-0.5Si alloy. The stability of these strengthening phases with increasing time of the creep exposure is subjected to an investigation.

Strength Anomaly of B2-FeAl Alloys: Olivier Calonne[1]; *Anna Fraczkiewicz*[1]; FranÁois Louchet[2]; [1]Ecole des Mines, SMS, 158, Cours Fauriel, Saint-Etienne 42023 France

The yield stress of B2-FeAl alloys increases with temperature in the 300-7000C range. This anomaly is characterised by very small values of the strain rate sensitivity (SRS), and by decompositions of <111> superdislocations into <001> and <110> ordinary ones close to the stress peak. The anomaly is obscured by vacancy hardening in as-quenched materials, which suggests a possible role of vacancies in the hardening mechanism. In situ TEM straining reveals APB tube dragging by <111> edge superdislocations in the anomaly domain. APB tube dragging is thought to stem from climb of leading superpartials as they meet vacancies on their slip planes, trailing superpartials being unable to restore the chemical order. The resulting drag force is large enough to be responsible for a thermally activated gradual exhaustion of superdislocations, resulting in a strength anomaly and a zero SRS. Superdislocation decomposition provides ordinary dislocations that allow high temperature deformation at lower stresses.

Metal Dusting of Fe3Al-Based Alloys: *AndrÈ Schneider*[1]; Jianqiang Zhang[1]; Gerhard Inden[1]; [1]Max-Planck-Institut f¸r Eisenforschung GmbH, Matls. Tech., Max-Planck-Str. 1, D¸sseldorf 40237 Germany

Iron aluminides are known for their resistance to high temperature oxidation and sulphidation. Only little information is available about carburisation and metal dusting of Fe-Al alloys. Metal dusting experiments with Fe-26Al , Fe-26Al-2M (M = Ti, V, Nb, Ta) and Fe-26Al-2M-1C alloys with MC carbides were conducted in CO-H_2-H_2O gas mixtures at 650∞C. The kinetics of the carbon transfer was measured using thermogravimetric analysis (TGA). The phases on the carburised samples were identified by X-ray diffraction (XRD) and observed in metallographic cross sections. Surface analysis were performed by means of scanning electron microscopy (SEM).

Mechanisms of Structure Formation of the Al9FeMnSi2 Intermetallic Compound, Processed by Pressure Assisted Reactive Sintering: Arturo Toscano Giles[1]; *Alfredo Flores Valdes*[1]; Armando Salinas Rodriguez[1]; [1]CINVESTAV, Unidad Saltillo, PO Box 663, Saltillo, Coahuila 25000 Mexico

The processing of quaternary intermetallics of the Al9FeMnSi2 type has been achieved successfully starting from the pure elements, in the range of temperatures from 600 to 800°C, using Pressure Assisted Reactive Sintering. It has been possible the obtention of alloys free of porosity, segregation, and structural inhomogenities resultant from the reactions taking place. Different experimental techniques have been used to understand the mechanisms of formation of the quaternary intermetallic structure, such as high temperature in-situ x-ray crystallography, scanning electron microscopy, differential thermal analysis, and optical microscopy, which are discussed in this work. The effects of temperature, pressure, and chemical composition of reactants are provided for the processing of such intermetallic compound, which has been proposed for the surface protection of low alloy steels, stainless steels, and Co-Cr-Mo alloys.

Intermetallic Phases Formed by Ruthenium-Nickel Alloy Interdiffusion: *B. Tryon*[1]; T. Pollock[1]; [1]University of Michigan, Dept. of Matls. Sci. & Eng., 3062 H. H. Dow Bldg., 2300 Hayward St., Ann Arbor, MI 48109 USA

Ruthenium additions to nickel-base superalloys have recently been of interest due to their influence on the high temperature properties of these materials. Diffusion couples were formed between ruthenium or ruthenium aluminide and two different nickel-base alloys. The couples were heat treated at various temperatures to evaluate diffusion paths and phase equilibria between the constituent materials. The interfacial layers have been evaluated by X-ray diffraction and electron microprobe analysis. A tantalum-rich, B2 intermetallic phase formed at the interface of each couple during heat treatment. Two buttons of similar composition to the interface layer were arc-melted and analyzed in an attempt to isolate the phase. The results of these analyses and their implications for nickel-based systems will be discussed.

High Temperature Oxidation of RuAl and RuAl Alloys: *D. Stobbe*[1]; T. K. Nandy[1]; Q. Feng[1]; Tresa Pollock[1]; [1]University of Michigan, Sch. of Eng., Dept. of Matl. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

Oxidation is one of the primary degradation modes in intermetallics intended for use in high temperature applications. The present investigation explores high temperature oxidation behavior of RuAl and RuAl alloys with Pt, Mo, and Nb as alloying additions. Cyclic oxidation experiments were performed in laboratory air at 1000, 1100, 1200, and 1300°C. The samples were oxidized for about 100-200 cycles (1 hr per cycle), recording mass loss periodically. Additional characterization used to further study the oxidation process included X-ray diffraction (XRD) and scanning electron microscopy (SEM). Possible rate limiting mechanics for oxidations and effects of different alloying additions will be discussed.

Formation of an Ultra-Fine Lamellar Microstructure in TiAl Based Alloys: *F. Meisenkothen*[1]; R. Banerjee[1]; G. B. Viswanathan[1]; H. L. Fraser[1]; [1]Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43016 USA

Conventionally processed, fully lamellar, TiAl based alloys ordinarily possess laths having a thickness on the order of hundreds of nanometers. Recent work has shown that polycrystalline samples having compositions between Ti-42Al and Ti-46Al can be solution heat treated in the α phase field, and then rapidly quenched, to produce a microstructure that consists entirely of supersaturated α_2 phase. Upon aging the supersaturated solid solution, in the α_2+γ phase field, a fully lamellar structure results. However, in contrast to the conventionally processed fully lamellar TiAl based alloys, the laths resulting from the aging treatment have a thickness on the order of tens of nanometers. Additionally, this nanoscale microstructure consists of rigidly alternating laths of α_2 and γ. A tranformation mechanism from supersaturated α_2 to ultra-fine lamellar α_2+γ via nucleation, glide, and cross-slip of dislocation loops will be discussed.

Moisture-Induced Embrittlement of Iron and Titanium Aluminides: *Gilbert HÈnaff*[1]; Anne Tonneau-Morel[1]; Anne-Marie Brass[2]; [1]ENSMA, LMPM, 1 Ave. C Ader, BP 40109, Futuroscope, Chasseneuil F-86961 France; [2]UniversitÈ Paris Sud, LPCES, CNRS UMR 8648, Bat 413, Orsay F-91405 France

This paper reports on investigations conducted during the last years on the understanding of the mechanisms responsible for the embrittlement of iron and titanium aluminides in a moist environment. Mechanical testings carried out under various environmental conditions provide evidences for a loss of performance associated with a hydrogen-assisted fracture mode. SIMS analysis of deuterium diffusion profile is used to characterize the water dissociation process, producing atomic hydrogen on the material surface, with respect to microstructural parameters. Hydrogen absorption is shown to depend on several parameters such as chemical composition, microstructure, temperature, deformation conditions, etc. Furthermore hydrogen absorption can be hindered by surface oxide scale but favoured by mechanically-induced damage. The relevance of these mechanisms to fatigue crack propagation is also examined. In particular the role of competitive adsorption of oxygen and the influence of temperature for these two types of compounds are discussed.

Phase Selection in TM-Al Intermetallics: *Guosheng Shao*[1]; *Panos Tsakiropoulos*[1]; [1]University of Surrey, Sch. of Eng. (H6), Mechl., Matls. & Aeros. Eng., Guildford, Surrey GU2 7XH England

Intermetallics present numerous challenges to the metallurgists who develop, process and use advanced metallic materials. A key challenge is to understand phase competition and phase selection in all stages of processing, to allow the selection of suitable alloy compositions and processing routes. At Surrey, research has concentrated both on aluminides and exotic intermetallics. The paper will concentrate on intermetallic alloys of TM-Al (TM = Cr, Mn, Fe, Nb) systems. A distinctive insight of the metallurgy of these materials has been possible by experimental studies of phase transformations in materials processed by near equilibrium and non-equilibrium methods that have been coupled with first principal calculations, thermodynamic modelling, time dependent and steady state nucleation theory and the experimental study of alloying behaviour at the electronic level. The A2 Æ B2 phase transformation and omega phase formation in TM-Al systems will be discussed.

Designing a New Family of High Temperature Wear Resistant Alloys Based on Ni3Al IC Experimental Results and Thermodynamic Modelling: *HÈlio Goldenstein*[1]; Yuri Nunes Silva[1]; Humberto N. Yoshimura[2]; [1]Escola PolitÈcnica da USP, Metall. & Matls. Eng., Av. Prof. Mello Moraes 2463, S„o Paulo, SP 05508-900 Brazil; [2]Instituto de Pesquisas TecnolÛgicas do ESP, Div. QuÌmica, Lab. de Cer‚mica

A new family of IC based alloys is proposed, as a substitute for high temperature abrasion and erosion resistance cobalt alloys. The aim are microstructures were hard carbides are dispersed in a continuous „í Ni-Al-Cr L12 ordered intermetallic matrix. Alloy design aimed at matrix compositions similar to IC-221, with chromium and carbon added. Modelling of the Ni-Al-Cr-C phase diagram was undertaken using ThermoCalc calculations. A customized thermodynamic database (Nicralc) assembled from different databases was used to study the liquidus, solidification sequence and equilibrium phases of compositions near the „í+ carbide field. The solidification sequence was studied experimentally using DTA, OM and SEM, X-ray diffraction of sections and of extracted carbides. Different carbide structures were obtained, depending on the Cr/C ratio. Hypoeutectic alloys presented „í/„ dendrites and interdendritic eutectic „í + carbide. Hypereutectic alloys presented a bi-modal carbide distribution, with large proeutectic carbides within a fine „í + carbide matrix.

Processing of Fine-Grained Mechanically Alloyed FeAl: *Helena Skoglund*[1]; Maria Knutson Wedel[1]; Birger Karlsson[1]; [1]Chalmers University of Technology, Matls. Sci. & Eng., Hˆrsalsv‰gen 7, Gˆteborg SE-412 96 Sweden

Mechanically alloying followed by hot consolidation is one route to produce ductile FeAl. Earlier, hot extrusion has been the only consolidation method used to attain a fine-grained microstructure. This work shows that HIP also results in a fine microstructure, grain size 2-5 µm, which in addition is isotropic in contrast to the textured extruded materials. In this work, a powder mix of 60 at% Fe and 40 at% Al has been milled in argon. Both powder and HIPed material have been characterised by XRD, SEM, TEM, ESCA, Auger and chemical analysis of oxygen content. The powder particles, sized 1-4 µm, are nanocrystalline with a grain size of 5-10 nm. The solid FeAl shows a distribution of Al_2O_3-precipitates and carbides. The tensile strength was 600 MPa (with predominantly intergranular fracture) after a heat treatment to reduce the non-equilibrium vacancy concentration, that also reduced hardness from 3,4 GPa (HV5) to 3.1 GPa.

Effect of Severe Plastic Deformation on the Mechanical Behavior of Ti-6Al-4V and Ti-6Al-4V Metal Matrix Composites: *G. Guven Yapici*[1]; I. Karaman[1]; [1]Texas A&M University, Dept. of Mechl. Eng., College Station, TX 77843 USA

The present work focuses on the severe plastic deformation of Ti-6Al-4V and Ti-6Al-4V reinforced with 10% TiC metal matrix composites using equal channel angular extrusion (ECAE). The bulk materials are extruded through two channels of equal cross section intersecting at an angle of 90°. Microstructure and mechanical properties of extruded billets are reported through electron microscopy observations and tension, compression and hardness experiments. Results are compared for different extrusion conditions including variations in temperature and processing route. Higher hardness values are obtained after ECAE compared to as-received values. These improvements are correlated with the grain refinement, phase refinement and texture produced during ECAE. The ultimate goal is to develop thermomechanical-processing maps for the selection of processing schedules to obtain desired end microstructures and mechanical properties in Ti-Al-V based materials.

The Investigation of Laser In-Situ Surface Alloying Al/Cu onto AA6061 Aluminum: *Jianjun Ding*[1]; *C. L. Choy*[2]; *Tengfei Wu*[1]; Mon-Yu Wei[3]; [1]Shanghai Jiao Tong University, Col. of Matls. Sci. & Eng., 1954 Huashan Rd., Shanghai 200030 China; [2]The Hong Kong Polytechnic University, Dept. of Appl. Physics, Hung Hom Kowloon, Hong Kong China; [3]Ta Hwa Institute of Technology, Automation Eng., 1 Ta Hwa Rd., Chiung-Lin, Hsin-Chu, Taiwan China

In this paper, the laser surface alloying of Cu on Al substrates were investigated. The microstructures of Al-Cu intermetallic compounds in laser alloying layers were observed by scanning electron microscopy. The microstructure morphologies of Al-Cu intermetallic compounds depend on the laser process parameters and Cu addition conditions. The microhardnesses of laser alloying layers depend mainly upon Cu concentration in Al-Cu intermetallic compounds. The research results indicate that the fine microstructures under laser rapid heating can avoid cracking trend of Al-Cu intermetallic compounds and increase the surface hardness of Aluminum materials and the laser surface alloying of Cu on Al substrates might become a novel surface strengthening process for a partial surface on some parts made of Al or its alloy.

Decomposition Kinetics in Magnetic Bulk Amorphous Alloy Nd60Al10Fe20Co10: *Jinkui Zhao*[1]; *Peng Yuan*[2]; Xun-Li Wang[1]; Wei-Hua Wang[2]; Yan-Dong Wang[2]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, 701 Scarboro Rd., Oak Ridge, TN 37830 USA; [2]Chinese Academy of Sciences, Inst. of Physics China

In situ small angle scattering experiments on the magnetic bulk amorphous alloy Nd60Al10Fe20Co10 at the Advanced Photon Source revealed that the bulk metallic glass decomposes via two distinctively different processes. At temperatures below ~749 K, a metastable crystalline phase is formed and the decomposition is characterized by constant nucleation with little growth. Above ~782 K, the alloy transforms into a final crystalline phase via a process consistent with spinodal decomposition. These decomposition processes are correlated with the magnetic properties of the alloy.

Calculation of Prototype Ternary Phase Diagrams by the Cluster/Site Approximation: J. Zhu[1]; S.-L. Chen[1]; *F. Zhang*[2]; Y. A. Chang[1]; W. A. Oates[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., Madison, WI 53706 USA; [2]CompuTherm, LLC, 437 S. Yellowstone Dr, Ste. 217, Madison, WI 53719 USA

Unlike the Bragg-Williams (BW) approximation, the cluster/site approximation (CSA) can give phase diagram topologies similar to those obtained from the cluster variation method (CVM) and Monte Carlo simulations. The CSA has the big advantage over the CVM, however, in that the independent variables are the point probabilities as in the BW approximation rather than the cluster probabilities as in the CVM. This makes it suitable for being applied to multicomponent system since there are far fewer point probabilities than cluster probabilities in a multicomponent system, To date we have concentrated in applying the CSA to binary system prototype and real alloy phase diagrams. We have now extended the application of the CSA to some prototype ternary phase diagrams and will report the results obtained thus far.

Effect of Matrix Microstructure on Precipitation of Laves Phase in Fe-10Cr-1.4W(-Co) Alloys: *Keisuke Yamamoto*[1]; Yoshisato Kimura[1]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., 4259, Nagatsuta, Midori-ku, Yokohama, Kanagawa-ken 226-8502 Japan

Ferritic heat-resistant steels involving intermetallic Laves phase have drawn a growing interest for the enhancement of creep strength, while the brittleness of Laves phase may lower the toughness of the alloy. We believe it is possible to modify the morphology of Laves phase precipitates by controlling the a-Fe matrix microstructure. In order to make clear the influence of matrix microstructures on age-hardening, precipitation behavior of Laves phase was investigated by transmission electron microscopy (TEM). Matrix of the Fe-10Cr-1.4W-4.5Co (at%) alloy is controlled by heat treatments as to provide three types of microstructures; ferrite, ferrite+martensite and martensite. Alloys with ferrite and ferrite+martensite matrix show age-hardening behavior comprised of two hardness peaks. At around the first hardness peak, it is revealed by TEM observation that fine particles precipitate coherently within the ferrite matrix. In martensite matrix, most of Laves phase precipitates exist on laths and dislocations.

Effects of Tantalum on the Microstructural Evolution in Nickel-Base Superalloys: *Liang Jiang*[1]; Dheepa Srinivasan[1]; Ganjiang Feng[2]; Michael F. Henry[1]; [1]General Electric Company, Global Rsrch. Ctr., One Research Cir., Niskayuna, NY 12309 USA; [2]Power Systems, 300 Garlington Rd., Greenville, SC 29615 USA

Electron microscopy techniques were employed to study the effects of tantalum on the microstructural evolution in three General Electric nickel-base superalloys.†Three tantalum-containing alloys were heat treated over a range of times and temperatures.†Phase identification was made by scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy dispersion spectroscopy (EDS).†Tantalum is a carbide former and strongly partitions to the gamma-prime phase in these alloys.†It was found that the tantalum-containing alloys have different equilibrium phases.†With the assistance of thermodynamic simulation and microstructural observations, the mechanisms of phase stability behavior were proposed and correlated to alloying additions.

Thermoelectric Properties of a Semiconducting Intermetallic Compound β-Zn_4Sb_3 in its Bulk and Thin Film Form: *Lanting Zhang*[1]; Makusu Tsutsui[1]; Kazuhiro Ito[1]; Masaharu Yamaguchi[1]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan

Bulk and thin film Zn_4Sb_3 specimens were prepared by hot-pressing and sputtering-deposition respectively. Transport properties such as electrical resistivity, Seebeck coefficient and thermal conductivity were measured from R.T. to 673K to investigate the effects of microstructure and dimensionality on the thermoelectric properties. Inclusion of Zn or ZnSb phase in the Zn_4Sb_3 matrix results in an increased thermal conductivity in the bulk specimens while the power factor does not change much. The power factors of the thin films are higher than their bulk counterparts. The slightly decreased Seebeck coefficient of a Zn-rich thin film can be overcompensated by its low electrical resistivity. The measured thermal conductivity of thin film decreases when the film thickness is decreased and is almost half of the bulk value for a ~300nm thick film. This gives rise to a high ZT of at least 1.2 at 460K for the Zn_4Sb_3 thin films.

States of Cubic Crystal Lattices of Metals, Subjected by the Hair Pressure: *Mikhail Alexandrovich Baranov*[1]; Alexey Ivanovich Krymskikh[1]; Evgenya Vladimirovna Chernyh[1]; Mikhail Dmitryevich Starostenkov[1]; [1]Altai State Technical University, Dept. of Gen. Physics, Lenin st 46, Barnaul 656099 Russia

Electronic shells of atoms in metal were subdivided on internal and external. The electronic density of the internal shells was considered to be the same as in the isolated ions and were calculated using by Clementy wave functions. The density of electrons of the external shells was considered to be the superposition of spherical symmetric Gauss distributions, located on each atom. The parameter of Gauss distributions was calculated from stability conditions of the crystal at the given size of the elementary cell and the sublimation energy. Thus it was supposed that the interaction of nucleus charges and electrons is electrostatic only. Dependences of the crystal internal energy and the elementary cell volume from external pressure were obtained.

The Description of the Stable Crystal Lattices of HCP Metals and Alloy: *Mikhail Alexandrovich Baranov*[1]; Evgenyi Alexandrovich Dubov[1]; Evgenya Vladimirovna Chernyh[1]; Roman Yurjevich Rakitin[1]; Mikhail Dmitryevich Starostenkov[1]; [1]Altai State Technical University, Dept. of Gen. Physics, Lenin st 46, Barnaul 656099 Russia

The stable state of HCP of crystal lattices of metals and alloys cannot be described using by the spherical symmetric modelling pair interatomic potentials only. Thatís why it was proposed the interatomic potential, depended not only from the distance between the atoms, but also from orientation of the bond vector of the atoms pair. Various analytical forms of anisotropic interatomic potentials were considered. In the elementary case the interatomic function may be presented as Morse function, modulated by the orientational-dependent multiplier. The values of modules of elasticity and the defects formation energies, calculated within the framework of model, are correlated with experimental data for the majority of HCP metals and some ordered alloys.

Interaction of Vacancies and Planar Defects in HCP Lattices of Metals and Ordered Alloys: *Mikhail Alexandrovich Baranov*[1]; Evgenyi Alexandrovich Dubov[1]; Evgenya Vladimirovna Chernyh[1]; *Mikhail Dmitryevich Starostenkov*[1]; [1]Altai State Technical University, Dept. of Gen. Physics, Lenin st 46, Barnaul 656099 Russia

The formation energies and the stable atomic configurations of isolated vacancies, fault defects (FD), antiphase boundaries (APB) as well as their complexes are calculated, using the different forms of anisotropic interatomic potentials. It was shown the advantage of vacancies migrations to the FD plane or the APB plane. Vacancy settled down consistently on the crystal planes being away on various distances from plane of FD or APB. Besides the vacancy had the opportunity to occupy the different nonequivalent positions on the given plane.

The Description of Stable Cubic Lattices of Metals Taking into Account Local Electronic Distributions: *Mikhail Alexandrovich Baranov*[1]; Kseniya Nokolaevna Bumazhnikova[1]; Nadezhda Vasiljevna Brazovskaya[1]; Evgenya Vladimirovna Chernyh[1]; [1]Altai State Technical University, Dept. of Gen. Physics, Lenin st 46, Barnaul 656099 Russia

The electronic density in metals with FCC and BCC lattices is calculated on the basis of the empirical data about crystals and in the assumption that atoms cooperate only by means of coulomb forces. Internal shells of atoms were presented located on nucleus and did not influence on atoms interaction. The electronic density of external shells of each atom was approximated spherical by Gauss function. The external electronic shells were assumed overlapped and non-deformed. As a result of integration on the energy electrostatic par interatomic interaction density the analytical expression for interatomic potential as simple two-parametrical function is received. The calculated values of elasticity modules of metals correspond with the experimental values. The potentials, described the interactions of atoms of different sort in alloys, are easy constructed by using elaborated methodics.

Construction of Configurations of the External Electronic Shell of Atoms in Cubic Metals: *Mikhail Alexandrovich Baranov*[1]; Inna Viktorovna Dyatlova[1]; Evgenya Vladimirovna Chernyh[1]; Roman Yurijevich Rakitin[1]; [1]Altai State Technical University, Dept. of Gen. Physics, Lenin st 46, Barnaul 656099 Russia

The stability of crystal modifications of some elements, existence of ordered alloys may be explained even on conditions that interaction between atoms is pair and central. However, it is necessary to take into account more complicated approximation of function of interatomic interactions. Perhaps, the reason of it is in various distribution of electrons of external shells of different atoms. In considered model it was supposed that interaction between atoms is stipulated only by coulomb interaction of electrons of the external shells and non-compensated internal shells of nucleus charges. The electrons density of the external shells of each atom reached the maximal values on some distance from a nucleus. The analytical type of function of interatomic interaction dependent on three parameters is received. These parameters were selected from the empirical conditions of the stability of a crystal lattice of this or that element. The calculated characteristics of crystals, which are not included in the model are correlated with experimental data. The elaborated methodics allows to design the interatomic potentials in multicomponent systems.

Establishment of Correlations Between Electronic Shell Configurations Shell and Crystals Symmetry: *Mikhail Alexandrovich Baranov*[1]; Evgenya Vladimirovna Chernyh[1]; [1]Altai State Technical University, Dept. of Gen. Physics, Lenin st 46, Barnaul 656099 Russia

It is impossible to explain stability of numerous crystal lattices, molecules, compounds, and also phase transformations, using approach of pair internatomic interactions only. We started from assumptions, that, firstly, symmetry of a crystal lattice is completely determined by a configuration of external electronic casings of atoms and, secondly, interaction between atoms is carried out by means of electrostatic forces only. The search optimum ìatom configurationî consisted in a finding of ìgeometrical figureî itself of atom electronic distribution and in a finding of parameters of this distribution. The criterion of correctness of definition of these parameters was the conformity of the calculated characteristics of unicomponent crystal to their experimental values. Analytical expression for the noncentral pair potential describing interaction of atoms of different grade considering their mutual orientation is received.

Experimental and Modeling Study the Defects in the B2 Intermetallic Phase PdIn: *Mianliang Huang*[1]; Y. Austin Chang[1]; W. Alan Oates[2]; [1]University of Wisconsin-Madison, Matls. Sci. & Eng., 1509 Univ. Ave., Madison, WI 53706 USA; [2]13 Hawdon St., Eaglemont, Vic 3084 Australia

Based on the X-ray and bulk densities determined at room temperature on samples annealed at 900, 1000, and 1100°C and quenched in water/ice mixtures, the vacancy concentrations in the intermetallic phase PdIn have been obtained as a function of composition at these temperatures. A generalized thermodynamic model is presented which considers the existence of antisite and vacancy defects on both sublattices without any dilute solution approximations. This model is able to describe all the available vacancy data and is suitable for calculating these values at compositions and temperatures when such data are not available. In addition, the model permits the calculation of the concentrations of the minority point defects. The experimental results and the modeling analysis confirm that PdIn is a near-triple defect type of intermetallic.

Industrial Manufacturing of Mo-Si-B Intermetallic Compounds: Pascal Jehanno[1]; *Martin Heilmaier*[1]; Heinrich Kestler[1]; [1]Plansee AG, Tech. Ctr., Reutte, Tyrolia A-6600 Austria

Regarding their extraordinary high temperature strength, refractory metals (RM) would be the premier choice for ultra-high temperature structural applications. However, they suffer from poor oxidation resistance and embrittlement due to internal oxidation.[1] Significant progress in developing oxidation-resistant RM-based materials has been gained by alloying Mo with Si and B.[2] A successful industrial implementation, however, requires a judicious balance of ambient temperature fracture toughness and high temperature mechanical properties and oxidation resistance. To approach this target, an industrial manufacturing route for the production of Mo-Si-B intermetallic compounds has been explored at Plansee AG. Based on gas atomization of presintered rods and subsequent consolidation via hot isostatic pressing, ingots with a weight of up to 20 kg were produced. Processing along this route leads to good chemical and microstructural homogeneity yielding impurity levels as low as known for powder-metallurgical RM. Elevated temperature mechanical properties were characterized utilizing tensile and 3 point bending tests. The material showed extended plasticity at temperatures as low as 1200∞C, indicating good hot workability. Additionally, the high temperature oxidation resistance of bulk material and SIBOR (Plansee trademark) coated samples has been characterized. A porous oxide scale was observed on bulk samples exhibiting limited molybdenum oxide sublimation but no total protection. In contrast, SIBOR coated samples showed no evidence of Mo-oxide sublimation up to 1650∞C. [1]C.T. Liu and H. Inouye, Metall. Trans. 5 (1974), 2515. [2]H. Nowotny, R. Kiefer and F. Benesovsky, Plansee-Berichte f¸r Pulvermetallurgie, 1957, Bd. 5, 86.

Analysis of Surface Reaction Behavior for Generating Atomic Hydrogen on Ni_3Al Surfaces: *Masahiro Inoue*[1]; Katsuaki Suganuma[1]; [1]Osaka University, ISIR, 8-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan

The environmental embrittlement of aluminide intermetallics is believed to occur through the following steps; surface reaction (chemisorption) of H_2O or H_2 molecules for generating atomic hydrogen (H), followed by diffusion and segregation of H. In this paper, the surface reaction behavior of H_2 and H_2O molecules on surfaces of Ni_3Al alloys was investigated by using a molecular orbital simulation technique. When these molecules approach to the surfaces, the electron transfer from the surfaces to the adsorbates occurs to decompose the molecules. The orbital interaction between 3d component of Ni atoms and 1s component of H atoms is important for the generation of H. The concept of molecular orbital interaction is expected to be useful for analyzing the surface reaction mechanism.

Production of Iron Based Cellular Structure from Electric Arc Furnance Dust: Milton Manrique[1]; [1]Universidad Simon Bolivar, Dept. Ciencias de Materiales, Valle de Sartenejas, Baruta, Caracas 1080 Venezuela

The modern steel industry uses Direct Reduction Iron(DRI)-Electric Arc Furnace (EAF) technology to manufacture steel. A major drawback of this technology is the production of dust and fumes, products that may cause severe environmental problems. In Venezuela more than 80% of the steel are produced by this technology. EAF dust, fumes and sludge from steel industry can be used to produce different value-added products based on metallic foam technology. Porous iron or steel metallic structures can be used as building materials, heat exchangers, high temperature filters, catalyst supports, etc. In this article is described a method for producing iron-based cellular structure using as a precursor a particulate material from wet scrubbers of Midrex Iron Oxide Reduction Plant. This method involves the preparation of an open-type uniform cells foam structure by the immersion of a polymeric foam in slurries, and mixing the solid precursor with organic materials prior to foam formation, thermal decomposition, sintering and reduction of raw materials to the metallic state. Aqueous slurries were prepared from Midrex sludge, (98% Fe2O3), and sintering additives. Impregnated PU sponges were heated following three main stages: a) PU volatilization in a controlled inert atmosphere, b) Oxide Reduction and partially sintering and c) Sintering Consolidation. Sintered iron based metal foam obtained had continuous pores distribution with some closed-cells. Volumetric contraction value was approximately 24%. X-ray diffraction patterns of iron based metal foam obtained shows a perfect mach to the a-iron diffraction pattern, Û-Fe 06-0695*. Optical microscopy analysis revealed a typical low carbon iron based microstructure. Scanning electron microscopy result has shown a completed sintered porous network iron structure. Good magnetic properties were found the iron base cellular structure.

Structure-Analytical Parameters of Point Defects and their Complexes in Thin Film of Ni3Al Intermetallide: *Mikhail Dmitryevich Starostenkov*[1]; *Evgenya Alexsandrovna Dudnik*[1]; [1]Altai State Technical University, Gen. Physics, Lenin st 46, Barnaul 656099 Russia

Structure-analytical characteristics of thin film of Ni3Al intermetallide near point defects and their complexes, such as vacancies, point displacement defects, point interstitial defects are studied by the method molecular dynamics. It is shown, that the areas of quazicrystal (nanocrystal) state form near the complexes of point defects of the plain (111). The given areas are defined by the appearance of the elements of symmetry of the fifth order and other ones. The density of such formations is connected with the type of point defects, forming the complexes, and the distance of the interaction of point defects. The biggest distortions are observed near point intersti-

POSTERS

tial defects. The presence of similar complexes has the essential change of physical and physics-mechanical properties of the materials.

The Research of the Stability of Intermetallic Phases in Thin Film of Ni-Al System: *Mikhail Dmitryevich Starostenkov*[1]; Gennadyi Mikhaijlovich Poletaev[1]; [1]Altai State Technical University, Dept. of Gen. Physics, Lenin st 46, Barnaul 656099 Russia

The methods of the construction of diagramms of crystal phases stability for two-dimensional system Ni-Al was constructed and approved. The process of solution of Ni and Al particles and formation of intermetallic phases were studied by the method of molecular dynamics. The phenomena, connected with polymorphous reorientation of crystal phases, play an important role at the solution of the components in Ni-Al system at solid-phase reaction. The rounded Ni particle, having the ability of turn relatively to Al matrix to coincidence of crystal orientations of Ni and Al phases, has the least velocity of solution. The density of dislocations of discrepancy decreases. The biggest velocity of the solution is observed at the solution of Ni particle of irregular form in Al matrix and at the solution of Al layer between two disoriented Ni particles. High density of dislocation is stipulated by the presence of two interphase boundaries and front of ìcontraryî polymorphism in a form of intergrain boundary in Al volume.

Processing and Microstructure of Cr-Ta and Cr-Ta-Mo Composites Reinforced by the Cr2Ta Laves Phase: D. F. Wang[1]; *P. K. Liaw*[1]; C. T. Liu[2]; L. Heatherly[2]; E. P. George[2]; [1]The University of Tennessee, Matls. Sci. & Eng. Dept., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

The Cr-Ta alloy with an eutectic structure has a good combination of high strength and oxidation resistance at elevated temperatures up to 1,200∞C. It is an ideal candidate for ultrahigh-temperature applications. However, the material shows low ductility and fracture toughness at room temperature. A possible way to improve the ductility and fracture toughness is to obtain an aligned microstructure of eutectic Cr-based alloys, using a directional-solidification (DS) process, in which the feed materials with eutectic compositions are preferred. In the present work, a quantitative technique was developed to assist in monitoring and controlling the compositions of the Cr-based alloys at each stage of the processing at elevated temperatures. The eutectic composition of the binary Cr-Ta alloy was determined to be Cr-9.7 at. % (atomic percent) Ta, and a drop-cast ternary Cr-9.7 at. % Ta-1.0 at. % Mo alloy was found to possess a fully eutectic structure. This research was partially supported by the Division of Materi als Science and Engineering, US Department of Energy under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

Phase Transformations in Sputter-Deposited NiMn and PtMn Thin Films: *Peter F. Ladwig*[1]; Eric S. Linville[2]; Ying Yang[1]; Y. Austin Chang[1]; [1]University of Wisconsin-Madison, Matls. Sci. Prog., 1509 Univ. Ave., Madison, WI 53706 USA; [2]Seagate Technology, 7801 Computer Ave., Bloomington, MN 55435 USA

Antiferromagnetic Ni-Mn and Pt-Mn thin films are of interest to the magnetic storage industry for use in giant magnetoresistive (GMR) devices. However, sputter deposited, equiatomic, NiMn and PtMn thin films are observed to possess metastable, nanocrystalline, chemically disordered, face-centered-cubic (fcc) structures which are not antiferromagnetic. This study investigates the microstructural evolution of these films to the desired antiferromagnetic, chemically ordered structure. Differential scanning calorimetry (DSC) experiments on these films reveal exothermic peaks, identifying the phase transformation. Important kinetic parameters, such as the activation energy and enthalpy of transformation, are calculated from these DSC scans. The incorporation of the Johnson-Mehl-Avrami analysis is used to simulate reaction kinetics. Transmission electron microscopy (TEM), and electron/X-ray diffraction of annealed films characterize the nucleation and growth conditions of the polymorphic phase transformation to the antiferromagnetic structure. Differences in thermodynamic and kinetic properties with varying film thickness were also investigated.

Compositional and Microstructural Design Considerations for Phase Stability of Single Crystal Superalloy Systems: *R. M. Kearsey*[1]; J. C. Beddoes[1]; [1]Carleton University, Dept. of Mechl. & Aeros. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada

Compositional design of single crystal (SX) superalloys must ensure both mechanical and microstructural stability over extended periods of high temperature application. For complex turbine blade alloys with numerous alloying additions, it is very difficult to precisely relate changes in microstructural stability to individual or even combinations of elemental species. This uncertainty is even further magnified with the compositional variation inherent within as-cast SX microstructures due to selective element partitioning that occurs upon solidification. Macroscopic segregation during solidification between the initial dendritic solid phase and the final interdendritic regions is a leading factor for the formation of deleterious topologically close-packed (TCP) phases during high temperature applications. Hence, characterization of the propensity of TCP phase formation with respect to specific refractory addition levels would prove to be a vital design tool for high temperature alloy manufacturers. To address these issues, a series of six experimental SX alloys are examined with respect to TCP phase formation as a function of Re, W, and Ru addition levels and element partitioning behavior. Quantitative partitioning ratios are determined for each as-cast alloy utilizing EPMA techniques, highlighting that Ru decreases compositional segregation effects. The extent of homogenization with respect to solution heat treatment parameters is also characterized by means of parallel differential scanning calorimetry (DSC) and interrupted furnace heat treatment experiments. The results indicate the microstructural transformation and diffusion characteristics that occur during the homogenization process, providing insight into the degree of chemical segregation of each alloy. Precipitate dissolution is observed to occur interdendritically at lower temperatures followed by a higher solvus temperature for precipitates within the dendritic core regions. Finally, the propensity of TCP phase formation with respect to time and temperature is mapped for each alloy and related to nominal composition levels.

Phase Stability and Lattice Defects in Titanium Aluminides: *Rajendra R. Zope*[1]; Y. Mishin[1]; M. J. Mehl[2]; D. A. Papaconstantopoulos[2]; [1]George Mason University, Sch. of Computl. Scis., Fairfax, VA 22030 USA; [2]Naval Research Laboratory, Ctr. of Computl. Scis., Washington, DC 20375-5345 USA

First principles calculations have been performed for the energies of experimentally observed and some hypothetical crystal structures of the Ti-Al system: Al, Ti, TiAl, Ti3Al and TiAl3. A new semi-empirical embedded-atom-type interatomic potential has been constructed by fitting to the first-principles and experimental properties of these phases. Using this potential, atomistic calculations have been performed for lattice characteristics, point defects, planar faults, and other properties of Ti-Al phases with a particular emphasis on gamma-TiAl. Point-defect energies, entropies, and equilibrium concentrations in TiAl have been calculated. Atomic mechanisms of Ti and Al diffusion in TiAl have been studied by several computer simulation methods including long-scale molecular dynamics. The dominant diffusion mechanisms have been identified and diffusion coefficients have been evaluated using the transition state theory and theory of jump correlations. The calculated diffusion coefficients are found to compare well with the experimental data.

Processing, Microstructure, and Mechanical Behavior of an Eutectic Mo-Si-B Alloy: *S. Bejnood*[1]; C. T. Liu[2]; P. K. Liaw[1]; R. A. Buchanan[1]; [1]University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6115 USA

The processing and microstructures of a Mo-Si-B alloy with an eutectic composition (Mo-10Si-13B, at.%) have been studied. The arc-melting process, followed by drop casting into a copper chill mold, was employed to fabricate the Mo-Si-B alloy. The specimens were annealed at 1200, 1400, and 1600°C for controlling multi-phase microstructures. The microstructures of the heat-treated samples were compared with the as-cast samples in light of the grain size and phase morphology. The hardness of the intermetallic phases, Mo5Si3 (T1) and Mo5SiB2 (T2), was investigated, and the fracture toughness of the as-cast sample and the annealed sample at 1600°C was determined. Moreover, the compression and tension tests were performed for both as-cast and annealed samples at room temperature. The high-temperature optical floating zone furnace will be used to process the drop-cast materials with the aim of developing aligned lamellar microstructures in the Mo-Si-B alloy in order to improve the ductility and fracture toughness. The National Science Foundation (NSF) through an Interactive Graduate Education and Research Training (IGERT) Program (DGE-9987548) supported this research with Dr. P.W. Jennings and Dr. L.S. Goldberg as contact monitors. This research was also partially supported by the Division of Materials Science & Engineering, Office of Basic Energy Sciences, US Dept. of Energy under contract DE-AC05-00OR 22725 with UT-Battelle, LLC.

Consolidation of a Glassy Metal and Crystalline Powder Blend by ECAE: *S. N. Mathaudhu*[1]; J. T. Im[1]; J. N. Robertson[1]; R. Barber[1]; I. Karaman[1]; I. E. Anderson[2]; K. T. Hartwig[1]; [1]Texas A&M University, Dept. of Mech. Eng., College Station 77843-3123 USA; [2]Iowa State University, Ames Lab., Ames, IA 50011 USA

Warm equal channel angular extrusion (ECAE) in 90∞ tooling is used to consolidate Vitreloy 106a (Zr58.5Cu15.6Ni12.8Al10.3Nb2.8) plus 30 vol.% pure tantalum or tungsten blended powders at temperatures between Tg and Tx for the glassy metal phase. A fully dense, 12 mm diameter bar of amorphous matrix composite is achieved after only one extrusion. Metallography results show good infiltration of the amorphous Zr-based alloy in between crystalline particles and uniform consolidation. Hardness, DSC and XRD measurements indicate retention of amorphous character in the amorphous phase. ECAE appears to be a viable process for consolidating bulk amorphous metal matrix composite materials. Experimental results are reported.

Thermodynamic Studies of Fe-Cr-C Ternary System: Teng Lidong[1]; S. Malin[1]; A. Ragnhild[1]; *S. Seetharaman*[1]; [1]Royal Institute of Technology (KTH), Dept. of Matls. Sci. & Eng., Stockholm 10044 Sweden

In view of the applications of intermetallic carbides in the hard materials industries, the thermodynamic properties of the Fe-Cr-C system have been measured and the results were employed in an assessment of the system using the CALPHAD approach. Solid-state galvanic cell technique with CaF_2 as the solid electrolyte was employed to measure the activity of chromium in Fe-Cr-C system. XRD and SEM techniques were used to identify the stable phases. A two-sublattice model is used for the interstitial solution phases, and a substitutional solution model for the liquid phase. A set of parameters describing the Gibbs energies of the various phases is given, and the calculated phase diagrams are compared with the experimental data. The experiments are extended to Fe-Cr-C-N quaternary system.

What is the Lowest Achievable Critical Cooling Rate in Pd-Based Glasses?: *Tong D. Shen*[1]; Ulrich Harms[1]; Ricardo B. Schwarz[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

Although glass formation by the undercooling of melts has often been attributed to, and explained by, the need to avoid homogeneous nucleation, it is still debated whether one can really remove all heteronucleants. Recent experiments using mm-size samples purified in B_2O_3 flux have demonstrated critical cooling rates of 0.09 and 0.067 K/s in $Pd_{43}Cu_{27}Ni_{10}P_{20}$ and $Pd_{42.5}Cu_{30}Ni_{7.5}P_{20}$ respectively. These low critical cooling rates, however, seem to be still limited by heterogeneous nucleation. We have found that as-prepared $Pd_{44}Cu_{26}Ni_{10}P_{20}$ melt has a critical cooling rate of approximately 0.1 K/s, comparable to those reported earlier. However, after performing cyclic recalescence inside B_2O_3 flux, we were able to reduce the critical cooling rates to approximately 0.01 K/s. Then, an isothermal annealing (following cyclic recalescence) shows increased undercooled liquid stability. Our results suggest that heterogeneous nucleation is difficult to avoid and that cyclic recalescence may be a practical method to force impurities to the surface of the melt, where they can be neutralized by the flux.

Thermal Conductivity in $Pd_{40}Ni_{40-x}Cu_xP_{20}$ Bulk Metallic Glasses: *Ulrich Harms*[1]; R. B. Schwarz[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

The thermal conductivity of metallic glasses is needed to calculate cooling rates during the synthesis of bulk metallic glasses and also to estimate local heating associated with narrow shear instabilities during plastic deformation. From thermal conductivity data for rapidly quenched thin-foil metallic glasses, researchers have noticed that multi-element amorphous alloys have lower thermal conductivity than simple binary glasses. The thermal diffusivity and electrical conductivity of $Pd_{40}Ni_{40-x}Cu_xP_{20}$ bulk amorphous alloys with x = 0, 20, and 30 have been measured from 300 K to 400 K. An increase in the Cu contend of the glass lowers the thermal conductivity, concomitant with a decrease in electrical conductivity. The electrical conductivity depends more on the density of valence electrons than on the number of elements. The thermal conductivity can be described by the Wiedemann-Franz law for the electronic part and a constant phonon contribution comparable to that of non-metallic glasses.

Processing MMC Reinforced with Small-Size Particles: Some Theoretical Aspects and Experimental Application: *Vladimir Alekseevich Popov*[1]; Andrej A. Aksenov[1]; Artem V. Marmulev[1]; Ekaterina V. Vershinina[2]; *Donald R. Lesuer*[3]; Oleg M. Smirnov[1]; [1]Moscow Institute of Steel and Alloys, Dept. of Internatl. Cooperation, Leninsky Prospect, 4, Moscow 119991 Russia; [2]Lomonosov Academy of Fine Chemical Technologies, Powder Metall. Dept., Vernadskogo Prospect, 86, Moscow 117571 Russia; [3]Lawrence Livermore National Laboratory, L-175, Livermore, CA 94551 USA

The theoretical estimation of correlation between size of particles and phase-formation of soluble component (in particular, Si) on the reinforcement particles (in particular, SiC) in the melt has been investigated. Mathematical analysis and calculation of thermodynamic equation for a alloy melt showed the smaller a size of particle the more favorable Si-segregation near SiC particles. One can conclude that application of casting methods for processing MMC with small-size particles is unfavorable as far as it causes significant gradient in chemical composition and degradation of properties. That is why the mechanical alloying as a method for processing MMC with small-size particles selected as the most prospective one. It allows to obtain the final product without liquid phase. Mechanically-alloyed granules were compacted by dynamic methods. The final material investigated with optical, scanning electronic and transmission electronic microscopes showed homogeneity of particle dispersion and chemical compositi on in a cross-section. No defects were observed in the ìparticle-matrixî interface. Theoretical estimation and practical investigation revealed advantage of method of mechanical alloying for processing MMC with small-size particles.

Effect of Composition and Cooling Rate on the Transformation of Alpha to Gamma Phase in TiAl Alloys: *Vinod Kumar Sikka*[1]; Wallace D. Porter[1]; Tadeu Carneiro[2]; Edward A. Loria[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA; [2]Reference Metals Company, Inc., 1000 Old Pond Rd., Bridgeville, PA 15017 USA; [3]Consultant, 1828 Taper Dr., Pittsburgh, PA 15241 USA

Microstructural control is critical in controlling the mechanical properties of TiAl-based alloys. The microstructural control is possible in three possible ways: alloy composition, thermomechanical (dynamic), and static heat treatments. The refinement of microstructure due to ëdynamicí processing is effective in controlling the room-temperature ductility and workability of TiAl-based alloys, whereas ëstaticí heat treatments causing the grain coarsening and phase stabilization are required for increase in high-temperature strength properties. In the present study, we have investigated the effect of composition and cooling rate on the transformation temperature from hcp alpha-phase to fct gamma-phase in the alpha and gamma phase field. The data generated for the four alloy compositions will be presented. These data show that the alpha and gamma transformation temperature varies by 70∞C by changing the cooling rate over a factor of 40. Comparative to cooling rate, effect of composition on the alpha-gamma transformation is minimal. The phase transformation data were compared with the continuous cooling transformation (CCT) diagram published in the literature. The published CCT diagram for Ti-48Al alloy suggests that for cooling from 1410∞C at cooling rates of lesser than or equal to 5∞C/s or 300∞C/min, the microstructure shows a lamellar structure. The CCT diagram also indicates that for cooling rates lesser than or equal to 2∞C/s or 12∞C/min, the lamellar structure also contains grains. Based on the phase transformation data and published CCT diagram, the four alloys of this study were heat treated in order to maximize the generation of lamellar structure. Tensile properties of the four alloys, along with their microstructure, will be presented in the paper. Data will also be used to recommend both the alloy composition and heat treatment for obtaining the best combination of room-temperature ductility and strength for structural applications of TiAl-based alloys.

Enhanced Ductility in Coarse Grained Fe3Al Alloys: *Xiaoxu Huang*[1]; Wangyue Yang[1]; Zuqing Sun[1]; [1]Ris National Laboratory, Ctr. for Fundaml. Rsrch. Metal Struct. in Four Dimensions, Matls. Rsrch. Dept., Frederiksborgvej 399, Roskilde DK-4000 Denmark

Recently, an interesting phenomenon, i.e., large tensile elongation of up to 300-600%, has been observed at warm and high temperatures in several intermetallic alloys including Fe3Al based alloys with coarse grained structures. ìSuperplasticityî has thus been used to refer to as this phenomenon. However, different from the conventional fine grain structural superplasticity, grain boundary sliding is not expected to dominate the deformation in these alloys due to their coarse grained starting structures. Microstructural observations have showed the formation of a cell/subgrain structure within the initial coarse grains, indicating the occurrence of dislocation processes during the plastic deformation. In this work, a Fe3Al based alloy was tensile deformed over a temperature range of 750-950°C, and as expected large values of elongation were achieved. The deformation mechanisms are examined and discussed based on detailed microstructural observations and measurements of strain rate sensitivity and activation energy.

Diffusion Coefficients of Solutes in fcc and Liquid Al: Critical Review and Correlation: *Yong Du*[1]; Y. Austin Chang[1]; Zhanpeng Jin[2]; Baiyun Huang[2]; F. Y. Xie[3]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., 1509 Univ. Ave., Madison, WI 53706 USA; [2]Central South University, State Key Lab. for Powder Metall., Chabgsha, Hunan 410083 China; [3]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA

Diffusion coefficients of a number of solute elements, such as Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Mg, Si, Ga, and Ge, in fcc and liquid Al were critically reviewed. Activation enthalpies and pre-exponential factors for diffusion were obtained when extensive data are available in the literature. For those elements with limited experimental data or no data at all, these parameters were estimated from semi-empirical correlations. The calculated diffusion coefficients for these solutes resulting from the estimated parameters are in reasonably agreement with measured data.

Diffusion Mechanisms in Intermetallic Compounds: *Y. Mishin*[1]; [1]George Mason University, Sch. of Computl. Scis., 4400 Univ. Dr., MSN 5C3, Fairfax, VA 22030-4444 USA

A brief overview is given of the recent progress of the understanding of diffusion mechanisms in ordered intermetallic compounds, particularly the structural intermetallics TiAl and NiAl. Atomistic simulation tools for studying diffusion in ordered structures are briefly discussed and their applications are demonstrated. Diffusion in TiAl involves sublattice diffusion, inter-sublattice jumps, and three-jump vacancy cycles. Diffusion in NiAl is governed by several mechanisms operating concurrently, including sublattice diffusion of Ni vacancies, six-jump vacancy cycles, and other processes. The dominant mechanism depends on the temperature and off-stoichiometry. The diffusion coefficients obtained by atomistic calculations compare well with experimental data.

Green Technology to Recovery Value Metal Compounds from Molten Slags: *Zhitong Sui*[1]; Li Zhang[1]; Taiping Lou[1]; Zhida Sui[1]; [1]Northeastern University, Sch. of Matls. & Metall., Shenyang 110006 China

Based on several case studies in precipitating behavior of value metal compounds (VMC) in molten slags a green technology to recovery VMC from molten slags is proposed, in which three steps are involved: (1) The selective concentrating of dispersed VMC into the designed mineral phase in molten slag; (2) The selective coarsening of the designed mineral phase to critical grain size in molten slag; (3) The selective separating of the grown mineral phase in solidified slag from tailing by dressing or hydrometallurgy processes. The features of the technology are economic, clean, intensive and comprehensive. The utilization of recovered VMC such as Titanium, Boron, Vanadium, chromium and Iron compounds was summarized as examples of technology application. It was confirmed by experiments that the precipitating of the designed mineral phases like Perovskite (CaTiO3), Suanite (2MgO o B2O3), Spinel (MgO oCr2O3) and Magnetite (Fe3O4) in molten slags are obviously affected by operation factors such temperature, chemical composition, heat-treatment, additives and so on. The precipitating kinetics and mechanism of VMC from molten slags during solidification processes were also investigated.

Microstructural Processes in Irradiated Materials: Poster Session I

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

Tuesday 6:00-8:00 PM Room: Upper Level City-Side Corridor Foyer
March 4, 2003 Location: San Diego Convention Center

Proton Irradiation of Model and Commercial Pressure Vessel Steels: *Gary S. Was*[1]; Mark Hash[1]; G. Robert Odette[2]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., 1921 Cooley Bldg., Ann Arbor, MI 48109-2104 USA; [2]University of California-Santa Barbara, Dept. of Mechl. & Environl. Eng., Santa Barbara, CA 93106 USA

In an effort to understand the mechanisms of irradiation embrittlement in reactor pressure vessels steels, irradiation hardening and microstructure evolution were characterized in simple model alloys, model commercial alloys and commercial alloys irradiated with 3.2 MeV protons over a range of doses, dose rates and temperatures. Irradiation hardening was determined from Vickers hardness measurements and the microstructures were characterized using asynchronous small angle X-ray scattering (ASAXS). Results showed that copper content had the greatest effect on hardening, while Ni and Mn also played key roles. Composition played a large role in the hardening rate with dose. Comparisons of these results with the corresponding effects of neutron irradiation on the yield stress of the model alloys suggests that while neutrons and protons produced qualitatively similar trends, proton hardening was shifted to modestly higher dpa, depending on the assumed hardness to yield stress change relation.

Behaviour of Fe-Cu-Mn-Ni Model Alloys After Low Dose Neutron Irradiation: *Abderrahim Al Mazouzi*[1]; Lorenzo Malerba[1]; [1]Nuclear Research Centre SCK.CEN, Reactor Matls. Rsrch., LHMA, 200 Boeretang, Mol 2400 Belgium

The development and improvement of the simulation tools to predict the microstructural changes in nuclear reactor materials can only be based on adequate experimental data. These are being obtained and analysed at SCK.CEN, where 8 different model materials (from pure Fe to a real steel, including Fe-Cu and Fe-Mn-Ni model alloys) have been irradiated at 5 different flux/fluence conditions, in the experimental reactor BR2. It is indeed one of the largest neutron irradiation campaigns ever launched for the validation of computational tools and, in general, modelling purposes. A wide range of microstructural characterisations from transmission electron microscopy (TEM) and small angle neutron scattering SANS up to positron annihilation spectra (PAS) and internal friction (IF) studies in correlation with the mechanical properties (tensile tests and Hardness), are being used to assess the irradiation induced changes in these materials. In parallel with the experimental activity, an effort is being made to gather computational techniques that allow direct comparison between simulation and experiment such as TEM image and PAS calculations.

Ultra-Fast Nano-Scale Phase Transitions Induced by Collisions in Solids: *Alfredo Caro*[1]; Edmundo Lopasso[1]; Magdalena Caro[1]; [1]Centro Atomico Bariloche, Bariloche 8400 Argentina

We study the thermodynamic forces acting on the evolution of the nanoscale regions excited by collisions of energetic particles into solid targets. We analyze the role of diffusion, thermo-migration, and the liquidus-solidus two-phase field crossing, as the system cools down from the collision-induced melt under different conditions of energy deposition. To determine the relevance of these thermodynamic forces, solute redistribution is evaluated using molecular dynamics simulations of equilibrium Au-Ni solid solutions. At low collision energies, our results show that the quenching of spherical cascades is too fast to allow for solute redistribution according to equilibrium solidification as determined from the equilibrium phase diagram (zone refining effect), and only thermo-migration is observed. At higher energies instead, in the cylindrical symmetry of ion tracks, quenching rate is in a range that shows the combined effects of thermo-migration and solute redistribution that, depending on the material, can reinforce or cancel each other. These results are relevant for the interpretation of the early stage of radiation damage in alloys, and show that the combination of ultra-fast but nano-scale characteristics of these processes can still be described in terms of linear response of the perturbed system. Additionally, we analyze the implications of the thermodynamics implicit in the models used to describe large scale molecular dynamics of alloys, with particular emphasis on the Au-Ni and Fe-Cu systems.

Molecular Dynamics Simulation of Phase and Precipitate Stability in the Al(Ni) System Under Irradiation: *Alexandre Cuenat*[1]; Rolf Gotthardt[2]; Robin Schaueblin[3]; [1]Harvard University, Div. of Eng. & Appl. Scis., 9 Oxford St., Cambridge, MA 02138 USA; [2]Ecole Polytechnique Federale de Lausanne, Lab. díÈtude des propriÈtÈs des microstructures, PHB-Ecublens, Lausanne, VD 1015 Switzerland; [3]Ecole Polytechnique Federale de Lausanne, Plasma Physics Rsrch. Ctr., Fusion Tech., Villigen-PSI, AG 5232 Switzerland

Medium energy nickel ions implanted into aluminium produce $Al_{0.75}Ni_{0.25}$ amorphous precipitates in a crystalline matrix with a high dislocation density. Molecular dynamics (MD) simulations using embedded atom method of displacement cascades in Al, Ni-Al solid solutions and inhomogeneous samples containing amorphous or crystalline Al_3Ni precipitates are presented. Influence of 5 keV displacement cascades on the formation and stability of the amorphous clusters is studied. It is shown that no segregation occurs inside the displacement cascade for this system. Crystalline precipitates with a Ni concentration larger than 20 at. % are amorphized by the cascade. Large enough amorphous clusters remain stable under the same condition. It is also shown that displacement cascade displace Ni atoms in interstitial position after a collision cascade for low Ni concentration alloy. Based on these results and on MD simulation of Ni migration in Al, a new model is presented to explain the experimental observations.

Some Features of the Interactions Between Point and Extended Defects in Metals: Angels Puigvi[1]; *Anna Serra*[1]; Nieves de Diego[2]; Yuri N. Osetsky[3]; David J. Bacon[3]; [1]Universitat Politecnica de Catalunya, Matematica Aplicada III, Jordi Girona 1-3, modul C-2,

Barcelona 08034 Spain; [2]Universidad Complutense, Fisica de Materiales, Madrid Spain; [3]The University of Liverpool, Matls. Sci. & Eng., Dept. of Eng., Liverpool L69 3GH UK

Interactions between radiation-induced defects and the existing microstructure give rise to the microstructure evolution. Reactions involving point and extended defects (defect clusters, dislocations and dislocation loops) are crucial for the estimation of changes in defect concentration. Such interactions are usually described within a continuum approach using elasticity theory that cannot involve the features of the crystalline structure and particular characteristics of defects. We present results of atomic-scale computer modelling of interactions between vacancies and clusters of self-interstitial atoms in bcc, fcc and hcp metals. This type of reaction is one of the most frequent because formation of SIA clusters, particularly glissile ones, is observed in displacement cascades in all metals and leads to significant supersaturation of vacancies. It is found that the interaction depends on the structure of the cluster and it is not necessarily described as an annihilation reaction. Moreover, it may lead to a change in the cluster mobility. The results are discussed from the viewpoint of differences observed in properties of irradiated metals of different lattice structure.

Replacement Collision Sequences Revisited by MD and its BCA: *Charlotte Becquart*[1]; *Abdelkader Souidi*[2]; Marc Hou[3]; [1]UniversitÈ Lille-1, Lab. de MÈtallurgie Physique et GÈnie des MatÈriaux, UMR 8517, Villeneuve díAscq, CÈdex F-59655 France; [2]Centre Universitaire de Saida, En-nasr, Saida 2000 Algeria; [3]UniversitÈ Libre de Bruxelles, Physique des Solides IrradiÈs CP234, Bd du Triomphe, Brussels B-1180 Belgium

Linear and replacement collision sequences in collision cascades are a characteristic effect of the crystalline structure of solids. Some sequences can be very long and transport matter far away from the cascade. As a result, the defects created by these sequences will not be able to recombine with the other defects. We have investigated by MD and its BCA the formation of such sequences and observed that they can be initiated by PKA of energies well above the focusing thresholds. The influence of the potential on the RCS length is also very important and will be discussed.

Radiation-Induced Defect Accumulation and Recovery Behavior of Proton Irradiated RPV Steels Studied by Positron Annihilation: *Eung-Seon Kim*[1]; Joo-Hag Kim[1]; Sang-Chul Kwon[1]; Jun-Hwa Hong[1]; [1]Korea Atomic Energy Research Institute, Nucl. Matls. Tech. R&D Team, PO Box 105, Yuseong, Daejeon 305-600 Korea

Radiation-induced defect accumulation and recovery behavior of proton irradiated RPV steels was investigated based on the microstructural difference through the application of intercritical heat treatment (IHT). Positron annihilation lifetime spectrometer was used for the characterization and quantification of radiation-induced defects. Microhardness tests were performed to estimate the activation energy of the recovery process after annealing experiments. Apparent differences between IHT treated and conventionally heat treated samples were observed in the results of positron lifetime and hardness measurements. Especially the application of the IHT increased resistance to radiation damage. The improved effect of the IHT on the radiation sensitivity of this steel was closely related with the newly formed composite structure of hard tempered martensite and double tempered-bainite.

Theoretical Study of Small Clusters Embedded in Matrix Under Strong Electronic Excitation: *Gervais Benoit*[1]; Sebastien Josse[1]; Eric Suraud[2]; [1]CEA, CIRIL, rue Claude Bloch, BP 5133, Caen 14070 France; [2]Paul Sabatier University, 118 rte. de Narbonne, Toulouse 31062 France

Small clusters or molecules embedded in wide band gap matrix is a well defined localised system that permits to study experimentally and theoretically the effects of electronic excitation by fast charged particles or short laser pulses. From a theoretical point of view, this system can be modelled by ab initio molecular dynamics, that allows to study explicitly the coupling between the electronic system and the other pieces of the whole system. The electronic dynamics of the cluster is described within time dependent local density approximation (TDLDA) with self-interaction corrections (SIC). The electronic system is coupled to the ions of the cluster and to the atom of the matrix described by classical mechanics. The polarisation of the matrix is accounted for by a set of dipoles located on each matrix atom. We shall focus on small sodium clusters embedded in an argon matrix and we present as a test case a comparison between Na_8 in the Ar matrix and a free Na_8 under a short laser pulse. Particular emphasis will be given to the coupling between the cluster and the matrix, and to the coupling between electronic excitation and the ionic dynamics.

ëJerkí, An Event-Based Kinetic Monte Carlo Model: Application to Microstructure Evolution in Irradiated Alpha-Iron: *Jacques Dalla Torre*[1]; Nguyen Van Doan[1]; Jean-Louis Bocquet[1]; Georges Martin[1]; [1]CEA Saclay, DMN/SRMP, Bat. 520, Gif-sur-Yvette 91191 France

Kinetic Monte Carlo simulations are widely used to investigate microstructure evolution of materials under irradiation. ëJerkí has the peculiarity to sample directly the probability that two defects meet during a given time interval (e.g. interstitial vacancy recombination or defect elimination at sinks). As a result, Jerk allows to study large time and length scales comparable with the experiments even under high defect diffusion conditions. We apply jerk to the electron or ion irradiation of alpha-iron and we present some results as a function of irradiation conditions e.g. temperature. Interstitial, vacancies, impurities and their aggregate are included in the simulations.

Microscopic Simulation of Radiation Damage in Relevant Materials for Fusion Reactors: *J. Manuel Perlado*[1]; MarÌa J. Caturla[2]; Dario Lodi[3]; *Jaime Marian*[4]; Luis Alberto MarquÈs[5]; Max Salvador[1]; Fernando Mota[1]; [1]Universidad PolitÈcnica de Madrid, Instituto de FusiÛn Nuclear (DENIM), E.T.S. Ingenieros Industriales, JosÈ Gutierrez Abascal, 2, Madrid 28006 Spain; [2]Lawrence Livermore National Laboratory, Universidad Alicante; [3]Instituto FusiÛn Nuclear, SCK CEN/MOL; [4]Lawrence Livermore National Laboratory, Instituto FusiÛn Nuclear (DENIM); [5]Universidad de Valladolid

This paper will present our simulation models and results for radiation damage in optics (SiO2), and structural components: SiC and ferritic steels. After results published on densification of SiO2, we will present Molecular Dynamics results on cascade formation by recoils of 2-5 keV in such material, and discussion of interatomic potentials. Last results of diffusion parameters in SiC will be reported by using a new model of tight-binding molecular dynamics. Multiscale Modelling simulations will be presented to understand the basic defect structures and their inter-conversion in Fe that explain some of the basic mechanisms in the radiation damage of ferritic alloys. The basic understanding of irradiation in Fe provided by Microscopic Simulation (using kinetic MonteCarlo) will be supported with discussion of experimental programs (REVE, VENUS-II). The validation of diffusion parameters in Fe will be discussed on the light of previous comparison, and the effect of pulse irradiation in Fe re-evaluated.

EDIP Modeling of Irradiation Defect Configurations and Elastic Responses in Silicon: Clark L. Allred[2]; Xianglong Yuan[1]; Martin Z. Bazant[1]; *Linn W. Hobbs*[1]; [1]Masschusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Rm. 13-4050, 77 Massachusetts Ave., Cambridge, MA 02139-4307 USA; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng. & Nucl. Eng., Rm. 13-4054, 77 Massachusetts Ave., Cambridge, MA 01239-4307 USA; [2]Charles Stark Draper Laboratory, 555 Technology Sq., Cambridge, MA 02139 USA

Defects in silicon introduced in even small concentrations can seriously compromise sensitive microelectromechanical (MEMS) devices in operating in radiation environments, through changes in dimension and elastic properties emanating from the mixture of isolated defects, defect aggregates and amorphous regions induced by displacive radiation processes. Such changes may be too small at the affecting fluences to measure by conventional analytical techniques, so computer simulation of defect configurations and their effects is an effective approach for predicting performance degradation. The configurations of a large number of potential interstitial and vacancy defects have been modeled using the environment dependent interatomic potential (EDIP) developed for Si, and their formation volumes and effect on 0 K elastic constants of crystalline Si calculated. The 0 K elastic constants for several models of amorphous Si were also calculated, as well as those of a composite comprising an amorphous region embedded in a crystalline matrix. Both vacancy and interstitial defects in random combination were found, regardless of configuration, to decrease Youngís modulus approximately linearly with defect content up to 0.3%, while Frenkel pairs in every case increased the volume. Amorphous zones of all configurations decreased the modulus.

Kinetic Monte Carlo Simulation of Cascade Ageing in FeCu Alloys: A Comparison of Techniques and Parameter Sets: *Lorenzo Malerba*[1]; Charlotte Becquart[2]; Christophe Domain[3]; Marc Hou[4]; [1]SCK.CEN, Reactor Matls. Rsrch. Unit, Boeretang 200, Mol 2400 Belgium; [2]UniversitÈ de Lille I, LMPGM-UMR 8517, Villeneuve díAscq 59655 France; [3]EDF-R&D, MMC, Les Renardieres, Moret-sur-Loing 77750 France; [4]UniversitÈ Libre de Bruxelles, Dept. Physique des Solides IrradiÈs, Brussels 1050 Belgium

A large effort is devoted, at international level, to the simulation of radiation effects in reactor pressure vessel steels. Key issue is the kinetics of formation of Cu-vacancy complexes in ferritic matrix, starting from displacement cascade debris. In this work molecular dynamics and corresponding binary collision approximation displacement cascades have been ìagedî using two different kinetic Monte Carlo techniques and different sets of parameters in Fe-Cu alloys. This exercise allows an assessment of the cascade features that mostly influence their long term evolution, as well as the effect of the computational technique used for the simulation on the physical description given by the model. The conclusions of this work should help guide the development of complex multiscale modelling suite of codes for the simulation of irradiation effects in materials.

TEM Characterization of Ion Damage in Fe and Fe-Cu Model Alloys: *Mercedes Hernandez-Mayoral*[1]; Dolores GÛmez-BriceÒo[1]; [1]CIEMAT, Dept. de Materiales Estructurales en Plantas EnergÈticas, Avenida Complutense, 22, Madrid 28040 Spain

The general objective of this work is to develop predictive tools to assess and quantify the effects of irradiation in reactor pressure vessel steels. A simple experiment was performed to obtain suitable data for a direct comparison with modeling results aimed at validate parameters used in a computational simulation carried out in parallel. This work is performed inside the Spanish Venus Project. Experimental conditions were chosen looking for a commitment between limits in computational simulation and in experimental facilities. Fe and Fe-Cu thin foils, to be characterized by TEM, were irradiated at 573K with 150keV iron ions, dose rate $4x10^{15}$ions∑m^{-2}∑$^{-1}$, and dose $2x10^{19}$ions∑m^{-2}. Preliminary results in Fe show an homogeneously distributed damage in the form of dislocation loops. Loop size variation is large, ranging from few nanometers to around 200 nm, with densities around $5x10^{20}$ m^{-3}. Interstitial dislocation loops with b of the type 1/2<111> and <100> were observed.

Effect of Neutron Irradiation on the Deformation Behavior of Alloys X-750 & 625: *Ram Bajaj*[1]; M. G. Burke[1]; [1]Bechtel Bettis, Inc., 814 Pittsburgh-McKeesport Blvd., W. Mifflin, PA 15122 USA

To develop and understanding of the effect of neutron irradiation on the mechanical behavior of Ni-base alloys, a detailed microstructural characterization study using analytical electron microscopy (AEM) has been performed on precipitation-hardened Ni-base alloys (Condition HTH AlloyX-750 and direct-aged Alloy 625). The tensile properties and deformation substructure of the unirradiated and irradiated alloys were compared to assess the role of irradiation damage on the subsequent mechanical behavior. Both as-heat-treated and prestrained material conditions were studied. The results of this investigation showed that deformation in irradiated Alloy 625 is dominated by finely spaced slip bands whereas a much coarser deformation structure developed in Alloy X-750. AEM analysis also revealed the existence of crack initiation associated with localized slip band intersection at intergranular carbides in Alloy X-750.

The Effect of Point Defect Transients in Low-Temperature Irradiation Experiments: *Roger E. Stoller*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, MS-6151, PO Box 2008, Oak Ridge, TN 37831-6151 USA

The kinetic models used to simulate the response of materials to irradiation typically assume that the point defect concentrations are in equilibrium with the existing sink structure. This assumption is generally valid if: (1) the response time of the point defect concentrations is much faster that the time scale on which the microstructure changes, and (2) point defect diffusion yields the quasi-steady-state point defect concentrations in a much shorter time than the duration of the experiment being simulated. For irradiation temperatures below about 250∞C, the time required to obtain steady state point defect concentrations can become long enough to influence the irradiation response of the material. A point defect transient effect has previously been invoked to explain unexpectedly high creep rates observed in low-temperature irradiation experiments. The hydraulic tube irradiation facility in the High Flux Isotope Reactor (HFIR-HT) is being used to further explore the potential impact of the point defect transient. Irradiation times in the HFIR-HT can be precisely varied from seconds to days to achieve a range of doses at a relatively high dose rate. Small tensile samples of AISI 316 stainless steel and an A533B reactor pressure vessel steel are being irradiated in a series of experiments at temperatures between 60 and 300∞C. Irradiations are being carried out to compare the effect of continuous irradiation for a fixed time with multiple, interrupted irradiation cycles that sum to the same total time. Differences between the damage accumulation under continuous and interrupted irradiation should appear when the total irradiation time is on the order of the time required for the vacancy concentration to reach steady state. The results of these experiments will be compared to model calculations, and the implications of the experiments for commercial reactor components will be discussed.

Self-Interstitial Diffusion in Vanadium: *Seungwu Han*[1]; Luis A. Zepeda-Ruiz[2]; Graeme Ackland[3]; Roberto Car[4]; David J. Srolovitz[1]; [1]Princeton University, Princeton Matls. Inst., Bowen Hall, 70 Prospect Ave., Princeton, NJ 08544 USA; [2]Lawrence Livermore National Laboratory, Chem. & Matls. Sci. Direct., PO Box 808, L-353, Livermore, CA 94551 USA; [3]University of Edinburgh, Physics & Astron., Edinburgh EH9 3JZ Scotland; [4]Princeton University, Chem., Princeton, NJ 08544 USA

The evolution of the mechanical properties of vanadium for first wall fusion reactors will be determined, in large part, by the evolution of the point defect distributions. In this presentation, we employ a combination of first-principles calculations, development of interatomic potentials, and molecular dynamics (MD) simulations to determine the stable interstitial configuration and how interstitials migrate. In particular, the first-principles calculations show that the <111>-dumbbell is stable and migrates with very low activation energy. We developed a new interatomic potential for V that is designed to give point defect properties matching the first-principles results and employ it in MD simulations of point defect trajectories. At low and intermediate T, the diffusion is one-dimensional, but becomes three-dimensional at high T. The apparent activation energy for migration increases with temperature as a result of a complex correlation effect, which will be described in this presentation.

Multi-Scale Modeling of Radiation Effects: First Results Obtained with RPV-1: *StÈphanie F. Jumel*[1]; Jean-Claude Van Duysen[2]; Christophe Domain[1]; Jacky Ruste[1]; Alain Barbu[3]; Charlotte Becquart[4]; Alexandre Legris[4]; Eric Van Walle[5]; Lorenzo Malerba[5]; David Bacon[6]; Marc Hou[7]; Manolo Perlado[8]; Mercedes Mayoral[9]; Philippe Pareige[10]; [1]ElectricitÈ de France, MMC, Les RenardiËres, Rte. de Sens, Moret sur Loing 77818 France; [2]ElectricitÈ de France, EIfER, Universitat Karlsruhe, Adernauerring, 32, Karlsruhe 76 Germany; [3]CEA, SESI, Ecole Polytechnique, Gif sur Yvette 91191 France; [4]CNRS, LMPGM, UniversitÈ de Lille1, Villeneuve díascq, Cedex 59655 France; [5]SCK-CEN, Boeretang, Mol 2400 Belgique; [6]University of Liverpool, Dept. of Eng. UK; [7]UniversitÈ Libre de Bruxelles, LSI, Bruxelles Belgique; [8]Universidad PolitÈcnica de Madrid, Inst. of Nucl. Fusion, Madrid Spain; [9]CIEMAT, Madrid Spain; [10]CNRS UniveristÈ de Rouen, Groupe de Physique des MatÈriaux, Mont Saint Aignan 76821 France

Within the REVE project (Reactor for Virtual Experiment), several organizations among US, Japan and Europe have joined their efforts to build numerical tools, so called Virtual Reactors, to perform multi-scale modeling of radiation effects in materials. A first virtual reactor, called RPV-1, is now available and allows for simulating the behavior of RPV steels under irradiation. RPV-1 is a suite of seven codes and three libraries of atomistic calculations. Its input parameters are the neutron spectrum, time and temperature of irradiation, the chemical composition of the steel and the mechanical test temperature and deformation rate. Its predicted results are the irradiation-induced increase of yield stress and the evolution of the microstructure. Several tens of parameters (some are well known while other are estimated) are used in those codes to describe the physical properties of the material at the different time and space scales. In the proposed article, we present the quantitative results obtained with RPV-1 using a first set of parameters as well as simulations performed to evaluate the influence of the main material characteristics and irradiation parameters on the results.

The Elastic Fields of Sub-Surface Dislocation Loops: A Comparison Between Analytical Continuum-Theory Solutions and Atomistic Calculations: Peihua Jing[1]; *Tariq Khraishi*[1]; Brian Wirth[2]; [1]University of New Mexico, Mechl. Eng. Dept., Albuquerque, NM 87131 USA; [2]Lawrence Livermore National Laboratory, Matls. Sci. & Tech. Div., Livermore, CA 94551 USA

The effect of irradiation on the microstructure of metals is typically a high number of damage defects in the form of interstitial platelets. In dislocation theory these damage clusters are treated as prismatic dislocation loops. The elastic fields of such loops are solved analytically in the literature typically for loops in an infinite material. Near a free surface, these fields are no longer valid as image stresses affect the solution. In this work, an analytically-derived solution for the elastic fields of a sub-surface rectangular dislocation loop in a half material is presented. The analytical solution is then compared to molecular dynamics (MD) results for Tungsten. A component of the work focuses on the topology change of the free surface due to the presence of these damage loops. Where applicable, comparison to experimental results will also be discussed.

Temperature Dependence of Irradiation Effects in Pure Titanium: *T. Leguey*[1]; Robin Sch‰ublin[2]; N. Baluc[2]; Max Victoria[2]; [1]Universidad Carlos III de Madrid, Dept. de FÌsica, LeganÈs 28911

Spain; [2]CRPP-EPFL, Fusion Tech. Matls. Div., Villigen-PSI 5232 Switzerland

The microstructural modifications due to irradiation in hcp pure metals and their consequences on the mechanical properties have been investigated. Experimental results for proton irradiated pure polycristalline titanium are presented and discussed. Samples have been irradiated with 590 MeV protons for a low dose range at two different temperatures, RT and 523 K. Defect sizes and densities as a function of dose have been measured by means of TEM observations, and hardening (increment of the yield stress) has been obtained from tensile stress-strain curves. The dose dependence of the irradiation hardening has been found to change with the investigated temperatures. These results are discussed in terms of the main deformation mechanism operating at each temperature.

A Comparison of Embrittlement Response of Linde 80 Welds Irradiated in Power and Test Reactors: *W. A. Pavinich*[1]; M. A. Sokolov[2]; M. J. DeVan[3]; [1]Constellation Nuclear Services, Knoxville, TN USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Framatome ANP, Lynchburg, VA USA

Linde 80 welds have been irradiated in Westinghouse-designed, B&W-designed, and test reactors. Small Angle Neutron Scattering (SANS) was used to evaluate microstructural changes occurring during irradiation. Then simple Russell-Brown model was applied to SANS data to predict radiation-induced hardening and embrittlement. In all but a few isolated cases, changes in mechanical test data are similar regardless of irradiation location. SANS data indicate similar volume fractions, number density, and effective radius of irradiation-induced copper-rich precipitates for welds irradiated in the test and power reactors. The predictions of the Russell-Brown model using the SANS data are in agreement with the increases in yield strength as a result of irradiation. Irradiation-induced shifts in Charpy 30 ft-lb temperature and the Master Curve index temperature, To, follow a linear trend with the square root of the volume fraction of the irradiation-induced precipitates measured by SANS. This paper presents the evidence for an effective copper level for Linde 80 welds. There are three categories of evidence: 1) physical, 2) mechanical testing, and 3) statistical. It was determined that the effective copper level for Linde 80 welds is approximately 0.25 wt%, which is less than the bulk copper content for many Linde 80 welds. The physical evidence is derived from the SANS data where volume fraction, number density, and effective radii are similar regardless of bulk copper content above 0.25 wt %. Changes in mechanical test data resulting from irradiation are similar for weld with bulk copper content above 0.25 wt %. Above a bulk copper content of 0.25 wt %, the changes in mechanical properties for Linde 80 welds can be considered a single population.

Atomistic Modelling of Precipitate Hardening in Iron-Copper Alloys: *Yuri N. Osetsky*[1]; David J. Bacon[1]; [1]The University of Liverpool, Matls. Sci. & Eng., Brownlow Hill, Liverpool L69 3GH UK

Ferritic pressure vessel steels used power reactors can suffer radiation-induced precipitation of copper and other elements such as manganese and nickel. These precipitates are about 2nm in diameter and remain coherent at the usual neutron doses experienced by pressure vessels and are believed to contribute significantly a shift in DBTT, thereby limiting the lifetime of vessels. Conventially, the hardening due to the coherent precipitates has been treated in terms of the Russell-Brown modulus hardening model. This model is based on the difference in elastic properties of precipitates, and matrix and does not include any atomic level mechanisms that may take place at such small scale. Recently, molecular statics simulation of the interaction of a screw dislocation with coherent copper precipitate in iron has shown that an induced bccfifcc transformation of copper can make a significant contribution to the hardening. In the present work we are extending this research to investigate edge dislocations and dynamical effects in the atomic-level mechanisms of precipitate strengthening. We have studied the motion of the Ω<111>{110} edge dislocation in iron as it cuts coherent Cu-precipitates with diameter up to 5nm under applied stress or strain. Different dislocation and precipitate densities and strain rates have been modelled. Specific phenomena such as formation of vacancies, dislocation climb and precipitate phase transformations have been observed, underlying the importance of atomic-scale consideration of this problem.

Atomic-Scale Dynamics of Dislocation Interaction with Vacancy Agglomerates in Neutron Irradiated Bcc Iron: *Yuri N. Osetsky*[1]; David J. Bacon[1]; Bachu N. Singh[2]; [1]The University of Liverpool, Dept. of Eng., Brownlow Hill, Liverpool L69 3GH UK; [2]Risoe National Laboratory, Matls. Rsrch. Dept., PO Box 49, Roskilde DK-4000 Denmark

Generation of defect clusters occurs directly in high-energy displacement cascades in metals. Further evolution of microstructure can result in formation of extended defects such as dislocation loops, vacancy voids and/or stacking fault tetrahedra, which are visible in TEM. Recent positron annihilation studies of neutron-irradiated iron have demonstrated that there is a significant population of small clusters containing up to a few tens vacancies that are invisible in TEM. In the present work we have studied the hardening due to the vacancy component of radiation damage in bcc iron. Two types of obstacle for dislocation motion have been considered: (a) compact three-dimensional microvoids containing up to ~5000 vacancies (5nm in diameter) and (b) loose vacancy clusters with a local vacancy concentration of 30 to 50%. Molecular statics and dynamics have been used to estimate the critical stress necessary to overcome such obstacles. The stress has been determined as a function of obstacle size and separation, crystal temperature and dislocation velocity. The mechanisms involved in a dislocation overcoming these two types of obstacle have been analysed.

Microstructural Processes in Irradiated Materials: Poster Session II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS)
Program Organizers: Lance L. Snead, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37830-6138 USA; Charlotte Becquart, Universite de Lille I, Laboratoier de Metallurgie Physique Et Genie des Materiaux, Villenueve síAscq, Cedex 59655 France

Tuesday 6:00-8:00 PM   Room: Upper Level City-Side Corridor Foyer
March 4, 2003   Location: San Diego Convention Center

The Sink Strength of Nanosized Grain Boundaries on Interstitial Defects: *M. Samaras*[1]; P. M. Derlet[1]; H. Van Swygenhoven[1]; M. Victoria[2]; [1]Paul Scherrer Institut, Villigen PSI CH-5232 Switzerland; [2]CRPP-Fusion Technology Materials, EPFL, Villigen PSI CH-5232 Switzerland

It is well known that grain boundaries (GB) act as sinks for interstitial atoms created during irradiation and that this results in a denuded zone around the GB. Experiments and modelling confirm this trend, showing a zone of 3 to 20nm present around a discrete dislocation free of interstitial defects. Molecular dynamics simulations are performed to study defect production in irradiated nanocrystalline Ni with mean grain sizes of 5, 12 and 20nm. It is shown that the interstitials are attracted to nearby GBs via replacement collision sequences or 1D/3D motion, where regions of misfit in the grain boundaries, present as GB dislocations, and triple junctions, both containing free volume, are responsible for the sink strength. The sink strength of GBs is investigated in terms of grain size and the non-equilibrium state of the GBs. The structure of the vacancy dominated defects remaining in the grain is shown to form stacking fault tetrahedra in the nanosized grains at energies of 10keV and greater, which would result in material hardening. (PRL 88(2002)125505, Phil. Mag. A 2002 in press.)

Atomistic Simulations of Radiation Damage in Materials for Inertial Fusion Energy Applications: Alison Kubota[2]; Lilian Davila[1]; Luis Zepeda-Ruiz[1]; *Maria J. Caturla*[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., L-353, PO Box 808, Livermore, CA 94550 USA; [2]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., PO Box 808, Livermore, CA 90550 USA

Understanding degradation of materials properties during irradiation is critical in the development of future fusion reactors. The lack of pulse 14 MeV neutron sources results in extrapolation of the experimental observations to those conditions that will be attained in fusion reactors. Under these circumstances predictive models can play a significant role in the selection and development of materials for these extreme conditions. We have focused our work in the study of radiation damage in two materials: fused silica and graphite. In a fusion reactor design such as the SOMBRERO reactor, a carbon composite material is considered as the main component of the chamber. Fused silica, on the other hand, is one of the candidate materials for the final focus lenses. The critical issues to be addressed depend strongly on the material. In the case of fused silica it is important to understand the degradation of the optical properties due to irradiation. We have performed molecular dynamics simulations to calculate the number of defects produced as a function of recoil energy. Oxygen deficient centers and non-bridging oxygen centers are considered responsible for increased absorption and therefore obscuration. Our simulations show how these defects are produced, how annihilation of these defects can

POSTERS

occur and how a saturation of the number of defects produced is reached, in agreement with experimental observations. For the case of carbon composites, issues such as degradation of thermal conductivity, swelling and tritium retention are of importance. In order to gain some basic understanding of these problems we have performed atomistic simulations of damage in graphite, using the empirical potential developed by Brenner. We have performed calculations of the number and type of defects produced at energies up to several keV. Binding energies of hydrogen to different defect types (vacancy, di-vacancy, etc.) have been calculated, as well as their effect in thermal conductivity. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

Comparison Between the Microstructure of Austenitic Steel SS316L and Ferritic/Martensitic Steel F82H After Irradiation in a Proton-Neutron Mixed Spectrum: *Xuejun Jia*[1]; Yong Dai[1]; Robert Rutherford[3]; [1]Paul Scherrer Institut, ASQ, CH-5232 Villigen PSI 5232 Switzerland; [3]Los Alamos National Laboratory, APT/TPO, MS-H809, NM 87545 USA

Austenitic steel SS316L and ferrtic/martensic (F/M) steels are candidate materials for the containers of the liquid target of the Spallation Neutron Source and ADS facilities. Although there were numbers of studies on the investigation of the mechanical properties and microstructure of these steels irradiated with 800MeV protons in the last few years, but due to the lower irradiation temperature (<230∞C), the irradiation temperature dependence and the helium effects at relatively higher irradiation temperature were rarely reported. In STIP-I (SINQ irradiation program-I), TEM samples of SS316L steel and F/M steels such as F82H and T91 were placed at different positions and a series of irradiation temperatures (80-360∞C) and doses (2.8-12 dpa) were obtained. Present results of the TEM investigation on these steels show that the irradiation induced defects namely black dot damage and dislocation loops were all observed at all irradiation doses in the microstructure of these steels. The density of defect clusters is much lower and the mean size of defect clusters is much smaller in F/M steels than that of SS316 steel at the same irradiation condition. The density of defect clusters decrease and the mean size increase rapidly after the irradiation temperature higher than about 255∞C. High-density helium bubbles of size >=1 nm are all observed in F/M steels at irradiation temperatures above about 200∞C, but not in SS316L irradiated at same conditions.The amorphization of precipitates was observed in both materials at irradiation temperature below 230∞C.

Dislocation Dynamics Simulations of the Effect of Irradiation-Induced Pressurized Helium Bubbles on the Mechanical Properties of Metals: Peihua Jing[1]; *Tariq Khraishi*[1]; Brian Wirth[2]; [1]University of New Mexico, Mechl. Eng. Dept., Albuquerque, NM 87131 USA; [2]Lawrence Livermore National Laboratory, Matls. Sci. & Tech. Div., Livermore, CA 94551 USA

Pressurized helium bubbles develop in large number densities in some metals as a result of irradiation. It is observed experimentally that the presence of these bubbles alters the mechanical properties of the host metals. In this work, the results of computationally intensive dislocation dynamics simulations are presented to quantify the effect of the He bubbles on the mechanical properties. The effect of He bubbles, with a mean size between 1.5 nm to 2.5 nm and number densities of about 1023/m3, is investigated over a range of He pressures. The simulations show a clear correlation between the number density, and the bubble properties, on the mechanical properties of the metal. Comparisons to available analytical models and experimental data will also be presented.

Irradiation Hardening of Single Crystaline Ni: *Zhongwen Yao*[1]; *Robin Sch‰ublin*[1]; Maximo Victoria[1]; [1]Ecole Polytechnique FÈdÈrale de Lausanne, Fusion Tech.-Matls., Ctr. de Recherches en Physique des Plasmas, PSI, Villigen, AG 5232 Switzerland

Single crystal nickel was irradiated to various doses at two temperatures. The irradiation induced defect microstructure has been observed by transmission electron microscopy (TEM). The irradiated and unirradiated tensile samples have been deformed and relaxation test were performed at temperature between 77 K and room temperature. The shear stress-shear strain relations are determined and the dose and temperature dependences on the flow stress have been investigated. In order to understand irradiation hardening, the deformation microstructures corresponding to different deformation stages are observed by TEM and the deformation mechanisms are discussed.

The Change in Tensile Properties and Hardness of Wrought LCAC and ODS Molybdenum Irradiated with Fast Neutrons: *Brian V. Cockeram*[1]; Jim L. Hollenbeck[1]; Lance L. Snead[2]; [1]Bechtel-Bettis, PO Box 79, ZAP 08D/MT, W. Mifflin, PA 15122-0079 USA; [2]Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6140 USA

Molybdenum alloys are known to be susceptible to neutron radiation damage at radiation temperatures < 800C that has limited their broader use in nuclear applications. Manipulation of alloy microstructure through the use of oxide dispersion strengthening and optimization of carbon and oxygen contents are being investigated as means to mitigate the undesirable effects of irradiation. Low Carbon Arc Cast (LCAC) and Oxide Dispersion Strengthen (ODS) molybdenum sheet (0.51 mm to 0.76 mm thick) specimens were irradiated in the High Flux Isotope Reactor (HFIR) at temperatures ranging from 300C to 1200C and neutron fluence between 10.5 to 64.4 X 1020 n/cm2 (E > 0.1 MeV), and compared with unirradiated companion specimens. Irradiation of ODS and LCAC molybdenum at nominal temperatures of 1000C to 1200C produced only small changes in hardness (-10% to +10% change in values), tensile strength (0% to 30% increase in yield strength), tensile elongation (4% to 40% decrease in total elongation), with no change in the Ductile to Brittle Transition Temperature (DBTT) as inferred from tensile fracture surfaces. The post-irradiated DBTT for ODS and LCAC molybdenum irradiated nominally at 1000C were lower than any values reported in the literature for irradiated molybdenum. Irradiation of LCAC molybdenum at 300C and 600C resulted in a significant increase in hardness (51% to 113%), increase in yield strength (from 772-800 MPa to 1517-821 MPa), decrease in tensile elongation (from 9%-19% to 0%-0.8%), and increase in DBTT (from below room-temperature to 300C to 600C). This behavior is characteristic of that reported for pure molybdenum following irradiation at temperatures < 800?µC. However, the DBTT values for LCAC molybdenum following irradiation at 300C (DBTT = 600C) and 600C (DBTT = 300C) were in the low range of DBTT values reported in the literature for unalloyed molybdenum. Examination of fracture surfaces of LCAC molybdenum irradiated at temperatures < 600C and tested at room-temperature revealed failure initiation from microcracks with a transgranular fracture path. The use of arc-cast processing, a low oxygen content, and high carbon to oxygen ratio to produce the LCAC molybdenum used in this work results in strong grain boundaries that are not the preferred fracture path. The relatively low irradiated DBTT was determined to be the result of the transgranular cleavage failure mode.

Ab Initio Atomic-Scale Determination of the Point Defects Structure and Interactions in hcp-Zirconium: *Christophe Domain*[1]; Alexandre Legris[2]; [1]EDF, MMC, Site des Renardieres, R&D, Rte. de Sens-Ecuelle, Moret sur Loing 77818 France; [2]LMPGM, USTL, Bat C6, Villeneuve DíAscq 59655 France

The structure of point defects is one of the key parameters controlling the mass transport properties in crystalline materials. Their influence becomes crucial under irradiation since their steady-state concentration are order of magnitude higher than under thermal equilibrium. We have used state of the art ab initio atomic scale simulations to study the self interstitial (SIA) and vacancy structure in hcp-zirconium. Contrary to previous results obtained using semi-empirical potentials, our results indicate the octahedral position could be the most stable one for the SIA, very close in energy to the basal octahedral and the basal crowdion. Concerning the vacancy, we measured a small migration anisotropy, the activation energy for the jumps in the basal planes being slightly smaller than for the jumps with a component along the c direction. After a characterization of isolated point defects, we investigated their interaction, in particular the formation of small point defects clusters.

Defect Generation, Damage Evolution and Multiple Cascade Interactions in SiC: *Fei Gao*[1]; William J. Weber[1]; Xianglong Yuan[2]; Linn W. Hobbs[2]; [1]Pacific Northwest National Laboratory, Fundaml. Sci. Direct., PO Box 999, MSIN K8-93, Richland, WA 99352 USA; [2]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Cambridge, MA 02139-4307 USA

Molecular dynamics (MD) methods have been employed to study structural evolution during multiple cascade interactions. A large number of displacement cascades have been randomly generated in a model crystal to simulate multiple ion-solid interaction and defect accumulation. The cascade-enhanced coalescence of clusters or local disorder regions leads to the complete amorphization of SiC. The mechanisms and topological structures of amorphization and the changes in mechanical properties related to strength and material swelling are investigated. Furthermore, the MD method has been employed to study the recombination of Frenkel pairs generated by cascades at different temperatures. The lifetime and activation energy for close pair recovery have been determined. Long-range migration of point defects has been studied over a temperature range from 0.3 to 0.7 Tm (Tm is the corresponding melting temperature), and a wide set of diffusional characteristics, including the self-diffusion coefficient, jump frequency and activation energy, are obtained.

Effect of Solute Additions on Radiation-Induced Segregation and Microstructure of Model Austenitic Alloys: *Jeremy T. Busby*[1]; Mark C. Hash[1]; Gary S. Was[1]; Edward A. Kenik[2]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., Ann Arbor, MI 48109-2104 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4515, MS-6064, Oak Ridge, TN 37831-6064 USA

Radiation-induced segregation, microstructure and hardening may all contribute to IASCC, although all are highly sensitive to alloy composition. The objective of this work is to systematically examine the influence of nine solute additions on RIS, microstructure and hardening in an austenitic stainless steel. High-purity Fe-18Cr-12Ni was used as a base condition and doped with C, Mo, P, Si, Nb, Ti, Ni, or Ni and Cr. All nine austenitic alloys were irradiated with 3.2 MeV protons at 360∞C to 5.5 dpa. Molybdenum had little influence on RIS, loops, or hardness, relative to the base condition while all radiation-induced effects were reduced with P additions. The addition of Si resulted in an increase of Cr depletion, dislocation loop size and density, and hardening. Conversely, oversize solute additions (Nb and Ti) dramatically reduced all radiation-induced effects, consistent with literature results. Finally, the high Ni alloys exhibited increased Cr and Ni segregation relative to the base alloy, with little change to microstructure or hardening. Support was provided by Cooperative IASCC Research (CIR) program through EPRI contract EP-P3038/C1434. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

Microstructural Deformation Processes in Irradiated Austenitic Stainless Steels: *James I. Cole*[1]; Todd R. Allen[1]; Douglas L. Porter[1]; Robert S. Daum[2]; Hanchung Tsai[2]; Brian D. Wirth[3]; [1]Argonne National Laboratory, PO Box 2528, Idaho Falls, ID 83403 USA; [2]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA; [3]Lawrence Livermore National Laboratory, PO Box 808, Livermore, CA 94551-0808 USA

Development of irradiation-induced defect microstructures and the changes in deformation behavior caused by such microstructures are analyzed. Irradiated austenitic stainless steel hexagonal duct materials removed from EBR-II following reactor shutdown have been tensile tested at strain rates ranging from 10^{-3} to 10^{-7} s^{-1} and temperatures ranging from 280 to 500°C. Microstructural examination of samples prior to and following tensile tests was carried out. Changes in deformation behavior as a function of dose, strain rate and test temperature will be analyzed. The EBR-II hexagonal duct materials contain a large density of dislocations (loops and networks), voids and precipitates. The role each of the defect species plays in hardening and embrittlement will be discussed in terms of existing mechanistic models and comparisons will be made to the hardening behavior in unirradiated stainless steels.

On the Character of Self-Intersititial Dislocation Loops in Vanadium Alloys: *Luis Zepeda-Ruiz*[1]; Jaime Marian[1]; Brian D. Wirth[1]; [1]Lawrence Livermore National Laboratory, MSTD/CMS, PO Box 808, L-353, Livermore, CA 94551 USA

Atomistic simulation techniques of molecular dynamics and molecular statics are used in conjunction with a Finnis-Sinclair N-body potential of Vanadium to investigate the structure of stable self-interstitial dislocation loops. The results are compared to experimental observations and recent results in Ferritic alloys which detail the formation mechanism responsible for the nucleation and growth mechanism of <100> dislocation loops. Notably, dislocation loops of <100> Burgers vector are not observed in Vanadium and these results provide a basis for understanding the experimental observations.

Effects of Irradiation on the Microstructure and Mechanical Properties of Nanostructured Materials: *Nobuyasu Nita*[1]; Robin Schaeublin[1]; Max Victoria[1]; Ruslan Z. Valiev[2]; [1]EPFL, CRPP, ODGA/105, PSI, Villigen, AG 5232 Switzerland; [2]Institute of Physics of Advanced Materials, USATU, 12k Marks str., Ufa 450000 Russian Federation

Nanostructured materials should present a good resistance to irradiation because the large volume fraction of grain boundaries can be an important sink for radiation-induced defects. The objective of the present study is to experimentally investigate the irradiation impact on nanostructured materials. Nickel specimens were synthesized by electro deposition (ED) and High Pressure Torsion. Mean grain size of unirradiated specimen is about 100 nm for the HPT material and about 30 nm for the ED. 590 MeV Proton irradiation was conducted up to 0.55 dpa at room temperature. Yield strength estimated from Vickers hardness increased from 1131 to 2115 MPa after irradiation in Ni. This significant increase of hardening is due to the defect clusters induced by irradiation, only stacking fault tetrahedral have been observed in nickel. Further discussion will be made in terms of the nature and density of defects, relaxation of grain boundaries and impact of grain boundaries on local irradiation induced defect density.

Irradiation Induced Phase Transformation in NiTi Shape Memory Alloy: *Thomas Lagrange*[1]; Robin Sch‰ublin[1]; David S. Grummon[2]; Christian Abromeit[3]; Rolf Gotthardt[1]; [1]Swiss Federal Institute of Technology Lausanne, IPMC, Lausanne 1015 Switzerland; [2]Michigan State University, Lansing, MI 48824 USA; [3]Hahn Meitner Institute, Berlin Germany

In the present work, ion irradiation has been used as a processing technique for modifying the transformation behaviour of a NiTi shape memory alloy. The current study focuses on the relationship between the irradiation damaged microstructure and the modification of the phase transition. NiTi samples were irradiated at room temperature at 5 and 100 MeV with Ni and Au ions, respectively. Irradiation induced microstructure is investigated using transmission electron microscopy. At relatively low ion energy, the implanted region presents an amorphous layer containing precipitates with a B2 structure, which size decreases with increasing dose. The observed nanocrystals may result from increased point defect densities that lower transformation temperatures and stabilize the B2 phase in regions between the displacement cascades. At high energy amorphous tracks are observed. The relative contribution of the electronic and nuclear stopping is discussed together with the formation mechanisms of the irradiation induced phases.

Microstructure in Austenitic Stainless Steel SS316L and Ferritic/Martensitic Stainless Steels After Irradiation in SINQ Target-3: *Xuejun Jia*[1]; [1]Paul Scherrer Institut, ASQ, Villigen PSI, Brugg CH-5232 Switzerland

Austenitic stainless steel SS316L and ferrtic/martensic (F/M) steels are candidate materials for the containers of the liquid target of the Spallation Neutron Source and ADS facilities. In STIP-I (SINQ irradiation program-I), TEM samples of SS316L and F/M steels F82H and T91 were irradiated, a series of irradiation temperatures (70-370∞C) and doses (2-12 dpa) were obtained for the TEM investigation. Present results show that: Irradiation induced defects namely black dot damage and dislocation loops were all observed at all irradiation doses in the microstructure of these material, the density of defect clusters is much lower and the mean size of defect clusters is much smaller in F/M steels than that of SS316 steel at the same irradiation condition. The density of defect clusters decrease and the mean size increase rapidly after the irradiation temperature higher than about 255∞C. High-density helium bubbles of size ≥~1 nm are all observed at irradiation temperatures above about 200∞C in F/M steels and the existence of bubbles causes a significant irradiation hardening and embrittlement.

Effect of Proton and Ne Irradiation on the Microstructure of Zircaloy 4: Xiaotao Zu[1]; *Michael Atzmon*[2]; Lu-Min Wang[2]; L. P. You[2]; F. R. Wan[2]; Wenhui Jiang[2]; Gary S. Was[2]; Ronald B. Adamson[3]; [1]Sichuan University, Dept. of Physics, Chengdu 610064 China; [2]University of Michigan, NERS, 2933 Cooley, Ann Arbor, MI 48109 USA; [3]Zircology Plus, PO Box 2651, Fremont, CA 94536 USA

Zircaloys have been used extensively as cladding materials for nuclear fuel in light-water reactors, since they combine low neutron absorption with mechanical strength and corrosion resistance. The original alloys were developed mainly based on corrosion studies of unirradiated samples, and the use of irradiations in alloy development is a more recent development. Since reactor irradiations are costly and time consuming, the use of ion beams to introduce radiation damage is a promising approach. In the present study, we have used 2 MeV protons and 600 keV Ne ions to perform radiation-damage studies in Zircaloy 4 at elevated temperature. Transmission electron microscopy (TEM) and high resolution TEM were used to monitor precipitate amorphization and the buildup of dislocation loops. The behavior under Ne irradiation was monitored in situ. In addition, Vickers hardness measurements were conducted. These results will be presented and compared with literature data on neutron irradiation.

Index

A

B

C

D

E

F

G

H

I

J

K

L

M

N

O

P

Q

R

S

T

U

V

W

X

Y

Z