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

MONDAY'S 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-PDF

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:______________________

______________________ 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:

		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	$ ________
03-5522	Energy Efficient Manufacturing Processes (4th MPMD Global Symp.)	2	_______	_______	$65	$92	$ ________

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

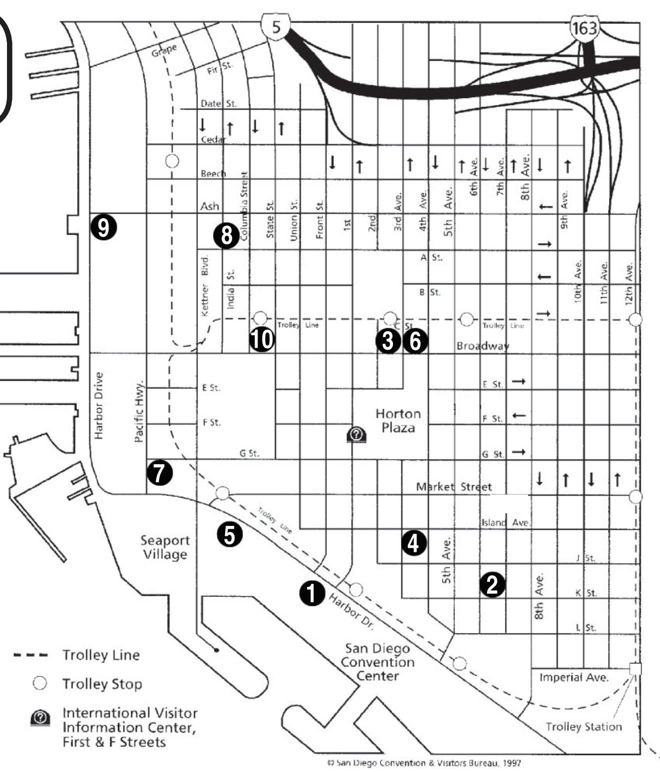
6. **U.S. Grant Hotel**
$179/single • $179/double

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 ______________________

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

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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

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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

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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 iRî 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 spacegroups. 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 non-crystalline 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, Nˆthnitzer 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 commerically 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 ($0.33 \leq x \leq 0.80$). 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 Assessement 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 (brightheners, 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 postgraduate 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 H^glund[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 (endpoint-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.

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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.

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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.

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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 pre-strained 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 pre-strain 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.

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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.

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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.

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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.

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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.

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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 a2g 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

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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 microhardness 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

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 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.

MONDAY PM

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

Monday PM — Room: 17B
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

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

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 $d(1/R_F)/dF-md(1/R_T)/dT + (1- m)(1/R_F)(1/R_T)= 0$ 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

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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.

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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.

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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.

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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.

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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

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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.

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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.

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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