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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

WEDNESDAY'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.

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

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 $ _____

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

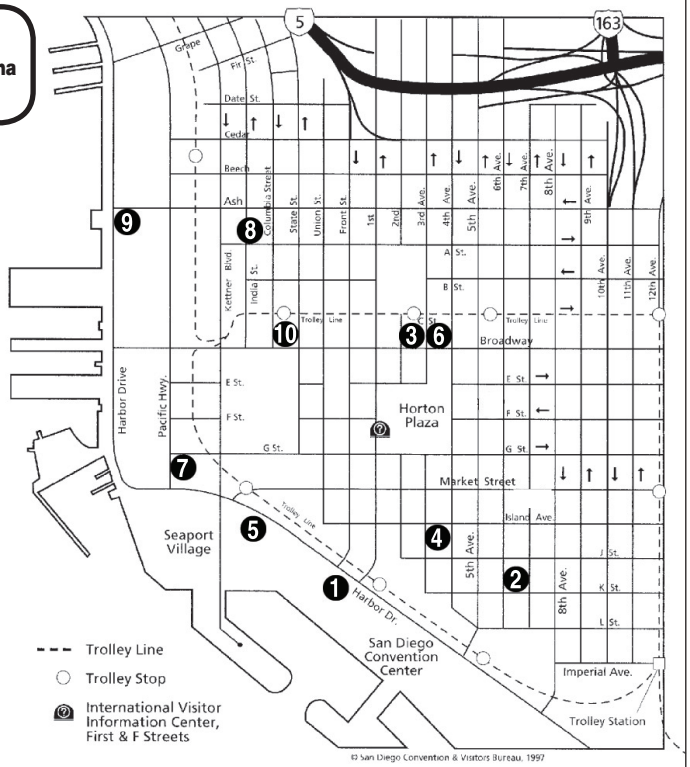
6. **U.S. Grant Hotel**
   $179/single • $179/double

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 _____

15th International Symposium on Experimental Methods for Microgravity Materials Science - I

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee
Program Organizers: Robert Schiffman, R. S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica & Instituto Nazionale di Fisica della Materia, Bologna 40127 Italy

Wednesday AM Room: 10
March 5, 2003 Location: San Diego Convention Center

Session Chair: C. Patuelli, Universita di Bologna, Dipto. di Fisica & Inst. Nazionale di Fisica della Materia, Bologna 40127 Italy

8:30 AM Introduction

8:45 AM
Precursor Phenomena in Melts Preceding the Solidification of In-50Sn and In-10Sn Alloys: *G. Costanza*[1]; F. Gauzzi[1]; R. Montanari[1]; [1]Universit‡ di Roma-Tor Vergata, Dipartimento di Ingegneria Meccanica, Via di Tor Vergata 110, Roma 00133 Italy

The solidification of two Indium-Tin alloys (in-50Sn and In-10Sn) has been investigated by X-ray diffractometry (XRD) in 1-g conditions. During cooling one of the alloys (In-10Sn) exhibits the eutectic transformation. The Sn content of other alloy (In-50Sn) is just lower than the range of peritectic transformation. The XRD spectra have been collected at decreasing temperatures for monitoring step by step the structural changes occurring in the liquid phase before the formation of the first solid and during the liquid-solid transformation till its completion. From the evolution of radial distribution function (RDF) it was possible to draw information about atom clustering in the melt preceding and accompanying the solidification of both the alloys and to evidence correlations between the structures of liquid and solid phases. To avoid convective motions in the liquid and to achieve the best experimental conditions, it is discussed the possibility to perform the same experiments under conditions of reduced gravity.

9:05 AM
Benard-Marangoni Interaction in Liquid Layers: *G. S.R. Sarma*[1]; [1]German Aerospace Center, Gottingen 37073 Germany

Convective instabilities induced by the presence of temperature and/or concentration gradients across liquid layers under the action of body forces due to Boussinesq, Coriolis, and Lorentz fields have been studied for a long time now but only a few of their results have been applied in a practical engineering sense. In the context of microgravity research the main focus has been on the Marangoni flows driven by surface tension gradients in view of their potential applications in crystal growth and other materials processing configurations where fluid-fluid interfaces occur. On the other hand, very few experimental studies have been even conceived to deal with the onset of convective instability in liquid layers under the simultaneous action of density Benard and surface tension (Marangoni) gradients across the layers. The former received by itself a lot of theoretical and experimental interest but the corresponding Marangoni problems definitely lagged behind due to inherent difficulties both in terrestrial and space environments. The pioneering Apollo-era experiments of Grodzka and Bannister from the 1970ís, despite their very preliminary data and inadequate characterization, are still the only ones that could be referred to during theoretical discussions of 1980ís. Basically, the problem here is one of interaction between two different instability mechanisms taken each by itself but which can act to oppose each other under certain circumstances and thereby stabilize the configuration. Although the underlying configuration is rather simple and can match fairly theoretical idealizations, there is, to date, no satisfactory ground based experimental data base to test even linear stability theories, leave alone non-linear aspects of pattern formation in such configurations. This situation offers potentially a plethora of possibilities for fluid physics studies within the microgravity program. The present contribution will attempt to describe such opportunities which seem to have been by-passed during the intervening years of active microgravity work. The starting point of the discussion will be motivated, for reasons of expediency, by the authorís own typical publications of the late 1980ís after which the topical area does not seem to have germinated any suitable experimental ideas. A review of the basic theoretical results available on the Benard-Marangoni interaction problem and a projection of possibilities for their eventual validation will be the main focus of the presentation.

9:25 AM
In Situ and Real Time Characterization of Solid-Liquid Interface Morphology: Directional Solidification in DECLIC Facility: *Nathalie Bergeon*[1]; HaÔk Jamgotchian[1]; Bernard Billia[1]; [1]L2MP, FacultÈ de St. JÈrÙme, Case 151, Av. Escadrille Normandie Niemen, Marseille, Cedex 20 13397 France

A directional solidification device dedicated to in situ and real time characterization of solid-liquid interface morphology on bulk samples of transparent materials is presented. This device, which will be implemented on ISS, is developed in the frame of the DECLIC project of the French Space Agency (CNES). The alloy is contained in a cylindrical crucible and observation is performed in two perpendicular directions: the growth one and the transverse one. In addition to direct observation by light transmission in those directions, an interferometer is set up in growth direction to provide the shape and the motion of the interface through an analysis of the interference fringes. Ground results on interface dynamics are shown: front recoil, triggering of instability, microstructure formation, that are all influenced by fluid flow. Microgravity experiments are planned to obtain critical benchmark data in diffusive environment and a better knowledge of convection influence.

9:45 AM
The Solidification Velocity of Undercooled Nickel and Titanium Alloys with Dilute Solute: *Paul R. Algoso*[1]; Alex S. Altgilbers[2]; William H. Hofmeister[1]; Robert J. Bayuzick[1]; [1]Vanderbilt University, Cheml. Eng., Box 1604, Sta. B, Nashville, TN 37235 USA; [2]Carrier Corporation, 284 Carrier Dr., Morrison, TN 37357 USA

The Ivantsov solution with marginal stability arguments (IMS model) is one of the most widely used models for relating solidification velocity to the undercooling of dilute metal alloys but fails to fit experimental results, particularly for alloy systems with a high equilibrium partition coefficient, k_E. The solidification velocity of undercooled binary alloys in electromagnetic levitation was measured utilizing high-speed thermal imaging techniques in order to reconcile differences between experimental results and theoretical predictions. Plateaus in the solidification velocity versus undercooling curve are observed. The first plateau at intermediate undercoolings is a result of solute addition, while the second plateau at high undercoolings is attributed to oxygen. IMS modeling is applied to the experimental data, and agreement is found when a value for k_E is used that is lower than the actual k_E of the alloy.

10:05 AM Break

10:25 AM
The Devitrification Process in ZBLAN Glasses: *Reginald William Smith*[1]; Ian Robert Dunkley[1]; [1]Queenís University, Matls. Sci. of Microgravity Applic., Nicol Hall, Rm. 228, Kingston, Ontario K7L 3N6 Canada

It is known that a reduced gravity environment will help reduce crystallisation when processing ZBLAN glasses, but the reason for this is still a matter of speculation. This talk will describe recent work at Queenís University concerned with the containerless processing and characterisation of ZBLAN glasses in preparation for reduced gravity studies of the crystallisation process in these glasses.

10:45 AM
Faceted Growth and Kinetics of Doped Germanium Single Crystals: Andrew David Deal[1]; Ercan Balikci[1]; *Reza Abbaschian*[1]; [1]University of Florida, Matls. Sci. & Eng., 100 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA

Semiconductor materials exhibit faceted growth from a melt, a phenomenon characterized by the lateral motion of elementary steps along an atomically smooth solid/liquid (s/l) interface. Understanding the fundamentals of this growth mechanism and the incorporation of the dopant into the solid is necessary to improve the quality and reproducibility of semiconductor devices fabricated within the electronic industry. A novel growth technique called Axial Heat Processing (AHP), which enables close control of geometrical and thermal boundary conditions near the s/l interface, was used to study the effects of several kinetic parameters on the morphological stability of a faceted s/l interface in doped Ge. AHP utilizes a submerged heater to

impose a planar thermal isotherms near the interface. The effects of the processing variable such as growth rate and melt height below the submerged heater on the radial and axial distribution of the solute will be presented.

11:05 AM
Wetting and Nucleation Behavior of Binary Immiscible Alloys: *L. J. Little*[1]; J. B. Andrews[1]; [1]University of Alabama-Birmingham, Matls. Sci. & Eng., 1150 10th Ave. S., BEC 254, Birmingham, AL 35294 USA

There are many potential applications for binary immiscible alloys if they can be produced to form composite-like structures (i.e. an aligned, fibrous structure or a solid dispersion with one phase uniformly distributed within the other). During processing of these alloys on Earth, the density difference between the two phases leads to massive segregation of the phases due to gravity-driven sedimentation. Processing the alloys in a microgravity environment should alleviate this problem. However, early experiments that were performed in microgravity conditions still led to massive segregation of the liquid phases. It is the purpose of this investigation to determine the cause of microgravity segregation in binary immiscible alloys. The Wetting Characteristics of Immiscibles (WCI) project was performed in November of 1997 as part of the USMP-4 shuttle mission. It was the objective of this project to determine if droplet migration and perfect wetting of the minor liquid phase were the major factors leading to segregation in these alloys under microgravity conditions. The immiscible transparent metal analog system succinonitrile-glycerol (SCN-GLY) was utilized in the investigation, and an estimated perfect wetting range of the system was identified. Currently, ground-based work is underway based on the results of the WCI shuttle mission. The investigation continues in determining factors that lead to massive segregation in binary immiscible alloys by focusing on the influence of perfect wetting on preferential nucleation of the minor immiscible liquid phase.

Actinide Materials: Processing, Characterization, and Behavior: Advanced Fuels and Materials I

Sponsored by: Light Metals Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS), LMD-Reactive Metals Committee
Program Organizers: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA; Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Wednesday AM Room: 4
March 5, 2003 Location: San Diego Convention Center

Session Chair: Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

8:30 AM
Prospects for Nuclear PowerñA System View: *Victor H. Reis*[1]; [1]SAIC

The end of the Cold War, increased international concern for climate change, electric restructuring in the United States have all raised hopes that there might be a resurgence of nuclear power during the first half of the 21st Century. For this to occcur it will require a combination of political, economic and technological factors, both domestically and internationally. Some of these factors will be discussed, and the results of some collaborative models will be described, in particular the need for closing the nuclear fuel cycle. The results indicate that significant, sustained government leadership will be required for nuclear power to be a major contributor to the global and domestic energy supply of the future.

9:15 AM
Cermet Nuclear Fuel Fabrication by Powder Metallurgy Methods: *Sean M. McDeavitt*[1]; M. C. Hash[1]; A. S. Hebden[1]; J. M. Runge[1]; C. T. Snyder[1]; A. A. Solomon[2]; [1]Argonne National Laboratory, Chem. Tech. Div., 9700 S. Cass Ave., Bldg. 205, Argonne, IL 60439-4837 USA; [2]Purdue University, Sch. of Nucl. Eng., W. Lafayette, IN 47907 USA

Cermet nuclear fuels enhance performance and safety through their high matrix thermal conductivity and low internal temperatures. The powder-in-tube drawing method is a simple low-temperature process for cermet fabrication that has advantages over hot methods, especially for fuel systems that require remote hot cell fabrication. Lab-scale equipment has been installed to develop this process for large-scale (~1.2 m length) and small-scale (~0.05 m length) samples. This method is being applied to zirconium matrix cermet fuels containing spray-dried (Th,U)O2 and resin-loaded (Pu,Am,Np)O2 microspheres. These actinide-bearing cermet fuels are being developed for application in advanced nuclear energy systems and actinide transmuter systems, respectively. Oxide microspheres (50 to 1000 mm diameter) and zirconium metal powders (~44 mm nominal diameter) are dry-mixed and loaded into alloy drawing tubes and vibratory-packed to obtain a green packing fraction of ~50%. The powder-containing tube is drawn to reduce the pinís outer diameter and compact the powders within. The internal microstructure is stabilized through a post-drawing anneal between 500∞C and 1000∞C to remove strain hardening and facilitate sintering and interfacial bonding. Multiple cycles are used with sequentially decreasing die sizes to achieve the desired level of densification.

9:40 AM
Investigations of Mononitride Actinide Ceramics for Transmutation Fuels: *Robert W. Margevicius*[1]; *Kenneth J. McClellan*[1]; [1]Los Alamos National Laboratory, Nucl. Matls. Tech., MS E505, Los Alamos, NM 87545 USA

Transmutation fuels work at Los Alamos National Laboratory is currently focused on mononitride ceramic fuel forms that have the advantages of high melting points, good thermal conductivity, and high chemical and thermal stability. The actinide fuels effort at Los Alamos emphasizes the synthesis and fabrication of actinide-bearing nitride fuel pellets. These pellets will be inserted into the Advanced Test Reactor early in 2003 and continue for the next couple of years. The nitride pellets are designed to contain varying amounts of Pu, Am, and Np. The composition ranges of the pellets go from having low minor actinide content (i.e., just PuN) to ones that have significant amounts of AmN (80%). Results presented will outline fabrication techniques which are designed to reduce the volatility of Am. Results from characterization of the powders and pellets will also be given.

10:05 AM
Atomistic Modeling of the Phase Stability in the Am System: *S. G. Srinivasan*[1]; M. I. Baskes[1]; M. Stan[1]; A. M. Niklasson[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

Actinide based ceramics have evoked renewed interest as potential next generation fuels for nuclear reactors. A reliable, many-body atomistic model of Am is necessary for its eventual application to the Am-N and Am-C systems. We describe the development of a semi-empirical model for Am based on the modified embedded atom method. Properties derived from both experimental and full-potential Linear Muffin-Tin Orbital (FPLMTO) method calculations, based on the local spin density and generalized gradient approximations, are used to develop our potential. Using this potential we investigate the phase stability, defect energetics, thermodynamic, and mechanical properties of Am using a combination of molecular dynamics and thermochemical calculations. Our results will be compared and contrasted with the existing experimental data. Lastly, we will also attempt to predict properties that have not been experimentally measured.

10:30 AM Break

10:40 AM
Phase Development in Pu-Zr Alloys with Np and Am Additions: *Dennis D. Keiser*[1]; J. Rory Kennedy[1]; Mitchell K. Meyer[1]; [1]Argonne National Laboratory, Eng. Tech. Div., PO Box 2528, Idaho Falls, ID 83403-2528 USA

Metallic alloys are being investigated as possible nuclear reactor fuels for transmuting long-lived fission products, like Np and Am, into short-lived fission products. The alloys that have been looked at are based on the Pu-Zr system with additions of up to 10 wt% Np and 12 wt% Am. Alloys have been cast using arc-melting techniques and then have been characterized using a scanning electron microscope equipped with energy-dispersive and wavelength-dispersive spectrometers (SEM/EDS/WDS). Additionally, X-ray diffraction analysis was performed to determine developed phases. Observed microstructures for the various as-cast alloys will be discussed along with the identified phases. Comparisons will be made with available phase diagrams.

11:05 AM
Thermal Characteristics and Behavior of Pu-Np-Am-Zr Bearing Alloys: *J. Rory Kennedy*[1]; [1]Argonne National Laboratory, Eng. Tech., PO Box 2528, Idaho Falls, ID 83403 USA

Recent efforts at the Argonne National Laboratory-West have included the fabrication of non-fertile inert matrix metallic fuels for

study as part of the DOE) Advanced Accelerator Applications (AAA) actinide transmutation program. Specifically, the binary Pu-40Zr and Pu-60Zr alloys, the ternary Pu-10Np-40Zr and Pu-12Am-40Zr alloys, and the quaternary Pu-10Np-10Am-40Zr alloy have been prepared via an arc-casting method. Of particular interest in the application of these fuels are the volatility of Am and the possible low melting behavior of Np containing phases. These have been addressed with thermal analysis studies including thermogravimetric analysis (TGA) and differential thermal analysis (DTA). Additional studies on fuel-cladding-chemical-interactions (FCCI) and microstructure and phase determination on annealed samples have been performed. The thermal behavior and characteristics of the alloyed materials will be presented and discussed in terms of fuel performance requirements.

11:30 AM
Wet/Dry Processing of Urania/Thoria Powders to Produce Dense Pellets: *Alvin Solomon*[1]; V. Chandramouli[1]; S. Anthonysamy[1]; S. Kuchibhotla[1]; [1]Purdue University, Sch. of Nucl. Eng., W. Lafayette, IN 47907 USA

Urania/thoria powder mixtures were produced by an optimized reverse-strike co-precipitation process to yield very fine powders of high surface area. Dry compaction with organic binders and sintering under controlled atmospheres at >1700C for 10 hours yielded a relative density of only 90%, due to agglomeration of the powder. XRD analysis showed that complete solid solutions were obtained. However, wet milling of a high zeta potential slurry with organic binders effectively broke up the agglomerates and dispersed the organic so as to produce high green and sintered densities above 97% under the same sintering conditions. The resintering behavior and thermal properties of these pellets are also reported.

11:55 AM
A Dynamic Model Describing the Morphological Changes of Powder in a Rotary Kiln: StÈphane HÈbrard[1]; Carine Ablitzer[1]; *Fabrice Patisson*[2]; Denis Ablitzer[2]; [1]CEA Cadarache, DEC/SPUA/LCU, Saint-Paul-lez-Durance 13108 France; [2]Ecole des Mines, LSG2M, UMR 7584 CNRS-INPL, Parc de Saurupt, Nancy 54042 France

A stage of the dry conversion process for producing nuclear fuel consists in defluorinating and reducing UO_2F_2 powder into UO_2 in a rotary kiln. We have undertaken mathematical modeling work that ultimately aims at predicting the morphological characteristics of the powder, which are crucial for the product quality, as a function of the kiln operating parameters. First, we have developed a new dynamic model of the solid flow in a rotary tube equipped with internal lifters. It calculates the flow-rate and hold-up of the powder in both the dense and the airborne phases. Then, a morphological change model, based on population balances of fractal agglomerates and using the results of the dynamic model, has been written to describe the agglomeration (by Brownian motion, by differential sedimentation, and shear-induced) and sintering phenomena. Results of the dynamic model, as well as the first findings concerning the morphological changes, are given and discussed.

Aluminum Reduction Technology: Fundamentals

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

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

Session Chair: Michel Reverdy, Aluminium Pechiney, Paris, Cedex 16 75218 France

8:30 AM
In-Situ Observations of Frozen Electrolyte in Laboratory Reduction Cells: *Craig W. Brown*[1]; [1]Northwest Aluminum Technologies, 1080 W. Ewing Pl., Ste. 202, Seattle, WA 98119 USA

Processes in molten salts, such as those associated with aluminum electrolysis, are difficult to study in-situ. This is because the environment is too harsh for most materials to withstand, so applicable techniques are limited. This paper presents a technique that extends the experimental options. Freezing the salts during electrolysis produces samples that can be studied by various means, including scanning electron microscopy. Examples presented were obtained in studies of low-temperature electrolysis with slurry electrolytes. Elemental maps generated from electron microprobe data obtained with such samples display evidence of mass transport phenomena near the electrodes.

8:55 AM
Error Analysis in the Measurement of Current Efficiency in Hall-Heroult Cells: *Guy Lawrence Fredrickson*[1]; [1]Hazen Research, Inc., 4601 Indiana St., Golden, CO 80401 USA

Upon cursory consideration, the determination of current efficiency (CE) for a Hall-HÈroult cell appears to be a deceptively simple task. After all, what else is required but the measurement of metal production as a function of the cumulative ampere-hours of current flow. However, when attempts are made to determine the CE of a single Hall-HÈroult cell, over a relatively short elapsed time (i.e., less than 48 hours), the data, more often than not, shows little accuracy, precision, or reproducibility. This paper discusses the statistical error, which is associated with the determination of CE, as a function of the various field and analytical measurements that are required for its calculation. A general statistical model is developed that can be applied to a number of different measurement techniques and strategies, and to any type and size Hall-HÈroult cell.

9:20 AM
Characterization of the Fluctuation in Anode Current Density and ìBubble Eventsî in Industrial Reduction Cells: Henrik Gudbrandsen[1]; *Nolan Richards*[2]; Sverre Rolseth[1]; Jomar Thonstad[3]; [1]SINTEF, Trondheim N-7491 Norway; [2]Consultant, 117 Kingswood Dr., Florence, AL 35630 USA; [3]Norwegian University of Science and Technology, Matls. Tech. & Electrochem., Sem Selandsv. 6, Trondheim N-7491 Norway

Whether the studies of CO_2 bubbles evolved from a carbon anode in an alumina reduction cell represents a less well understood aspect of the electrolytic process or a compelling aspect of the anode effect, the subject has attracted increased attention in recent years. In this work the character and impact of ìbubble eventsî were quantified for anodes of industrial cells. The data about the impact and frequency of the bubbles was derived from in situ measurements of the anode overpotential using a fast data logger. Oscillations in overvoltage were correlated with the dynamics of anodic current density and hence, variations in the screening and bubble coverage of the horizontal surfaces of the anodes.

9:45 AM
Measurements of the Anode Overvoltage in Industrial Alumina Reduction Cells: Henrik Gudbrandsen[1]; *Nolan Richards*[2]; Sverre Rolseth[1]; Jomar Thonstad[3]; [1]SINTEF, Trondheim N-7491 Norway; [2]Consultant, 117 Kingswood Dr., Florence, AL 35630 USA; [3]Norwegian University of Science and Technology, Matls. Tech & Electrochem., Trondheim N-7491 Norway

The anode overvoltage, η, in any industrial alumina reduction cell represents 10-15% of the total cell voltage. These laboratory results have been the basis for calculating the η in the mathematical modeling of total voltage for designing and setting the parameters for operating cells. An empirical allowance has often been added for the ìbubble resistanceî. In this presentation the anodic overvoltages were measured on both prebaked anodes with and without slots, as well as Soderberg anode in industrial cells using a robust, stable reference electrode that eliminated the ohmic drop across any bath layer. The ηís were determined over a range of current densities and alumina concentrations on anodes of different geometries and ranged 0.6 to 1.0 volt depending upon alumina concentration and extent of bubble coverage. Overvoltages were followed to anode effect. The actual values reported are higher than would be calculated from currently accepted mathematical models. Some attributions for these differences are presented.

10:10 AM Break

10:20 AM
Addition of Spent Potlining to Aluminum Reduction Cells to Produce High Si Alloys: *Bj□rn Moxnes*[1]; HÂvard Gikling[1]; Halvard Kvande[2]; Sverre Rolseth[3]; [1]Hydro Aluminium, Tech. Ctr. ≈rdal, ÿvre ≈rdal NO-6885 Norway; [2]Hydro Aluminium, Metal Products, Oslo NO-0246 Norway; [3]SINTEF Materials Technology, Electrochem. & Ceram., Trondheim NO-7465 Norway

Tests with crushed potlining as feed material have been performed on laboratory cells and in industrial scale in S□derberg cells. The purpose was twofold: To produce Si-rich alloys directly in the cells and to utilize spent potlining that otherwise has to be deposited as hazardous waste. The spent potlining also contains bath material and alumina that could be recovered. Carbon was separated from the collected spent potlining materiel before it was crushed to a powder that could be handled like normal alumina. Results from the laboratory tests showed

that it took twice as long to dissolve the spent potlining in the bath compared to primary and secondary alumina. The S□derberg test cell was operated continuously for five month with a feed consisting of a mixture of alumina and spent potlining. The operational results were acceptable, the level of Si content in the aluminum exceeded 10%.

10:45 AM

An Improved Method for Removing Light Metals from Molten Aluminum in a Reduction Plant: *Nolan Richards*[1]; Helge O. Forberg[2]; [1]Consultant, 117 Kingswood Dr., Florence, AL 35630 USA; [2]Consultant, 5118 New Bedord Place, Marietta, GA 30068 USA

Both modern magnetically compensated and modernized cells using low ratio bath with quiet metal pads can produce aluminum with sodium content higher than preferred. Older potlines using LiF-modified bath as a means to enhance profitability, produce virgin Al with 10-20 PPM Li in addition to some Na. To improve both quality and efficiency in processing Al to obtain premium grades or meet specifications for certain products, the light metals should be removed before the tapping crucibles are loaded into holding furnaces. Technologies ranging from a bed of [charcoal + AlF_3] to treatment-in-crucible (TAC) with powdered AlF_3 added to a vortex for dispersion are being used to lower the concentration of light metals. Here, an improved TAC method is described based upon the efficient dispersion of powdered $NaAlF_4$ introduced on a carrier gas stream down a hollow impeller, which further disperses the fluoride, which, because of its higher vapor pressure, vaporizes and improves the contact with molten Al. The system includes other advantageous features. This process has the expectation for removing light metals more efficiently and will reduce concentrations of Na and Li to 1 PPM within 10-12 minutes.

11:10 AM

Anode Reaction in Aluminium Electrolysis Prior to and During Anode Effect: *Hongmin Zhu*[1]; Jomar Thonstad[2]; [1]University of Science & Technology Beijing, Dept. of Physl. Chem., 30 Xueyuan Rd., Beijing 100083 China; [2]Norwegian University of Science and Technology, Matls. Tech. & Electrochem., Sem Selandsv. 6, Trondheim N-7491 Norway

Anode reactions in aluminium electrolysis prior to and during anode effect were studied by cyclic voltammetry and gas chromatography. Three voltammetric peaks at around 2.2 V, 2.6 V and 4.3 V were observed in low-alumina melts. The currents of the first two peaks increased sharply by the addition of alumina. In a highly acidic melt, a sharp rise in current was observed above 5.8V. CO was the dominant oxygen-containing gas in low-alumina melts. CO2 was only found at low potentials (< 4V). CF4 generation was detected at potentials above 2.8 V. The rate of CF4 generation was similar in the various melts, and it was in all cases low. It has been shown elsewhere that alumina depletion precedes a potential shift in the positive direction, to reach the potential region for generation of CF4. This is accompanied by a sudden drop in current, marking the onset of the anode effect.

11:35 AM

Technique and Mechanism of Aluminum Floating Electrolysis in Molten Heavy Na3AlF6-AlF3-BaF2-CaF2 Bath System: *Lu Huimin*[1]; *Yu Lanlan*[1]; [1]University of Science and Technology Beijing, Metallurgl. Eng. Sch., 30 Xueyuanlu Haidian, Beijing 100083 China

In this paper, the authors studied a new low temperature aluminum electrolysis process in 3000A experimental cell with Na3AlF6-AlF3-BaF2-CaF2 bath system. The influences of cathodic current density, electrolytic temperature and density differences between bath and liquid aluminum on current efficiency (CE) were studied; when electrolytic cryolite ratio was 1, w(BaF2) and w(CaF2) were 20mass% and 15mass%, respectively, CE reached 90.57% and specific energy consumption was 12.27kWh/kg Al. Because aluminum obtained floated on the surface of molten electrolyte, named this electrolysis method as low temperature aluminum floating electrolysis. The mechanism of aluminum floating electrolysis was also studied at 1023K and Al2O3 concentration of 5.8◊10-4mol/cm3 by use of potential sweep voltammetry at tungsten electrode. It was found that the reduction of aluminum was a simple and rapid process involving the exchange of three electrons: Al3+ + 3e = Al and the diffusion coefficient of aluminum ions was 1.893◊10-5cm2/s.

Aluminum Reduction Technology: Modernization

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

Wednesday AM Room: 6B
March 5, 2003 Location: San Diego Convention Center

Session Chair: Jean Paul Aussel, Pechiney Aluminium Smelter, Voreppe Cedex 38341 France

8:30 AM

Research on the Instabilities in the Aluminum Electrolysis Cell: *Panaitescu Aureliu*[1]; Moraru Augustin[1]; *Panaitescu Ileana*[1]; [1]University ìPolitehnicaî of Bucharest, Electl. Eng., Spl. Independentei 313, Bucharest 77206 Romania

In order to reduce the specific electrical energy consumption of the aluminum electrolysis cells their instabilities were experimentally studied. The study was focused on the instabilities produced during the cellsí operation by reducing the anode-to-cathode distance below a certain value and on the instabilities due to the anode effects. The researches were performed in the electrolysis halls of the ALRO Slatina plant in Romania, on standard cells which operate without a vertical magnetic filed compensation and also on new designed cells with a global compensation of Lorentz forces. The method of study consist in the real time visualization of the molten metal surface shape. The experimental research didnít evidence the existence of traveling waves on the aluminum surface but only vertical local oscillations (Pinch effects). The MHD phenomena are due to the asymmetric distribution of Lorentz forces in the molten media. For the electrolysis cells designated in order to obtain the global compensation of the Lorentz forces the vertical oscillations of the molten metal surface are strongly reduced. These cells can operate with small anode to cathode distance and therefore average specific consumption of 12800 kWh/t were obtained. The anode effects are considered in this paper as instabilities in the cell operation. Anode effects appear in the area where the bath circulation velocity is small. In these places alumina concentration is low, and are small also the electrical conductivity of the bath and the anode current density. The results of our researches have also shown that the device for the visualization in real time of the current distribution in the electrolysis cells allows the identification of the most proper area for the alumina feeding.

8:55 AM

Process Modelling as a Key Factor in AP14 Retrofit at Aluminij d.d. Mostar: *Christian Droste*[1]; Ingo Eick[1]; [1]Hydro Aluminium Primary Metals, Process Modlg., Georg-von-Boeselager-Str. 25, 53117 Bonn Germany

In October 2001, Aluminij d. d. Mostar began a major upgrade of its 92,000-tpy smelter in Bosnia-Herzegovina. By introducing the latest potroom technology, the smelter became internationally competitive with respect to production costs, working conditions and environmental protection. The main part of the modernisation project consisted of converting 256 side-worked, prebake AP14 cells, originally built in 1981, to centre-worked, point-fed cells and installing a modern pot control system and a pneumatic alumina transport system. The hot change of the cells was completed in May 2002. The financial success of the project is closely related to a substantial increase in metal production after modernisation. Before embarking on the modernisation project, the expected production gains from converting the cells, increasing the amperage and improving current efficiency had to be forecast reliably. The limitations and bottlenecks of the existing technology were therefore evaluated carefully with the help of well-established process modelling tools. The results showed that, in addition to the conversion from side to centre operation, some modifications to the busbar system and pot lining were also necessary to meet the ambitious target of a 25% increase in metal yield.

9:20 AM

The 3D Modeling of MHD-Stability of Aluminum Reduction Cells: *S. A. Shcherbinin*[1]; A. V. Rozin[2]; S. Yu Lukashchuk[3]; [1]PSC Bratsk Aluminum Plant, Bratsk 665716 Russia; [2]Moscow State University, Inst. of Mech., 1 Michurinskii Pr., Moscow 119899 Russia; [3]Ufa Air Technological University, 12 K. Marx St. 450025 Russia

The wide range and complicated character of technical problems, facing ìRussian Aluminumî Company from the date of its foundation

in 2000, formed the background of intensive development of the important sector of mathematical simulation. In particular, the simulation of magneto-hydrodynamic processes followed several directions. One of researches in the area of MHD-stability was initiated by ìRussian Aluminumî Bratsk Aluminum Plant. The aforementioned MHD-model allowed to estimate 3D non-stationary hydrodynamic processes in metal and bath, including the cell wall/anode region. Two-parameter turbulent model was used. The electromagnetic fields, gravity and surface tension force were taken into account. The model response to external impacts (such as current jumps during anode effects) were used for estimation of MHD-stability of aluminum reduction cell. Different variations of the cell configuration, bath properties, busbar arrangements were analyzed in order to study the dynamical behavior of MHD-parameters.

9:45 AM

Twenty Years of Progress at HINDALCOís Aluminium Smelter: *S. C. Tandon*[1]; R. N. Prasad[1]; [1]Hindalco Industries, Ltd., Renukoot-231217, Sonebhadra 231217 India

Hindalcoís aluminium smelter was initially started in 1962 with 20,000 tpy capacity and technology was based on 1950ís generation pots incrementally from 20,000 tpy to 120,000 tpy by early 80ís by adding number of potlines but the basic design of pots remained unchanged. In view of the energy price hike and to keep pace with developments in aluminium smelting, Hindalco undertook serious efforts to modernise its smelting facilities in mid 80ís by retrofitting new technologies and since then continuing improvement has been the normal mode of operation in the smelter. Now Hindalco smelting capacity stands at 275,000 tpy and two potlines are under construction to complete brownfield expansion to achieve 350,000 tpy by end of 2003. Today plant is competitive in the world market being one of the lowest cost producer of primary metal, operating at 94.5% current efficiency and 14.2 DC KWH/Kg specific energy consumption with pot life of 2800 days. This paper traces the evolution of the retrofit technology that has been applied to smelter which includes revised cathode and insulation design, mechanised aluminium feeding and conveying, computerised potline control, bath chemistry, anode improvements, environment protection and waste utilisation.

10:10 AM Break

10:20 AM

Conversion of the AP 14 Potline at Aluminij d.d. Mostar from Side to Point Feeding: *Volker von der Ohe*[1]; Bruno Cale[2]; Zdravko Cuturic[3]; [1]Hydro Aluminium Primary Metals, Eng., Koblenzer Str. 122, Neuss 41468 Germany; [2]Aluminij d.d. Mostar, Dir. Technique, ul 25 Studeni bb, Mostar Bosnia Hercegowina; [3]Aluminij d.d. Mostar, Mgr. Potroom, ul 25 Studeni bb, Mostar Bosnia Hercegowina

Without the loss of production, the 256 side-worked AP 14 cells at Aluminij d.d. Mostarís aluminium smelter were converted to centre-worked point-feeder cells in a hot-change operation lasting only 9 months. 2 cells were converted every day between September 2001 and May 2002. Each cell was equipped with three point-feeders, two crust breakers, an ELAS process controller and network to a central control room for observation and adjustment of the pot parameters. An alumina transport system was installed and the gas cleaning system upgraded to meet international environmental protection standards and working conditions. Auxiliary plants, such as the anode rodding shop and compressor station and operating procedures at the smelter, e.g. cavity cleaning and anode covering, were also adapted to fit the new technology. To ensure optimum performance, it was essential to fine-tune pot operation and pot tending to the upgraded technology and give the Aluminij workers intensive training. The main technological challenge of the project was to replace each pot superstructure in the potroom within a few hours. This involved widening the anode centre channel and separating the anodes in the hot bath to allow the installation of point-feeders while keeping the power cut-off time of the cell as short as possible. Subsequent production results show that such a pot conversion can be controlled technically and technologically and that a project of this kind can be carried out cost effectively.

10:45 AM

The Development of GAMIís Large Aluminum Reduction Cell: *Yang Yi*[1]; Yao Shihuan[1]; [1]Guiyang Aluminum and Magnesium Design and Research Institute (GAMI), Guiyang 550004 China

The stability of an Aluminum reduction cell is the main factor affect the cellís current efficiency. We introduce the method of increase the stability of cells which used in GAMIís 230KA, 280KA, 320KA and 350KA pre-bake side-by-side cellís design, for example, optimizing the bus-bar configuration, modifying the cellís length-width ratio, increasing the size of anode and cathode, i.e., as well as the model and real value of velocity and magnetic fields of cell, are presented in the paper.

11:10 AM

Development of 230KA Prebake Pots: *Leng Zhengxu*[1]; Li Hongpeng[1]; Li Yun[1]; Yang Caohong[2]; Yang Yi[2]; Yao Shihuan[2]; [1]Guizhou Aluminum Smelter, Guiyang 550014 China; [2]Guiyang Aluminum and Magnesium Design and Research Institute, Guiyang 55004 China

In this paper, we introduce 230KA experimental pre-bake cell in Guizhou Aluminium Plant. We describe the structure and magnetic of the cell and the new lining material. Via the two years practiced, it is proved the stability of the cell. The cellís current efficiency reached 94%, DC cost less 13400Kw/t-Al, AE<0.1. The new type cell will be used in the new project of Guizhou Aluminum Smelter.

Carbon Technology - III

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Amir A. Mirchi, Alcan Inc., Arvida Research and Development Centre, Jonquiere, QC G7S 4K8 Canada; Don T. Walton, Alcoa Inc., Wenatchee Works, Malaga, WA 98828-9784 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday AM Room: 6D
March 5, 2003 Location: San Diego Convention Center

Session Chair: Christian Dreyer, Aluminium Pechiney, St. Jean De Maurienne 73303 France

8:30 AM

Exhaustion, Pneumatic Conveyor and Storage of Carbonaceous Waste Materials: *Paulo Douglas Santos Vasconcelos*[1]; [1]Albras AlumÌnio Brasileiro SA, Carbon Plant, Rod. PA 483, km 2, Vl de Murucupi., Barcarena, Par· 68447000 Brazil

In smelters that use prebaked anodes, operations such as butt cleaning, butt and anode reject crushing and grinding, handling of coke packing material in the bake furnace, floor sweeping and discharge of dust from bake furnace cranes causes significant problems with the high generation of carbon dust and, consequent environmental pollution. To control the dust pollution is a very difficult task. Another problem, meanwhile, is how to convey and store the dust collected. This paper presents the problem existing in the carbon plant of Albr·s, describing the pneumatic conveying system developed by the Carbon Plant Engineering department, and the storage of the dust collected in the carbon plant. Examples of application of the system to use the carbon dust to sale to the cement industry, or to generate water vapor for the carbon plant.

8:55 AM

The Impact of the Firing and Control System on the Efficiency of the Baking Process: Jaffar Ameeri[1]; *Khalil M. Khaji*[1]; *Wolfgang K. Leisenberg*[2]; [1]Aluminium Bahrain, Carbon & Metal Services, Manama Bahrain; [2]Innovatherm, Carbon Div., Leisenberg GmbH + Co., Roter Lohweg 22, Butzbach, Hessen D-35510 Germany

Based on the recent conversion of two existing anode baking furnaces at ALBA, Bahrain, the paper will demonstrate the improvements that can be achieved by using new features of firing and control system for baking furnaces. The evaluation covers the most relevant parameters of the baking process such as quality, consistency, fuel efficiency and the combustion of volatiles. In addition to the advanced built-in features of the new system, which already offered a distinct improvement over the existing one, fine tuning of the system by the carbon plant management in close co-operation with the system designer enabled the team to adapt the advanced features of the new system to the actual furnace performance, in order to unleash the full potential of the system. New features, such as prediction of anode temperatures and the modification of the firing strategy have been tested.

9:20 AM

Performance Enhancement of Carbon Anode Baking Plant at Hindalco, India: *Subhash Chandra Tandon*[1]; [1]Hindalco Industries Ltd., Reduction Plant, PO Renukoot, UP 231217 India

Hindalco is Indiaís largest integrated aluminium producer and amongst the worldís lowest cost producers. It is a flagship company of Aditya Birla Group, Indiaís second largest business house, committed to Global benchmarking. The company is known for its culture of innovation, experimentation and creativity. Under the same spirit, performance enhancement plan of Anode Bake Furnaces was under-

taken to optimise fuel oil consumption, improved fire cycle time, enhanced baked anode quality and longer refractory life. The objectives were achieved by regulating the heat distribution in furnaces through appropriate process control systems with customised software. The control strategy was developed from Hindalcoís long experience in plant operation. Hamilton Research and Technology Pvt., Ltd. (HART), India, Hindalcoís development Partner and Hindalco had jointly conceptualized and implemented the system. This paper describes the technology and strategy used. The results of the technology and improvement on plant performance have been presented with actual plant data.

9:45 AM
Creep and Sodium Expansion for Cathode Carbon Materials: Experimental Results, Constitutive and Numerical Modelling: Alexander Zolochevsky[1]; J□rund G. Hop[1]; Guillaume Servant[1]; Trygve FoosnÊs[1]; *Harald A. ÿye*[1]; [1]Norwegian University of Science and Technology, Dept. of Chem., Sem SÊlands veg 12, Trondheim N-7491 Norway

The creep strain and sodium expansion of semigraphitic cathode material have been measured on solid and hollow cylinder samples under various values of applied external pressure. Experimental results have been obtained for various current density values using the Rapoport-Samoilenko-type apparatus. A constitutive model for cathode carbon materials which is able to reproduce the relationship between the sodium expansion and time as well as the relationship between creep strain, external pressure and time during the Rapoport-Samoilenko-type test has been proposed. Parameters required in the proposed model have been calculated from experimental data by a least-square minimisation process. The constitutive model has been extended to a three-dimensional case and has been implemented into the commercial finite element code ANSYS. The distributions of sodium concentration, coordinate components of stress and von Mises equivalent stress in the semigraphitic cathode block with time have been obtained.

10:10 AM Break

10:20 AM
Mathematical Modeling of Sodium Expansion in Prebaked Aluminium Reduction Cells: *Yang Sun*[1]; Qianpu Wang[2]; Morten S□rlie[3]; Harald A. ÿye[1]; [1]Norwegian University of Science and Technology, Dept. of Chem., Trondheim N-7491 Norway; [2]National Research Council Innovation Centre Canada, 3250 E. Mall, Vancouver V6T 1W5 Canada; [3]Elkem Aluminium ANS, Rsrch., PO Box 8040 Vaagsbygd, Kristiansand S N-4675 Norway

A two dimensional transient mathematical modeling of an aluminium reduction cell was developed to study thermomechanical movements and stresses during heat up and early operation using the ANSYS program. In this nonlinear finite element model, material non-linearity and influence of temperature on the mechanical properties were taken into account. Then sodium concentration distribution with time in the cathode carbon was calculated, and the stress distribution and deformation were calculated and analyzed.

10:45 AM
Reactivity of Carbon Materials Against Metallic Sodium: *Noboru Akuzawa*[1]; Ryu Nakajima[1]; Masashi Yamashita[1]; Masahide Tokuda[2]; Chihiro Ozaki[2]; Kenji Ohkura[2]; [1]Tokyo National College of Technology, 1220-2 Kunugida, Hachioji, Tokyo 193-0997 Japan; [2]SEC Corporation, 3-26 Osadano, Fukuchiyama, Kyoto 620-0853 Japan

Carbon blocks being used as the cathode of aluminum-reduction industry are subject to severe degradation under practical operation conditions. One of possible reasons of the problem may be the formation of intercalation compounds induced by the attack of sodium on the cathode blocks. The present investigation aims at clarifying the reactivity of carbon blocks, prepared from petroleum needle cokes, with different heat-treatment temperatures (HTT) against metallic sodium. HTT-2800 carbon gave intercalation compound (NaCx) with the stage 8 structure of identity period (Ic) of 2.8 nm. The electrical resistivity of NaCx showed metallic temperature dependence with a unique hysteresis between 200 and 350 K. HTT-1000 carbon, on the contrary, fixed a large amount of sodium leading to the stage 2 structure (Ic=0.79 nm). Resistivity measurements suggested that the reaction with sodium brought about a fatal damage on the carbon matrix.

11:10 AM
Comparative Characterisation of Graphitised and Graphitic Cathode Blocks: Amir A. Mirchi[1]; *Weixia Chen*[1]; Michel Tremblay[1]; [1]Alcan Inc., Arvida R&D Ctr., 1955 Mellon Blvd., PO Box 1250, JonquiËre, QuÈbec G7S 4K8 Canada

Graphitised and graphitic blocks have been used in many plants over the last decade in order to reduce the cathode voltage drop and energy consumption as well as to adapt to increased amperage. Graphitised and graphitic blocks have higher thermal and electrical conductivities and lower resistance to erosion as compared to the amorphous and semi-graphitic blocks. A better understanding of the properties of these blocks and their equilibrium between cell productivity and life would allow their use to be considered for certain cell technologies. The present study, aimed at building a portrait of each type of block, compares the mechanical, thermal and electrical properties of the commercial graphitised and graphitic cathode blocks.

Cast Shop Technology: Grain Refining

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

Wednesday AM Room: 6C
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Clark Weaver, Alcan Inc., Jonquiere, QC G7S 4L2 Canada; Paul Cooper, London & Scandinavian Metallurgical Company, Rotherham, S. Yorkshire S60 1DL Great Britian

8:30 AM
Grain Refinement in Secondary Al-Si Casting Alloys: M. N. Binney[1]; *D. H. StJohn*[1]; A. K. Dahle[1]; J. A. Taylor[1]; E. C. Burhop[2]; P. S. Cooper[3]; [1]The University of Queensland, Div. of Matls. Eng., CRC for Cast Metals Mfg., Brisbane, Qld 4072 Australia; [2]Metallurg Aluminium, Newfield, NJ 08344 USA; [3]London & Scandinavian Metallurgical Company, Ltd., Metall. Aluminium, Rotherham S60 1DL England

Secondary aluminium alloy castings generally have inferior ductility compared to primary alloy castings because the higher levels of impurity elements tend to cause formation of brittle intermetallic phases. It is possible to reduce the detrimental effect of these impurities by grain refinement since it assists in reducing the size of particles. However, these impurities may also complicate the efficiency of the grain refinement treatment. This work investigates the effects of Cu and some typical impurities in secondary alloys on the grain size of the primary aluminium-silicon alloy A356. The addition of 3wt% Cu to the A356 alloy had little or no effect on grain size. However, a combined addition of Cu plus Fe, Cr, Zn and Mn to A356 to make a pseudo-secondary 319 alloy significantly increased the grain size. When the impurity elements Fe, Cr, Mn and Zn were added individually, Fe and Cr were identified as the elements primarily responsible for causing the increase in grain size.

8:55 AM
Characterisation of a New Generation of Grain Refiners for the Foundry Industry: *Paul Stephen Cooper*[1]; Angela Hardman[1]; Dave Boot[1]; Ed Burhop[2]; [1]London & Scandinavian Metallurgical Company, Ltd., Fullerton Rd., Rotherham, S. Yorkshire S60 1DL England; [2]Metallurg Aluminium, West Blvd., PO Box 768, Newfield, NJ 08344 USA

There have been a number of studies in recent years relating to the mechanisms of grain refinement, poisoning and fade of Al-Ti-B refiners. In addition there have been several new grain refining products introduced for the foundry sector, such as TiBAlloy and Strobloy, for which studies have also been performed, albeit in a more limited way. The new generation of grain refiners have a number of specific beneficial attributes, which distinguish them from the traditional Al-Ti-B refiners. These benefits are explored by characterising the refiner particle types in terms of composition, size, poisoning tendency and fade characteristics.

9:20 AM
Modeling of Microporosity Formation During Solidification of A356 Aluminum Castings: *Rong Ding*[1]; Daan M. Maijer[1]; Steve L. Cockcroft[1]; Jane Howard[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada

The control of microporosity in aluminum alloy castings produced for the automotive industry posses a substantial challenge because of

high cast surface and mechanical performance quality standards. Typically, trial and error methodologies have been used to optimize casting design and casting process parameters, such as cycle time and cooling programs. Microporosity formation during the solification of A356 aluminum castings has been modeled using finite element analysis. Microporosity has been calculated using two fundamentally based approaches. The first is a semi-empirical criterion which takes into account the effect of hydrogen and inclusion content. The second approach employs a critical nucleation pressure and microporosity growth kinetics. The model was developed and implemented in the commercial finite element software, ABAQUS. Calculated results are compared with the experimental data from lab scale test castings and industrial scale wheel castings. The various parameters influencing the formation of microporosity, e.g. the initial hydrogen content, the ingot temperature and the volume shrinkage, are discussed.

9:45 AM
Microstructure Evaluation and Microporosity Formation in AlSi7MgO3 Alloys: *Petru Moldovan*[1]; Gabriela Popescu[1]; Gheorghe Dobra[2]; Carmen Stanica[2]; [1]Polytechnic University Bucharest, Dean of Matls. Sci. & Eng. Fac., Splaiul Independentei 313, Sect. 6, Bucharest 77206 Romania; [2]S. C. ALRO S.A. Slatina, 116 Pitesti St., Slatina 0500 Romania

This article combines the experience of foundry men with research of the theory and prediction of porosity formation. The influence of flux treatment was determined and also the influence of Sr and Sb refining of AlSiMgO.3 alloy over the porosity formation was studied. The densities and densities index (D.I.) were determined of the air and vacuum solidified samples using for this purpose excellent equipment for taking liquid samples and solidifying in vacuum and in air (VAC TEST SYSTEM) and also a device called DENSITY TERMINAL from testing of materials quality laboratory on Materials Science and Engineering Faculty. To establish the mechanism of porosity formation and to evaluate the gaseous porosity, respectively the shrinkage porosity, was used the quantitative microstructure analysis: metallographic microscope BX6M, video camera KPM1 and BUEHLER OMNIMET EXPRESS with a program in 4.0 versions from S.C. ALRO S.A. Slatina. From experimental data the density index (D.I.) and from microstructure analyses it was successfully realize the establishing of the porosity formation mechanism in AlSi7MgO.3 alloy and the influence of different factors over the gaseous porosity and shrinkage porosity.

10:10 AM Break

10:20 AM
Growth-Restriction Effects in Grain Refinement of Aluminium: *Tom Quested*[1]; Lindsay Greer[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

The solute content in aluminium alloys affects grain refinement predominantly through the growth rates of solid grains, or by poisoning the nucleation on refiner particles. Decreases of as-cast grain size with solute content can be described in terms of a universal growth-restriction parameter Q. The fundamental definition of Q is examined. Solute effects, rather than thermal, predominate in growth restriction, even in commercial-purity alloys. The linearity of Q with solute content and its additivity for combined solutes are tested, making use of the thermodynamics software MTDATA. This facilitates separation of growth restriction and poisoning in interpreting measured grain-size trends. Solute addition can accelerate growth, but not in a regime relevant for refinement of conventional alloys; increases in grain size reported to accompany some increases in solute content are attributable entirely to poisoning. Finally, effects of solute diffusivity in, and flow of, the liquid are considered.

10:45 AM
A Comparison of the Family of AlTiB Refiners and their Ability to Achieve a Fully Equiaxed Grain Structure in DC Casting: *Wolfgang A. Schneider*[1]; T. E. Quested[2]; Lindsay Greer[2]; Paul S. Cooper[3]; [1]Hydro Aluminium Germany, R&D, Georg von Boeselager Str. 25, Bonn 53117 Germany; [2]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK; [3]London & Scandinavian Metallurgical Company, Ltd., Fullerton Rd., Rotherham S60 1DL UK

There are a number of grain refiners, based on the AlTiB system, used in the DC casting process. These vary in both concentration and Ti:B ratio. An in depth experimental programme has been performed to compare some of these refiners in the DC casting process. One of the key success criteria used, was to compare the quantity of TiB2 particles from the different refiners, required to achieve a fully equiaxed grain structure. The key role of TiB2 particles is highlighted. The results are discussed in relation to recent theories and modelling on the key characteristics of the TiB2 particles. In particular the particle size distributions of the different refiners are reviewed in relation to the theoretical ideal.

11:10 AM
Effect of the Grain Refinament Process on the Kind and Morphology Intermetallics Phases in the Unalloying Aluminium: *Tomasz Stuczynski*[1]; [1]Institute of Non-Ferrous Metals, Light Metals Div., Pilsudskiego 19, Skawina 32-050 Poland

Effect of the grain refinement process on the kind and morphology of intermetallic phases existing in the two varieties of the unalloying aluminium (Fe+Si=0,5 and Fe+Si=1,0) solidifying at difference cooling rate have been presented. The five kinds of intermetallic phases type AlxFeySiz and AlTFe(Si) have been classified taken as criterion their chemical composition. The main conclusion is, that the grain refinement process conducted by AlTi5B1 (100 ppm Ti) addition promotes to growth the intermetallic phases type AlTFe(Si) (Si/Fe=0,13-0,04) as eutectics grains.

11:35 AM
Production of Al-B Master Alloys from Boron-Bearing Salts Using Different Techniques: *Ibrahim Hamed Aly*[1]; M. A. Shaheen[2]; Abdel-Nasser M. Omran[3]; [1]Minia University, Cheml. Eng. Dept., El-Minia Egypt; [2]Suez Canal University, Metallurgl. Dept., Suez Egypt; [3]R&D Aluminum Company of Egypt, Nag-Hammady Egypt

An Al-B alloy containing about 5% boron has been obtained by reacting of potassium fluoroborate (KBF4) with aluminum. The parameters affecting the reaction process such as: temperature, time, mixing speed, potassium fluoroborate to aluminum weight ratio and particle size were studied. The experiments were carried out in three techniques: first by using KBF4 only, second adding mixture of KBF4 and granular aluminum and third using mechanical alloying. The results indicated that the second technique is considered to be the best one. Microstructure examinations and X-ray diffraction tests were carried out on the produced alloys.

Computational Phase Transformations: Effect of Internal Defects and External Fields

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

Wednesday AM Room: 11B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: John W. Morris, University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA; Yunzhi Wang, The Ohio State University, Dept. Matls. Sci. & Eng., Columbus, OH 43210 USA

8:30 AM Invited
Phase Field Microelasticity in Modeling of Multi-Phase, Multi-Dislocation and Multi-Crack Systems: Yu U. Wang[1]; Yongmei M. Jin[1]; *Armen G. Khachaturyan*[1]; [1]Rutgers University, Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA

Phase Field Microelasticity as a particular case of the Phase Field theory has been developed to describe evolution of stress-generating structural inhomogeneities in solids. These inhomogeneities can be coherent new phase precipitates or structural domains formed as a result of diffusional transformations (decomposition, ordering, polymorphic transformation) or diffusionless martensitic transformations. The greatest advantage of the phase field approach is that it does not impose any a priori constraints on possible options in morphological changes during microstructure evolution - the system itself ëíchoosesíí

the optimal way of microstructure rearrangement. Recently, this theory has been extended to the cases of arbitrary multi-void, multi-crack, and multi-dislocation systems spontaneously developing under applied stress. This extension also allows one to characterize an elastically inhomogeneous system regardless of a value of the elastic modulus misfit. In particular, it extends the theory and method to the case of polycrystalline systems. Being combined, these theories and corresponding models can realistically describe simultaneous evolution of three-dimensional nano- and mesoscale microstructures in phase transformation, plastic deformation, and fracture as well as their response to the applied stress. The latter permits to find a relation between structural and mechanical properties of the system. The development of this theory and availability of high-speed computers open the way to a realistic 3D simulation of technologically important materials, such as intermetallic alloys, shape memory alloys, and structural ceramics. Examples of 3D simulations of decomposition in decomposing intermetallics, martensitic transformation in polycrystals of Shape Memory Alloys as well as evolution of self-multiplied cracks and dislocations will be presented.

9:00 AM Invited
Microstructure Evolution Under the Influence of Internal Defects and External Constraints: Yulan Li[1]; Shenyang Hu[1]; Zikui Liu[1]; *Long-Qing Chen*[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

Phase transformations and the accompanying microstructure evolution can be significantly influenced by the presence of internal defects and external constraints. This talk will be focused on the effect of structural defects such as dislocations and mechanical constraints. A three-dimensional phase-field model will be presented. It may be applied to predicting the domain structure evolution in an elastically anisotropic single crystal film, taking into account of the presence of both arbitrary spatial distribution of dislocations and mechanical constraints imposed by a substrate. A number of examples involving structural transformations in thin films with coherent or semi-coherent interfaces will be discussed. It is shown that the mechanical constraint by a substrate can drastically change the relative volume fractions of differently orientated domains and the domain-wall orientations while dislocations can affect the nucleation, domain wall motion, and the final spatial distribution of domains.

9:30 AM Invited
Modeling Topological Defects in Non-Equilibrium Elastic Media: *Ken Elder*[1]; [1]Oakland University, Physics, Rochester, MI 48309-4487 USA

Topological defects created in non-equilibrium processing often play a key role in determining the physical and mechanical properties of materials. Understanding the creation and kinetics of such defects is a challenging theoretical problem since these defects correspond to a limit in which classical elasticity theory becomes singular. In this talk I would like to discuss the use of continuum or phase fields to model the role of topological defects in non-equilibrium elastic media. In particular I would like to focus on a new model that treats elastic and plastic deformations exactly in media with multiple grains and free surfaces.

10:00 AM
Phase Field Modeling of Metal Hydride Growth in a Plastically Deforming Matrix: *Christopher R. Krenn*[1]; Adam J. Schwartz[1]; Wilhelm G. Wolfer[1]; Yongmei M. Jin[2]; Armen G. Khachaturyan[2]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci. Direct., PO Box 808, L-353, Livermore, CA 94551 USA; [2]Rutgers University, Dept. of Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA

Charging many metal films or powders with hydrogen results in the growth of second phase metal-hydride particles. A large volume difference between hydride and metal can induce plastic deformation and result in a significant hysteresis upon removal of the hydrogen. For a palladium hydride system (with an 11% volume change), we model this hysteresis using a three-dimensional phase field model that incorporates a simple model of plasticity. We examine the effects of yield strength and relative nucleation rate on the hysteresis and on the microstructural evolution. Finally, we discuss the applicability of our models for other phase transformations involving large plastic accommodations. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

10:20 AM Break

10:35 AM Invited
Pattern-Directed Phase Separation in Stressed Thin Films: *William C. Johnson*[1]; Steven M. Wise[1]; Jerome Favergeon[1]; [1]University of Virginia, Dept. of Matls. Sci. & Eng., PO Box 400745, Charlottesville, VA 22904-4745 USA

Simulations of phase decomposition in binary and ternary thin films on patterned substrates, are performed using coupled Cahn-Hilliard and phase field equations. Phase formation, growth, coarsening, and the resulting two-phase microstructures are strongly influenced by the substrate geometry and composition, film thickness, the composition dependence of the surface energy density, composition and epitaxial strains, film and substrate elastic constants, and alloy composition. Phase decomposition on a patterned substrate can yield self-organized structures of small particles of various sizes and spacings. Particle sizes are often much smaller than the pattern dimensions and the technique may provide a method for developing monolayers of periodic nanostructures from an initially homogeneous alloy. This work is supported by the US National Science Foundation through the Center for the Nanoscopic Design of Materials and Grant DMR-9902110.

11:05 AM Invited
Elastic Domains in Epitaxial Layers: *Alexander L. Roytburd*[1]; Julia Slutsker[2]; [1]University of Maryland, Matls. & Nucl. Eng., College Park, MD 20742 USA; [2]National Institute of Science and Technology, Matls. Sci. & Eng. Lab., 100 Bureau Dr., Gaithersburg, MD 20899 USA

Theory and phase-field modeling of formation of polydomain structures in epitaxial layers due to relaxation of misfit stress are presented. These structures consisting of the periodic arrangement of domains of the product phase (twins) are results of phase transformations (1st or 2nd orders) of epitaxial layers. Engineering of constraint (changing misfit and the crystallographic orientation of epitaxial layers) is used to design different structures of elastic domains. Results of modeling are compared with homogeneous theory and analytical solutions for simplest polytwin structures. Available experimental data are discussed.

11:35 AM Invited
Effects of Elastic Inhomogeneity on the Diffusional Microstructure Evolution in Two Phase Solids: *Ingo Schmidt*[1]; [1]Institut fuer Mechanik, TU-Darmstadt, Hochschulstr. 1, Darmstadt 64289 Germany

Many of the properties of metallic materials are caused by their microstructure. Specifically, the properties of phase separated alloys are closely related to the morphology of the precipitate particles. A particular microstructure is typically the result of a phase transformation which is interrupted at a certain stage to produce desired material properties, a well known example being single crystalline super alloys. There, the presence of a lattice misfit between the phases results in a morphological evolution driven by the competition between the elastic strain energy and the interfacial energy. The paper deals with the simulation of the diffusional evolution of particle morphologies in orthotropic media, with special attention directed to the influence of the elastic inhomogeneity in the system. Two different approaches are followed in studying the problem, namely the search for equilibrium morphologies of a given number of precipitates in an infinite matrix and the simulation of the temporal evolution of such systems. In particular, shape bifurcation- and rafting phenomena as well as the stability of particle arrangements are addressed. Continuum descriptions assuming a sharp interface between the phases are evaluated with boundary and finite element methods in two and three dimensions. The results are compared with a ëdiscrete atomí model in which the phases are represented by a large number of elementary particles of two different species which are interconnected with linear springs. Here, the evolution of the morphology is simulated using a Monte-Carlo method. This work is joint with Dr. R. Mueller and Prof. D. Gross.

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

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

Wednesday AM Room: 17A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Richard Wagner, GKSS, Geesthacht 21502 Germany; Kwai Chan, Southwest Research Institute, Dept. of Matls. Sci., San Antonio, TX 78228-0510 USA

8:30 AM Invited
Association Between Twinning and Fracture in Two-Phase Titanium Aluminides: *Fritz Appel*[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str., Geesthacht D-21502 Germany

Mechanical twinning has long been recognized as an important deformation mode in γ -based titanium aluminides. The mechanism apparently compensates for the lack of independent slip systems that can operate at comparable stresses and, thus, supports the plasticity of polycrystalline materials. On the other hand, the octahedral planes of γ -TiAl serve as slip planes, twin habit planes and cleavage planes. Thus, blocked slip may easily lead to fracture. This complex association of twinning and fracture is investigated in the present paper. The major areas of the study are: twin nucleation and growth, effects of solid solution and precipitation hardening and stability of twin structures. The study is based on conventional and high resolution transmission electron microscope observations of defect structures in deformed TiAl alloys.

8:55 AM
Twinning as a Crack Tip Deformation Mechanism During Fracture: *Diana Farkas*[1]; [1]Virginia Tech, Matls. Sci. & Eng., Blacksburg, VA 24061 USA

We report atomistic studies of the deformation mechanisms in the crack tip region during low temperature fracture of fcc and bcc metals. The large scale simulations use empirical potentials to study the crack tip response. Twinning is observed in both fcc metals such as Al and bcc metals such as Fe, in the favorable crystallographic orientations. In the case of Al the twins are formed by the emission of Shockley partial dislocations in adjacent slip planes of the (111) type. In the case of bcc Fe, the twins are also formed by the emission of partial dislocations in adjacent slip planes of the {112} type. The importance of this mechanism is discussed.

9:15 AM Invited
Grain Boundary Character Distributions of Zr and Ti Grain-Refined Through Equal Channel Angular Pressing: S. Yu[1]; H. S. Ryoo[1]; *S. K. Hwang*[1]; [1]Inha University, Sch. of Matls. Sci. & Eng., 253 Yonghyun-Dong, Nam-Gu, Incheon 402-751 Korea

Commercially pure Zr and Ti were grain-refined through equal channel angular pressing (ECAP). With variation of the die design and the processing parameters such as the temperature and subsequent annealing, varieties of microstructures resulted in. Up to a strain of 1.8 was obtained by using a 90?a/20?a die, which resulted in a grain refinement to about 200nm with repeated ECA-pressing. The two metals showed a considerable difference in the initial texture. However, ECA-pressing converted the texture into a basically same preferred orientation, a strong (0001) texture inclined to the specimen axis. The grain boundary character distribution of both metals was such that the high angle boundaries were predominant while the fraction of the low angle boundaries was slightly higher than that in a random microstructure. Both the texture and the misorientation distribution of the severely deformed Zr and Ti persisted during the subsequent exposure to an elevated temperature.

9:40 AM
Ab Initio Calculations of the Structure and Energy of the (11-21) Twin Boundary in HCP Metals: *James R. Morris*[1]; Yiying Ye[1]; Man H. Yoo[2]; [1]Iowa State University, Metal & Ceram. Scis., Ames Lab., USDOE, Ames, IA 50011 USA; [2]Oak Ridge National Laboratory, Metall. & Ceram. Div., Oak Ridge, TN 37831-6115 USA

The (11-21) twin boundary is important in controlling the deformation behavior of HCP metals under tensile strain along the c-axis, and can affect the polycrystalline ductility of a material. We have examined two possible structures of this boundary using accurate ab initio calculations. The two structures have different symmetries: one is a pure mirror twin, the other has a glide plane symmetry. Both structures have been seen experimentally. While empirical potentials predict different energies for these structures, we find the surprising result that the two structures are nearly identical in energy, for all materials examined. We compare the results with calculations from various empirical potentials. The close energy is consistent with the experimental observations that both may occur. The results suggest that the structure found from deformation experiments may be due to the dynamics of twinning, rather than from energetic considerations of the boundary.

10:00 AM
Monazite Deformation Twins: *Randall S. Hay*[1]; David B. Marshall[1]; [1]AFRL/MLLN, Bldg. 655, Area B, WPAFB, OH 45433-6533 USA

Polycrystalline monazite (monoclinic $LaPO_4$) was deformed at room temperature by a spherical indenter. Deformation twins were identified by TEM in 70 grains. Twinning on (100) was by far the most common. Twinning on (001) and (120) was less common, and twinning on (122) was rare. (120) twins were kinked. The twinning modes on these planes were inferred from the surface expression of twinning shear, predictions of classical deformation twinning theory, and other crystallographic considerations. Twinning modes appear to be strongly influenced by the required atomic shuffling. Twin abundance may be related to twin interface energy. The three most common twins have low strain to low Sigma CSLs. Abundance of (100) twins may be related to a low-energy interface structure of a layer of xenotime of Ω unit cell thickness. The (001) twin forms a xenotime interface structure similar to that for (100). Minimal shuffling requires the twin interface to be a glide plane with displacement vector R=Ω[010] for (001) twins, and R=Ω[011] for (122) twins.

10:20 AM Break

10:40 AM Invited
Nucleation of Twins in Face Centered Cubic Metals: Y. Q. Sun[1]; *P. M. Hazzledine*[2]; [1]University of Illinois, Dept. of Matls. Sci. & Eng., Urbana, IL 61801 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

The nucleus for a twin, even one as small as a single loop of Shockley partial dislocation, may be modeled as an Eshelby inclusion embedded in a matrix of untransformed material. Shear nuclei of this type, consisting of one or many dislocation loops, interact with each other attractively through their elastic fields. Because of this interaction, the nucleation process becomes collective and therefore more probable than it would be in the absence of the interaction. In this paper, a model is presented of collective nucleation of twins. The model shows that, at a given temperature, the onset of twinning is a sudden process which occurs at a critical stress. The value of the critical stress falls gradually, and almost linearly, with increasing temperature.

11:05 AM
Linking Electronic and Dislocation Parameters to Fracture Resistance in Metals and Intermetallics: *Kwai S. Chan*[1]; [1]Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA

The possible linkage of electronic and bonding variables to material parameters that describe dislocation nucleation, dislocation mobility, and the fracture resistance of metals and intermetallics are investigated in this paper. Using appropriate analytical methods, the d-level energy, bond order, unstable stacking energy (USE) and the Peierls-Nabarro (P-N) barrier energy are computed for a variety of intermetallics either with or without alloy additions. Similar calculations are also performed for Nb-Ti-Al solid solution with either the bcc or B2 structure. The computed values of d-level energy, bond order, USE and P-N barrier energies are compared and correlated against fracture toughness data to establish (1) relations between electronic variables, dislocation parameters, and fracture resistance, and (2) means for improving the fracture resistance through alloying additions. Work supported by AFOSR through Contract No. F49620-01-C-0016, Dr. Craig S. Hartley, Program Manager.

11:25 AM Invited
Investigation on the Superdislocation Dissociation Widths in $L1_0$ TiAl: Xiping Song[1]; *Guoliang Chen*[1]; Y. R. Ren[1]; [1]University of Science and Technology Beijing, State Key Lab. for Adv. Metals & Matls., Beijing 100083 China

The paper systematically investigates the effects of anisotropic elasticity, superdislocation dissociation configurations, deformation temperatures as well as lamellar interfaces on the superdislocation dissociation widths at the condition of the same stacking fault energy (SFE). The calculation results showed: 1. In the anisotropic elasticity condition both the superdislocation dissociation widths and the difference in the dissociation widths between edge and screw superdislocations become smaller than that in the condition of isotropic elasticity. 2. The superdislocation dissociation widths of the superlattice intrinsic stacking fault (SISF) in the 3-fold or 4-fold superdislocation dissociations configurations were calculated to be smaller than that in 2-fold dissociation. 3. The superdislocation dissociation widths were also affected by the non-coplanar dissociation configuration. The influence of non-coplanar dissociation configuration is related to the Burgers vectors of dissociated superdislocations. 4. The experimental results showed that deformation temperatures had little effect on the superdislocation dissociation widths. 5. The dissociation width of superdislocations is affected by the dislocation location. The dissociation width became wider from the twin interfaces of γ-γ_T type than inside of the γ lamellar. On the basis of the investigation the accuracy of the measurement of SFE by using weak beam TEM technology was improved significantly, and the dislocation mobility is also discussed.

11:50 AM Invited
Encounters with Twinning: *Terence E. Mitchell*[1]; [1]Los Alamos National Laboratory, Struct./Prop. Relations, MST-8, MS G755, Los Alamos, NM 87545 USA

Man Yoo has made numerous contributions to the science of twinning. The present author has also had occasional but regular encounters with the phenomenon of twinning starting with his doctorate studies. These have included both deformation twinning in simple fcc, bcc and hcp metals (copper, tantalum and hafnium respectively), in ceramics such as sapphire and in intermetallics such as the C15 Laves phase HfV_2. Transformation twinning has also been encountered in a variety of ceramics such as zirconia, silicon carbide and perovskite-based high temperature superconductors and in a variety of intermetallics such as CuAu and $MoSi_2$. Twinning has also been found in incommensurate structures of $ReSi_{2-x}$. More recently twinning has been encountered in plutonium and uranium, two materials of vital importance to Los Alamos. Stories of these serendipitous encounters will be woven together to address the question of why twinning is so widespread but why it is not universal.

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

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

Wednesday AM Room: 16B
March 5, 2003 Location: San Diego Convention Center

Session Chair: Lawrence E. Murr, University of Texas at EI Paso, MetallurgI. & Matls. Eng., EI Paso, TX 79968-0520 USA

8:30 AM
Grain Size Dependence of the Flow Stress of Metals from Millimeters to Nanometers: *Hans Conrad*[1]; [1]North Carolina State University, Matls. Sci. & Eng., Raleigh, NC 27695-7907 USA

The effect of grain size ranging from mm to nm on the flow stress of metals at low and intermediate homologous temperatures is considered. Three grain size regimes are identified: Regime I (d=10^-6-10^-3m), Regime II (d=10^-8-10-^6m) and Regime III (d= -10^-8m). Grain size hardening occurs in Regimes I and II and grain size softening (inverse Hall-Petch effect) in Regime III. Regimes I and II correspond to intragranular dislocation activity, with Regime II being characterized by the absence of dislocation cells and twins. Regime III is characterized by the absence of intragranular dislocations and results from grain boundary shear produced by the applied stress alone.

9:00 AM
Grain Size Effects on the Strength and Work Hardening in HCP Metals Titanium, Zirconium, and Cadmium: *Placid Rodriguez*[1]; [1]Defence Research & Development Organisation, Recruit. & Assess. Ctr., Lucknow Rd., Timarpur, Delhi 110054 India

Ronald W. Armstrong is a pioneer in combining Hall-Petch analysis with thermal activation strain rate analysis (TASRA). One of his early predictions was that for HCP metals that deform by basal slip, the Hall-Petch slope K would be dependent on temperature and strain rate. (To maintain continuity across grains, more than one slip system has to be activated and temperature dependence of flow stress will be controlled by the more difficult prism or pyramidal plane.) The situation is different for zirconium and titanium; multiple slip is possible on the primary prism plane and K is expected to be independent of temperature and strain rate. This paper presents the results of combining TASRA (strain rate change, stress relaxation and temperature cycling experiments) with Hall-Petch analysis in titanium; K is shown to be independent of temperature and strain rate. The results are compared with earlier results on cadmium and zirconium. The results on zirconium are also analyzed to highlight the influence of dynamic strain ageing on the Hall-Petch parameters.

9:30 AM
Computational Modeling of the Mechanical Response of Polycrystalline and Nanocrystalline Metals: *Hsueh-Hung Fu*[1]; David J. Benson[1]; Marc A. Meyers[1]; [1]University of California-San Diego, Mechl. & Aeros. Eng., La Jolla, CA 92093-0411 USA

The grain size effect on the mechanical response of polycrystalline metals was investigated computationally. A phenomenological constitutive description is adopted to build the computational crystal model. The material is envisaged as a composite; the grain interior is modeled as a monocrystalline core surrounded by a mantle grain boundary with a higher work hardening rate response. Both a quasi-isotropic approach and a crystal plasticity of the Pierce-Asaro-Needleman type are used to simulate the grain interiors. The grain boundary is modeled by a Voce equation. Elastic and plastic anisotropy are incorporated into this simulation. An Implicit Eulerian Finite Element Formulation with von Mises plasticity or rate dependent crystal plasticity is used to study the nonuniform deformation and localized plastic flow. The computation predictions are compared with experimentally determined mechanical response of copper with grain sizes ranging from millimeters to the nanocrystalline domain. Research supported by the National Science Foundation and the ARO MURI.

9:45 AM
On the Strength and Ductility of Nanocrystalline Materials: *Marek Doll·r*[1]; Anna Doll·r[2]; [1]Miami University, SEAS, Oxford, OH 45056 USA; [2]Miami University, Mfg. & Mechl. Eng. Dept., Oxford, OH 45056 USA

In the early nineties, there was a widespread belief that nanocrystalline materials would exhibit the unique combination of room temperature ductility and ultrahigh strength. However, the hopes have not materialized. In the present study, we analyze plastic deformation and strengthening mechanisms in the nanometer range, based on results of our own research studies on nanocrystalline NiAl and the literature review. The analysis indicates that the nanomaterials are stronger than their coarse grain counterparts but not as strong as predicted using the conventional Hall-Petch approach. Also, in the nanometer range, grain size softening with decreasing grain size seems to be prevalent as a result of the predominance of diffusional mechanisms and/or the deterioration of sample quality. In intermetallics, and ceramics, the enhancement of diffusional mechanisms results in measurable, though limited, room temperature ductility. In nanocrystalline metals the increase in ductility provided by diffusional mechanisms is insignificant compared to the loss of dislocation controlled ductility.

10:00 AM
Grain Size Effects in the Fracture of Nanocrystalline Fe: A Simulation Study: *Diana Farkas*[1]; Antoine Latapie[1]; [1]Virginia Tech, Matls. Sci. & Eng., Blacksburg, VA 24061 USA

Crack propagation studies in nanocrystalline bcc iron samples with grain sizes ranging from 6 to 12 nm are reported at temperatures ranging from 100K to 600K using atomistic simulations. For all grain sizes, a combination of intragranular and intergranular fracture is ob-

served. Mechanisms such as grain boundary accommodation, grain boundary triple junction activity, grain nucleation and grain rotation are observed to dictate the plastic deformation energy release. The effects of grain size on the observed deformation mechanisms is discussed.

10:15 AM

Molecular-Dynamics Study of Mechanical Deformation in Nanocrystalline Metals: *Kai Kadau*[1]; Timothy C. Germann[2]; Peter S. Lomdahl[1]; Brad Lee Holian[3]; Dirk Kadau[4]; Peter Entel[5]; Magnus Kreth[5]; Frank Westerhoff[5]; Dietrich E. Wolf[5]; [1]Los Alamos National Laboratory, Theoretl. Div., T-11, MS B262, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Appl. Physics Div., X-7, MS D413, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Theoretl. Div., T-12, MS B268, Los Alamos, NM 87545 USA; [4]Gerhard-Mercator-Universitaet Duisburg, Theoretische Physik, Lotharstrasse 1, Duisburg 47048 Germany; [5]Gerhard-Mercator-Universitaet Duisburg, Theoretische Tieftemperaturphysik, Lotharstrasse 1, Duisburg 47048 Germany

Nano-crystalline metals, in which the size of grains is in the nanometer range, exhibit mechanical properties different from ordinary polycrystalline materials, thus these materials are of technological interest. One important example is the increasing hardness with decreasing grain size due to dislocation immobilisation at the grain boundaries, known as the Hall-Petch effect. However when decreasing the grain size below a critical value, sliding processes between the grains decrease the hardness which is called the reverse Hall-Petch effect. We report on molecular-dynamics simulations of tensile loading of nano-phase Cu and Al modelled by an embedded-atom method (EAM) potential. Usage of two different preparation methods of the nano-phase material allow us to compare mechanical properties for different sample qualities: A Voronoi-constructed polycristal exhibit nearly no pores and has different mechanical properties compared to a material that is sintered under pressure and temperature from nano particles.

10:30 AM Break

10:45 AM

Characterization of Nanocrystalline and Microcrystalline Copper Consolidated Using Equal Channel Angular Extrusion: Mohammed Haouaoui[1]; *Ibrahim Karaman*[1]; K. Ted Hartwig[1]; [1]Texas A&M University, Dept. of Mechl. Eng., College Station, TX 77845 USA

Materials with ultrafine grains and nanostructures (<100 nm) have attracted considerable interest because of their unique properties as compared with conventional materials. Although this class of materials seems to offer new opportunities for small scale applications, utilizing them in large scale structural applications is still a challenge due to the difficulty of fabricating nanocrystalline materials in bulk. The present work is focussed on fabrication of full density bulk micro and nanocrystalline materials from powder precursors using equal channel angular extrusion (ECAE). The initial powder sizes that were consolidated were 50 nm, 150 nm and 45 micron. Different processing routes was selected for comparison purposes and to determine the best processing route for specific end microstructures. The density of consolidates is compared to the fully dense theoretical value as a first assessment of consolidation performance. The microstructure is characterized for different ECAE routes and number of passes. Indentation techniques are utilized to measure the hardness. The stress strain responses are determined by tension and compression tests at room temperature. The effect of initial powder size and resulting consolidate grain size on the mechanical properties are discussed in view of the Hall-Petch relationship. SEM is utilized to quantify the residual porosity. Mechanical behavior of the consolidates is compared with. In this talk, some of these experimental observations will be presented in comparison with microcrystalline consolidates and with severely deformed grain refined pure copper. This study helps to clarify the relationship between different ECAE processing parameters, mechanical properties and the microstructure of nanocrystalline copper.

11:00 AM

The Effect of Grain Size on Low Cycle Fatigue of Polycrystalline Al-4.5wt.%Cu Alloy: *Aezeden Omar Mohamed*[1]; Yassir Fouad El-Madhoun[1]; M. N. Bassim[1]; [1]University of Manitoba, Dept. of Mechl. & Industl. Eng., 15 Gillson St., Rm. 356, Eng. Bldg., Winnipeg, Manitoba R3T 5V6 Canada

The effect of grain size on low cycle fatigue of Al-4.5wt.%Cu alloy was studied. Two different grain sizes, 90mm and 150mm, were tested at room temperature. The fatigue response of the alloy was evaluated macroscopically in terms of cyclic stress strain curve and microscopically in terms of appearance of cyclic slip bands. The cyclic stress strain curve exhibited a plateau region where the saturation stress remained constant with plastic strain. Grain size of 90mm exhibited lower cyclic saturation stress and longer plateau than grain size of 150mm. This result contradicts the Hall-Petch relationship of grain size effect. Microscopic observations using optical and scanning electron microscope revealed the presence of persistent slip bands (PSBs) where the strain is highly localized. It was shown that the 90mm grain size contains a higher volume fraction of PSB than the 150mm grain size. Such higher volume fraction of PSBs would result in lower saturation stress since these slip bands act as soft regions within the matrix of cyclically deformed metals.

11:15 AM

Grain Size Effects on Deformation Behavior: A Two-Dimensional Discrete Dislocation Simulation: *James R. Morris*[1]; S. Bulent Biner[1]; [1]Iowa State University, Metal & Ceram. Scis., Ames Lab., USDOE, Ames, IA 50011 USA

The evolution of the flow stress for grain sizes ranging from 2 to 16 microns under shear deformation was simulated using two dimensional discrete dislocation dynamics. The analyses were confined to a single slip system and to the collective behavior of a large number of edge dislocations. Flow stress values increased with decreasing grain size and correlated with grain size with a classical Hall-Petch relationship $d^{-1/2}$, or in the form of d^{-1}. The flow stress values for different grain sizes unified to a single curve when expressed as a function of the dislocation density normalized by the grain size. It was observed that dislocation pileups can activate neighboring dislocation sources and also shutdown the active dislocation sources. This work was performed for the US Dept. of Energy by Iowa State University under contract W-7405-Eng-82. and supported by the Office of Basic Sciences.

11:30 AM

A Critical Assessment of the Grain Size Effects on Fatigue Deformation and Fracture of a Metastable Austenitic Stainless Steel: K. Bhanu Sankara Rao[2]; Sardari Lal Mannan[2]; *Placid Rodriguez*[1]; [1]Defence Research & Development Organisation, Recruit. & Assess. Ctr., Lucknow Rd., Timarpur, Delhi 110 054 India; [2]Indira Gandhi Centre for Atomic Research, Kalpakkam 603 102 India

The effects of grain size (75, 300 and 700 mm) on LCF life, cyclic stress response, and deformation and damage mechanisms of a type 304 stainless steel in the temperature range of 300 to 1023K were investigated through strain-controlled fatigue tests employing strain amplitudes in the range ± 0.25 to 1.25%. The cyclic deformation modes and damage mechanisms were significantly influenced by grain size and the temperature and strain amplitude employed during LCF test. The combined influence of temperature and grain size on cyclic stress response and life is explained on the basis of slip character, martensitic transformation, dynamic strain ageing, creep deformation, deformation induced precipitation and oxidation in their respective temperature domains of predominance. Hall-Petch relationship is valid at 823K and 923K between half-life stress and grain size; it is found not valid at 300K in this metastable steel due to the occurrence of a pronounced phase change from austenite to martensite.

11:45 AM

Grain Size Effects on Fatigue: *Shankar M.L. Sastry*[1]; [1]Washington University, Mechl. Eng., CB 1185, One Brookings Dr., St. Louis, MO 63130 USA

The effect of grain size on fatigue behavior was investigated in polycrystalline Ti-6Al-4V, dispersion strengthened Cu, precipitation strengthened Al, and low carbon steel. Ultra fine grain sized samples were produced by severe plastic deformation processing by equal channel angular extrusion and concurrent or post deformation recrystallization annealing. A reduction in grain size from 20 to 2 mm resulted in an improvement of fatigue life by three orders of magnitude and 30% increase in fatigue strength in Ti-6Al-4V. Similar but less dramatic improvements were observed in other materials.

Friction Stir Welding and Processing II: Friction Stir Joining: Structures & NDE

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Thomas J. Lienert, University of South Carolina, Mechanical Engineering Department, Columbia, SC 29208 USA; Rajiv S. Mishra, University of Missouri-Rolla, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Wednesday AM Room: 7B
March 5, 2003 Location: San Diego Convention Center

Session Chair: Ali A. Merati, National Research Council Canada, Inst. for Aeros. Rsrch., Ottawa, Ontario K1A 0R6 Canada

8:30 AM
Enabling Technologies for Manufaturing Metallic Cryogenic Tanks: *Ray Miryekta*[1]; Carolyn Russell[2]; [1]Boeing, Integrated Defense Sys. USA; [2]Marshall Space Flight Center, Huntsville, AL 35812 USA

Engineers and scientists from NASA and Boeing are exploring the feasibility of the developmental aluminum technologies related to materials and related processes necessary for manufacturing metallic cryogenic tanks. This team currently is in the process of developing manufacturing technologies suitable for processing aluminum-lithium alloys. Feasibility of using these alloys is greatly dependent on their optimized properties required for the above application. Extensive effort is being directed towards identifying a suitable joining process with superior capabilities, quality, and minimum properties loss inherent to the joining process characteristics. Friction Stir Welding (FSW) technology is being seriously pursued. To date, the preliminary weld test results for welds made using conventional FSW equipment have higher mechanical properties over equivalent fusion welds. Similar studies in the areas of friction stir plug-welding (FSPW) technologies were also conducted by different groups of engineers. Results of these activities showed that a local rework can be performed by push or pull plug-welding methodologies. This talk will discuss some of this ongoing work.

8:55 AM
Monitoring of Friction Stir Welding Process Using Acoustic EmissionñA Preliminary Study: *Changming Chen*[1]; *Radovan Kovacevic*[1]; [1]Southern Methodist University, Rsrch. Ctr. for Adv. Mfg., 1500 International Pkwy., Ste. 100, Richardson, TX 75081 USA

Acoustic emission (AE) generated during friction stir welding (FSW) is detected and a preliminary study is being conducted in order to investigate the possibility of applying this sensing technique for an in-process monitoring of FSW. Using two, static transducer sensors well-coupled with the workpieces, the AE signals are acquired for various welding parameters: the tool rotational speed, the traverse speed and down force, the probe, the tool with a probe, and the tool without a probe. In addition to the normal samples, two butted 6061 aluminum alloy plates with three, equal-spaced gaps made of two notches aligned along the butt joint of the parts are also used for the AE investigation. The AE signals are characterized by the hit rate vs. amplitude, and the rise time and energy vs. time. The analysis of the AE signals provides useful indicators of the tool transient movement and weld quality during FSW, and can be used to identify tool penetration, tool pullout and material defects formed during FSW.

9:20 AM
New Developments of the Ultrasound Phased Array and Eddy Current Arrays Technologies for the Evaluation of Friction Stir Welds: *Colin Bird*[1]; AndrÈ Lamarre[2]; [1]TWI Ltd, Granta Park, Great Abington, Cambridge CB1 6AL UK; [2]R/D Tech, Industl., 505 Blvd. du Parc-Technologique, QuÈbec G1P 4S9 Canada

The use of friction stir welding techniques (FSW) to assemble thin aluminum plates in aerospace applications brought about the need for a high-resolution, non-destructive testing technology to find and characterize the small defects, as kissing bonds, that may occur when using FSW. Phased array ultrasonics and eddy current arrays have been applied to FSW with success. This paper will discuss phased array ultrasonics and eddy current arrays and its application to friction stir welding of thin aluminum plates. Emphasis will be on detection of kissing bonds with both techniques.

9:45 AM
A New Method of Obtaining the Part-Through Fracture Toughness (KIe): *Bahram (Bob) Farahmand*[1]; [1]Boeing Technical Fellow, 5301 Bolsa Ave., Huntington Beach, CA 92647 USA

This work reports on a new approach for estimating the part-through fracture toughness, KIe. The KIe is used to calculate the wall thickness in life analysis of the base and weld regions of pressurized tank. It can be estimated through the ASTM-E740 and its value depends on specimen thickness, crack size, and the characteristic of the material. Improper value of KIe can yield unacceptable results. Smaller KIe value results in higher wall thickness, and a larger KIe value can provide smaller thickness, which can cause premature failure of the tank. Therefore, it is recommended to estimate a meaningful KIe for life analysis that is most suitable for direct application to design. That is, a KIe test that simulates the service condition and without violating the linear elastic fracture mechanics assumptions. The KIe versus plate thickness for several surface crack specimens were investigated with this approach. Results were plotted for base and friction stir weld metals.

10:10 AM Break

10:20 AM
Materials Fracture Properties Estimation by Eliminating Physical Testing: *Bahram (Bob) Farahmand*[1]; [1]Boeing Technical Fellow, 5301 Bolsa Ave., Huntington Beach, CA 92647 USA

In material selection study of aircraft or aerospace components, fracture toughness value and fatigue crack growth rate data are important parameters for service life analysis. When calculating the number of cycles to failure, both quantities must be available through ASTM testing standards, which are costly and time consuming. The proposed analytical approach can provide fracture toughness and fatigue crack growth rate curve by using only static parameters. The energy absorption rate at the crack tip is derived from Griffith concept and used to calculate fracture toughness. This quantity is used to establish region III of the da/dn curve. The threshold region was estimated through the Kitagawa concept. Two additional points were estimated in region II and were used to establish the Paris constants. Several alloys were selected and results were compared with this approach. Excellent agreement between experimental data and analytical method were found. The application of this concept will result in significant saving.

10:45 AM
Friction Stir Welding of Steels: *Tracy W. Nelson*[1]; Carl D. Sorensen[1]; Colin Sterling[1]; Scott M. Packer[2]; [1]Brigham Young University, Mechl. Eng., 435 CTB, Provo, UT 84602 USA; [2]Advanced Metal Products, 2320 North 640 W., W. Bountiful, UT 84087 USA

Friction stir welding (FSW) is a well established joining process for welding aluminum and other lower melting temperature metals. The application of this process to steels and stainless steels has primarily been limited by the availability of suitable tool materials. This paper will present the result of FSW in hi-hard armor steel. FSW were successfully made using polycrystalline cubic boron nitride (PCBN) tool material which exhibited negligible wear after 5 meters of weld. Transverse metallographic samples indicate excellent weld quality. Post weld transverse tensile properties were in excess of 70% of the base metal yield and tensile strengths. More detailed characterization of tool life along with weld microstructure and mechanical properties will be presented.

11:10 AM
FSW of Titanium Turbine Engine Components: *Timothy J. Trapp*[1]; [1]Edison Welding Institute, Navy Joining Ctr., 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

This abstract describes a USAF Metals Affordability Initiative Project being perfromed by GE Aircraft Engines and EWI to develop, demonstrate, and deploy friction stir welding for joining titanium engine components. The goal of this project was to qualify FSW procedures for titanium alloy applications. Other goals included improved joint strength, production of welds that were free of tool debris, and reduced manufacturing cost and distortion. To achieve these goals, significant advancement in FSW tool design and materials were required. The project developed new FSW tool designs and materials, developed welding procedures for dissimilar titanium alloy combinations, and provided metallurgical and mechanical property data. This presentation will provide a brief overview of the project and potential applications. The presentation will discuss in detail the FSW tool and procedure development efforts, the FSW microstructure and mechanical properties, the effect of pre and post weld heat treatment on microstructure and properties.

11:35 AM
FSW of the Advanced Amphibious Assault Vehicle: *Timothy J. Trapp*[1]; [1]Edison Welding Institute, Navy Joining Ctr., 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

This abstract describes a Navy MANTECH Project to develop and demonstrate FSW for joining Aluminum 2519-T87 armor on the Advanced Amphibious Assault Vehicle (AAAV). Alloy 2519, which is known for high strength and superior ballistic performance, is being used on the Marine Corpsí AAAV. General Dynamics Land Systems, the AAAV Program Office, and EWI conducted the project. The goals of this project were to produce welds with equivalent or greater strength and ductility compared to conventional arc welds, to successfully pass the ballistic shock test, to develop procedures to reduce weld distortion and manufacturing costs. Efforts included development of robust FSW tools, of a FSW distortion process model, and of procedures for groove, corner, and t-joint configurations. This presentation will provide a brief overview of the project and discuss in detail the weld joint designs, resultant microstructures and mechanical properties, and ballistic test results.

12:00 PM
Metallurgical and Mechanical Properties of a Friction-Stir Welded Aluminium Alloy 6056 T4 and T6 Conditions: L. Roldo[1]; C. Schilling[1]; M. Chludzinski[1]; *J. F. dos Santos*[1]; T. Strohacker[1]; [1]GKSS Forschungszentrum GmbH, Inst. for Matls. Rsrch. Joining Tech., Max-Planck-Str., Geesthacht D-21502 Germany

The present work has been carried out as part of the European project WAFS ñ Welding of Airframes with Friction Stir and addresses the structure property relationships in a friction stir welded Al 6056 alloy produced with different energy inputs in T4 and T6 conditions. The microstructure has been investigated using electron microscopy. The local properties of the weld zone have been determined using microflat tensile testing and an optical deformation measuring system. Additionally, thermal cycles have been measured during welding in order to support the understanding of metallurgical transformations in the weld zone.

High Temperature Alloys: Processing for Properties: Wrought Alloys

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee
Program Organizers: Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA; Jacqui B. Wahl, Cannon-Muskegon Corporation, Muskegon, MI 49443-0506 USA

Wednesday AM Room: 13
March 5, 2003 Location: San Diego Convention Center

Session Chair: Gerhard E. Fuchs, University of Florida, Dept. of Matls. Sci. & Eng. , Gainesville, FL 32611-6400 USA

8:30 AM
Processing of Cold-Rolled Nickel by Directional Recrystallization: *I. Baker*[1]; J. Li[1]; B. Iliescu[1]; A. Badmos[1]; H. J. Frost[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA

The effects of hot zone velocity and temperature gradient ahead of the hot zone during directional annealing at 1273 K have been investigated for 10 mm thick, cold-rolled nickel sheets using both electron back-scattered patterns on a scanning electron microscope and optical microscopy. Some cold-rolled nickel specimens were first statically recrystallized at 643 K prior to directional recrystallization at 1273K in order to examine whether directional recrystallization at 1273 K was by primary or secondary recrystallization. It was shown that directional recrystallization to a columnar grain structure or, under the optimum conditions, a single crystal was always by directional secondary recrystallization. Columnar grains could be produced over a wide range of hot zone velocities (2-100 mm/h) when a large temperature gradient (1000°C/cm) was maintained ahead of the hot zone, the column width decreasing with increasing hot zone velocity. In contrast, for a low temperature gradient ahead of the hot zone, only equiaxed microstructures were produced. The results will be compared with simulations of the processing. Research supported by AFOSR grant F49620-00-1-0076 and NSF grant DMI9976509.

8:50 AM
An Investigation of Processing-Microstructure-Property Relationships of Inconel 718 Through Grain Boundary Engineering: *Carl Boehlert*[1]; Nate Eisinger[2]; Serkan Civelekoglu[1]; Gaylord Smith[2]; James Crum[2]; [1]Alfred University, Sch. of Ceram. Eng. & Matls. Sci., CEMS/McMahon Hall, 2 Pine St., Alfred, NY 14802 USA; [2]Special Metals Corporation, Huntington, WV USA

Since the development of Inconel 718 by H. L. Eiselstein almost 40 years ago, this nickel-based superalloy has gained wide industrial acceptance due to its properties, fabricability, and cost effectiveness. It is the most widely used superalloy, and its applications include ranging from rotating and static components in aircraft jet engines to high-temperature tooling for extrusion and shearing to components of nuclear reactors and space vehicles. Recently an effort to improve the elevated-temperature creep resistance of this alloy through grain boundary engineering has been initiated. Grain boundary engineering involves evaluation of the grain boundary character distribution (GBCD) as a function of processing. Grain boundary engineering of face-centered-cubic metals and alloys, including nickel-based superalloys, has shown that dramatic improvements in creep resistance are possible when the concentration of low-angle boundaries (LABs) and coincident-site lattice boundaries (CSLBs) is increased. This may be accomplished through sequences of cold rolling and annealing inducing strain recrystallization. Using electron backscatter diffraction (EBSD), the GBCD of Inconel 718 was evaluated after cold rolling 0, 10, 20, 30, and 40% followed by annealing at temperatures between 1610-1850∞F. After subsequent aging treatment, the alloy was tensile-creep tested and the influence of GBCD on creep rupture life and elongation was evaluated. In addition the room-temperature tensile strength, elongation, and hardness were evaluated. With increased cold rolling the concentration of LABs+CSLBs increased and this was correlated to increased tensile strength, hardness, and creep rupture life. The potential for optimization of properties for Inconel 718 through grain boundary engineering is discussed.

9:10 AM
Realistic Subscale Evaluations of the Mechanical Properties of Advanced Disk Superalloys After Supersolvus Quenching Heat Treatments: *Tim P. Gabb*[1]; Jack Telesman[1]; John Gayda[1]; Peter T. Kantzos[2]; William A. Konkel[3]; [1]NASA Glenn Research Center, 21000 Brookpark Rd., MS 49-3, Cleveland, OH 44135 USA; [2]Ohio Aerospace Institute, 21000 Brookpark Rd., MS 49-7, Cleveland, OH 44135 USA; [3]Wyman-Gordon Forgings, 10825 Telge Rd., Houston, TX 77095 USA

In order to fully utilize the higher temperature combustor and airfoil concepts under development, there is a need to increase the temperature capabilities of superalloy turbine disks. One approach to meet this goal is to modify the processing and chemistry of advanced alloys, while preserving the ability to use supersolvus heat treatments to achieve coarse grain microstructures. An important step in this effort is to understand the key high temperature mechanical properties of advanced alloys after such supersolvus heat treatments. However, it can be very expensive to screen the effects of alloy and process modifications on mechanical properties using full-scale, production disk shapes having thick bore and narrow rim sections. In this study, the mechanical properties of several advanced disk superalloys were compared using subscale pancakes after supersolvus heat treatments with realistic cooling paths. Selected results will also be compared to properties from large disks.

9:30 AM
Alternate Processing of Two Heat-Resistant Alloys for Improved Properties: *L. M. Pike*[1]; S. K. Srivastava[1]; [1]Haynes International, Eng. & Tech., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

A number of sheet applications exist in the aerospace and LBGT industries in which the operating temperatures are in the intermediate range (1000∞F to 1400∞F). These include pneumatic ducting, tailcones, thrust reverser parts, bellows, recuperators, etc. While stainless steels are often used in that temperature range, many of these applications require greater tensile and low cycle fatigue (LCF) strength. Heat resistant alloys such as 625 and HR-120Æ alloys offer substantially improved mechanical properties, but in many cases their processing is controlled to produce a microstructure which provides optimum properties in the high temperature range (1400 to 2000∞F) where creep strength is of primary concern. In this paper we will describe how alternate processing routes have led to the development of two alloy products (625SQÆ alloy and fine-grained HR-120 alloy) which have improved tensile and LCF properties in the intermediate temperature range.

9:50 AM Break

10:10 AM
Optimization of Oxide Dispersion Strengthened FeCrAl and Fe3Al Alloy Properties: *Bimal Kad*[1]; James Heatherington[1]; Claudette G. McKamey[2]; Ian G. Wright[2]; Vinod K. Sikka[2]; Rod R. Judkins[2]; Gaylord Smith[3]; Mark A. Harper[3]; [1]University of California-San Diego, 409 University Ctr., La Jolla, CA 92093-0085 USA; [2]Oak Ridge National Laboratory, M & C Div., MS-6114, Oak Ridge, TN 37831-6114 USA; [3]Special Metals Corporation, 3200 Riverside Dr., Huntington, WV 25705-1711 USA

Mechanically alloyed oxide dispersion strengthened (ODS) Fe-Cr-Al and Fe_3Al alloy thin walled tubes and sheets, produced via powder consolidation methodologies, are promising materials for eventual use at temperatures up to 1200°C in the power generation industry, far above the temperature capabilities of conventional alloys. Target end-uses range from gas turbine combustor liners to heat exchanger tubes. Grain boundary creep processes at service temperatures are the dominant failure mechanisms for such components. The processed ODS alloy microstructure consists of elongated grains parallel to the tube axis, a result of dominant axial metal flow which aligns the dispersoid particles and other impurities in the longitudinal direction. This dispersion distribution is unaltered by recrystallization treatments and the high aspect ratio grain shape typically obtained limits transverse grain spacing and consequently the hoop creep response. This represents a critical materials design and development challenge that must be overcome in order to fully exploit the potential of ODS alloys. We will describe our attempts to improving hoop creep in ODS-alloy components by manipulating the factors that dictate and control the recrystallization behavior. *Research sponsored by the Office of Fossil Energy, Advanced Research Materials Program, US DOE under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

10:30 AM
High Temperature Microstructural Stability of a MA/ODS Ferritic Alloy: *Michael K. Miller*[1]; David T. Hoelzer[1]; Suresh S. Babu[1]; Edward A. Kenik[1]; Kaye F. Russell[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6136 USA

Mechanical-alloying (MA) of fine pre-alloyed metal and Y2O3 powders has been shown to produce oxide dispersion-strengthened (ODS) ferritic alloys with dramatically improved high temperature mechanical properties. Atom probe tomography has revealed that the improved high temperature mechanical properties are correlated with the presence of ultrastable 4-nm-diameter Ti-, Y- and O-enriched particles and significant enrichments of Cr, W, Ti, Y, O, C and B in the vicinity of dislocations. These ultrafine particles were stable during long term creep experiments and annealing at temperatures of up to 1300∞C. The Y and O levels in the ferrite matrix were significantly higher than the equilibrium levels. These results may be related to the O-Ti, O-W, O-Y and O-Cr solute atom interactions influencing solute diffusion. Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Laboratory Directed Research and Development Program and the Division of Materials Sciences and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

10:50 AM
Microstructural Refinement of Pure Nb by Severe Plastic Deformation: *K. Ted Hartwig*[1]; *Don Bryant*[1]; [1]Texas A&M University, Mechl. Eng., 319 Eng. Physics Bldg., College Station, TX 77843-3123 USA

Electron beam remelted and cast pure niobium was deformed at room temperature up to strains of 9.2 by multipass equal channel angular extrusion through a tool containing an abrupt 90° angle. The as-worked microstructure shows a progression from dense dislocation tangles to subgrains with dimensions on the order of several hundred nanometers at a strain of 4.6. Recrystallization temperature, recrystallized grain size and the uniformity of the recrystallized microstructure as a function of the level of prior cold work will be reported.

11:10 AM
Microtexture Analysis in a Forged Alpha/Beta Ti Alloy: Crystallographic and Morphological Aspects of Phase Constituents: *Dhriti Bhattacharyya*[1]; Gopal Viswanathan[1]; Rajarshi Banerjee[1]; David Furrer[2]; Hamish Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Ladish Company, 5481 S. Packard Ave., Cudahy, WI 53110 USA

Texture is an important component in Ti alloy development both in terms of processing and the mechanical properties. While the macrotexture is important for understanding the flow characteristics of the forging process, the microtexture is crucial for the mechanical properties. Especially, the crystallography of the alpha and beta phases and the orientation relationship between them have a prominent role in the microtexture and the mechanical properties of alpha/beta Ti alloys. In this study, the micro texture measurements have been obtained from selected (alpha+beta) and beta forged Ti-6246 alloy by Orientation Imaging Microscopy (OIM). The results indicate that in beta forged alloy the a/b colonies having close crystallographic relationship but morphologically different are present both within and across prior beta grain boundaries. TEM samples have been extracted from these special boundaries using Focussed Ion Beam (FIB) slicing technique and detailed analysis have been conducted. This analysis combined with the micro texture measurements from OIM will be presented and possible explanations for the observed phenomena given.

Hot Deformation of Aluminum Alloys: Processing, Structure and Property - I

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

Wednesday AM Room: 6E
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Amit K. Ghosh, University of Michigan, Matls. Sci. & Eng., Ann Arbor, MI 48109-2136 USA; Armand J. Beaudoin, Jr, University of Illinois at Urbana-Champaign, Dept. of Mechl. & Industl. Eng., Urbana, IL 61801 USA; Menno van der Winden, Corus Research, IJmuiden Techl. Ctr., IJmuiden 1970 CA The Netherlands

8:30 AM Invited
Friction Stir Processing for High Strain Rate Superplasticity: *R. S. Mishra*[1]; Z. Y. Ma[1]; I. Charit[1]; [1]University of Missouri, Dept. of Metallurgl. Eng., 218 McNutt Hall, Rolla, MO 65409 USA

Friction stir processing is a new solid state technique for microstructural modification in metallic materials. This has been used to develop very fine grained microstructure in various aluminum alloys. Friction stir processing leads to predominantly high angle grain boundaries. Combination of very fine grain size and high grain boundary misorientation leads to high strain rate superplasticity in many commercial aluminum alloys. However, abnormal grain growth limits the upper temperature in some aluminum alloys. Various metallurgical issues related to use of friction stir processing for high strain rate superplasticity will be discussed. The authors gratefully acknowledge the support of (a) the National Science Foundation through grant DMR-0076433 and the Missouri Research Board for the acquisition of a friction stir welding and processing machine, and (b) the National Science Foundation through grant DMI-0085044.

8:55 AM
Modeling, Analysis, and Validation of Friction Stir Welding Processes: *Abe Askari*[1]; Stewart Silling[2]; Blair London[3]; Murray Mahoney[4]; [1]The Boeing Company, Bellevue, WA 98008 USA; [2]Sandia National Laboratory, Albuquerque, NM 92647 USA; [3]California Polytechnic State University, Matls. Eng. Dept., San Luis Obispo, CA 93407 USA; [4]Rockwell Scientific Company, Thousand Oaks, CA 91360 USA

Friction Stir Welding (FSW) is a solid-state joining process that has found wide spread use in aerospace applications. Because FSW is an emerging technology, significant advances in innovation and technology transition are possible. Science-based simulation and modeling toolkits, once validated, enable the rapid expansion of FSW technology by providing long-term facility for process design and optimization. These models facilitate rapid innovations in such key areas as optimal pin and shoulder design, new joint designs, optimal tool and work-piece material, joining of dissimilar materials, and optimization of feed, speed, and power requirements. Boeing, in collaboration with Rockwell Science Center and Sandia National Laboratories, has developed predictive computational model for FSW. The model is based on a three-dimensional Eulerian code with complete thermo-mechanical coupling. The code models all the important physical effects with a minimum of assumptions. It includes frictional heating and energy dissipation due to plastic work. Thermal convection and conduction,

including conduction into the tool and base-plate, are included. The Eulerian nature of the code permits very large strains to be modeled. It also allows for prediction of mixing between the work-piece materials. All relevant geometrical details, including pin screw threads and shoulder design are included in the model. The model also predicts detailed thermal and deformation histories that impact the final microstructure of the weld zone and can be exploited to improve fracture, fatigue, and stress corrosion properties of the weld. Detailed welding tests and post weld analysis in high-strength aluminum alloys are used to validate the computational models and improve understanding of basic aspect of FSW process. Placement of both tracers and thermocouples in the weld path, metallographic examination, and computed tomography are used to investigate metal flow dynamics and the mixing of material as well as thermal profile and history in the weld zone.

9:15 AM Invited
High-Temperature Mechanical Behavior of Cryomilled Nanostructured Al Alloys: *E. J. Lavernia*[1]; B. Q. Han[1]; F. A. Mohamed[1]; [1]University of California-Irvine, Dept. of Cheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

Deformation mechanisms of nanostructured materials have attracted intensive investigation recently. We use a processing called consolidation of cryomilled aluminum powders successfully to manufacture large amounts of bulk nanostructured or ultrafine grained aluminum alloys. In the present presentation, the cryomilling processing and the characteristic of microstructure of bulk cryomilled Al-7.5%Mg alloys were reviewed, and mechanical properties at elevated temperatures of cryomilled Al-7.5%Mg alloys, together with microstructure characteristics by transmission electron microscopy and X-ray diffraction patterns, were investigated. The correlation of microstructural characterization, temperature-dependence of strength, plastic deformation mechanisms was discussed in terms of dislocation activity.

9:40 AM Invited
Severe Plastic Deformation of an Al-6061 Metal Matrix Composite: *Yi Huang*[1]; Cheng Xu[1]; Minoru Furukawa[2]; Zenji Horita[3]; Terence G. Langdon[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matls. Sci., 3650 McClintock Ave., OHE430, Los Angeles, CA 90089-1453 USA; [2]Fukuoka University of Education, Dept. of Tech., Fukuoka 811-4192 Japan; [3]Kyushu University, Dept. of Matls. Sci. & Eng., Fukuoka 812-8581 Japan

Equal channel angular pressing (ECAP) is an ideal procedure for processing metallic alloys. This report describes the application of ECAP to an Al-6061 metal matrix composite. It is shown that the use of a solid solution treatment prior to ECAP provides the opportunity of performing ECAP at room temperature for up to five passes through the die. This paper discusses the mechanical properties after ECAP including the effect of a post-ECAP peak aging treatment.

10:05 AM
Development of a Homogeneous Microstructure in Pure Aluminum Processed by ECAP: *Cheng Xu*[1]; Terence G. Langdon[1]; [1]University of Southern California, Depts. of Aeros. & Mechl. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

Equal-Channel Angular Pressing (ECAP) is a newly-developed process utilizing simple shear to refine the grains of materials without changing the dimensions of the samples. It has been found that aluminum alloys processed by ECAP achieve high tensile ductilities and hardness and there is even a potential for attaining superplasticity at high strain rates in some alloys. This report shows that the processing of pure aluminum by ECAP leads to a significant increase in the microhardness and there is a tendency to reach a greater homogeneity with the imposition of increasing strain. The tendency of developing a homogeneous microstructure is advantageous in industrial applications.

10:25 AM Invited
Large-Strain Softening of Aluminum in Shear: *M. E. Kassner*[1]; M. Z. Wang[1]; M. T. Perez-Prado[2]; S. Alhajeri[3]; [1]Oregon State University, Mechl. Eng., Matls. Prog., Rogers Hall, Corvallis, OR 97331-6001 USA; [2]CENIM, CSIC, Madrid Spain; [3]Kuwait Institute Tech, Kuwait City 7065 Kuwait

Pure aluminum deformed in pure shear at elevated temperature reaches a broad ìpeakî stress and then undergoes about a 17% decrease in flow stress with deformation with, roughly, 1-2 equivalent uniaxial strain. Beyond this strain the flow stress is approximately constant. The sources for this softening are unclear. The suggested basis includes texture softening, microstructural softening, enhanced dynamic recovery, and discontinuous dynamic recrystallization. Experiments were performed where specimens were deformed in torsion to various strains within the softening regime followed by compression tests at ambient and elevated temperature. Analysis of the compressive yield strengths indicate that the softening is most likely substantially explained by a decrease in the average Taylor factor.

10:50 AM
Mechanical and Material Responses of an Al-Zn-Mg-Cu Alloy at Intermediate Strain Rates: *Zhe Jin*[1]; Paul Wang[1]; [1]Alcoa Technical Center, Alcoa Ctr., PA 15069 USA

Hot torsion tests were conducted at strain rates from 10/s to 300/s and temperatures from 300F to 880F to study the mechanical and material responses of aluminum alloys over the friction stir welding conditions. The material used was a partially rolled Al-Zn-Mg-Cu aluminum alloy. All test samples were immediately quenched after testing to freeze the deformation structure. Flow softening was observed at lower temperatures for all strain rates studied. At high temperatures, however, the flow softening was insignificant. The flow stress was observed to drop monotonically with increasing temperature and increase with increasing strain rate. The fast decay of flow stress with temperature was seen to occur at 525F. The flow stress could be 1 GPa at the high strain rate and low temperature in this study. The deformed samples were characterized using optical and electron microscopy to understand the microstructure evolution during deformation. The observed mechanical response of the material was discussed based on the microstructure observations.

11:10 AM
Hot Rolling Textures in Al Alloys: *J. Hirsch*[1]; [1]Hydro Deutschland GmbH, R&D, Bonn 53014 Germany

The microstructure and texture evolution during hot rolling of aluminium alloys is described and analyzed for industrial process parameters like single-stand or multi-stand hot rolling. Systematic variations in texture intensity occur which depend in a complex form on the combination of a number of process parameters like temperature, strain, strain rate, interstand time. Simulation tools have been developed and applied that can help to clarify the correlations and to describe them on the basis of the physical processes involved. The evolution of microstructure and flow stress in several non-heat-treatable Al alloys during high temperature deformation was analysed experimentally in laboratory tests. They are quantitatively described and used in hot rolling simulation models for the prediction of metallurgical effects like the amount of recrystallization, grain size and texture formation. For hot-line gauge recrystallization textures the cube texture is the dominant feature characteristic for most Al alloys and of major technical importance for the control of anisotropy. It is shown how quantitative prediction of texture formation during hot rolling can be applied in practice for on-line process control and off-line material optimization strategies.

11:35 AM
Parameters Affecting Strain Rate Sensitivity Index of Al-Mg Alloy: *Jyoti Mukhopadhyay*[1]; [1]Hindalco Industries, Ltd., R&D, Renukoot, Dt. Sonbhadra, Uttar Pradesh 231217 India

The values of strain rate sensitivity index (m) of Al-Mg alloys for two different grain sizes (16 & 25 µm) were determined under uniaxial tests at ambient as well at high temperatures in the range of 523-723 K and strain rates of 0.69 to 1.4 x 10-2 S-1. The maximum values of strain rate sensitivity for both grain sizes were found to occur at 723 K and at a strain rate of 1.4 x 10-3 S-1. At high temperatures these values were approximated to be around 0.34 and 0.3 respectively. They were strongly positive, whereas negative strain rate sensitivity values at ambient temperature were also observed for both grain sizes. The negative strain rate sensitivity index strongly suggests that dynamic strain ageing mechanism is operative in Al-Mg alloys. Measurements of strain rate sensitivity (m) as a function of strain and temperature were also evaluated. The value of (m) for a grain size of (16 µm) as compared to 26µm was found to be higher for all parameters.

11:55 AM
Grain Refinement in an Al-Li-Mg-Sc Alloy During Intense Plastic Straining at 300°C: *Rustam Kaibyshev*[1]; Ksenya Saytaeva[1]; Fanil Musin[2]; Yoshinobu Motohashi[2]; [1]Institute for Metals Superplasticity Problems, Khalturina, 39, Ufa, Bashkortostan 450001 Russia; [2]Ibaraki University, Rsrch. Ctr. for Superplastisity, Nakanarusawa-cho, 4-12-1, Hitachi, Ibaraki 316-8511 Japan

Microstructural evolution has bee studied in an Al-Mg-Li-Sc alloy with an initial grain size of ~60 µm during equal-channel angular extrusion at a temperature of 300°C and a strain rate of ~10^{-2} s^{-1}. It was established that the formation of ultrafine grains with an average size of ~0.9 µm takes place during intense plastic straining by subsequent evolution of microstructure via continuous dynamic recrystallization. At ε~1, the structure has become subdivided into bands of elongated subgrains alternating with areas of equiaxed subgrains. With increasing

strain the essentially equiaxed arrays of subgrains form and low angle boundaries gradually convert to true high-angle boundaries. After ε~4, the mixed arrays consisting of boundaries with low and high angle misorientation were observed. Fully recrystallized structure is evolved after a true strain of ~12. Mechanism of grain refinement is discussed.

International Symposium on Gamma Titanium Aluminides: Processing - IM, PM, and Sheet

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

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

Wednesday AM Room: 6F
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Paul A. McQuay, Howmet Corporation, Whitehall, MI 49461-1895 USA; Lee S. Semiatin, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA

8:30 AM Invited
Melting and Casting of Gamma Titanium Aluminide Ingots: *J. R. Wood*[1]; [1]Allvac, R&D, 20/20 Ashcraft Ave., PO Box 5030, Monroe, NC 28111 USA

Allvac operates two plasma arc melt (PAM) hearth furnaces which have been used over the years for the melting and casting of gamma titanium aluminide (TiAl) ingots. The PAM furnaces operate with He as the carrier gas and are capable of casting round ingots from 150 mm (6 in) to 760 mm (30 in) diameter and weighing from 140 kg (300 lb) to 6000 kg (13,600 lb). Input materials consist of titanium sponge, master alloys and other alloying elements which are precisely weighed, blended and pressed into compacts for charging into the furnace by a drum feeder. A variety of alloys have been made ranging from basic gamma TiAl chemistries to more complex chemistries for specific applications. A description of the melting process in each furnace and various alloys produced is presented herein.

9:00 AM Invited
Status and Prospects of Gamma TiAl Ingot Production: *Volker Guether*[1]; Anita Chatterjee[1]; Helmut Kettner[1]; [1]GfE Metalle und Materialien, Nuremberg 90431 Germany

The development of an industrial production process for homogeneous gamma-TiAl ingots has enabled processing industries to introduce final parts with excellent properties based on wrought materials. Since the first use of TiAl components in racing car engines in 2000, the demand for ingots increased remarkably. The paper describes the present status of ingot production with the focus on quality aspects and gives an outlook for the midterm future. Improved ingot manufacturing technologies will be directly adjusted to the specific materials requirements and widen the field of gamma-TiAl applications. Based on this and under the assumption of cost reductions of semi-finished material due to the effects of a mass production and a more efficient manufacturing procedures, wrought gamma-TiAl components are expected to enter service in gas turbines, aircraft engines and premier class vehicles within the next 4-6 years.

9:30 AM
Microsegregation in Major-Alloyed Gamma TiAl Based Alloys: *Ze-Wen Huang*[1]; Wayne Voice[2]; Paul Bowen[1]; [1]The University of Birmingham, Dept. of Metall. & Matls., Elms Rd., Edgbaston, Birmingham B15 2TT UK; [2]Rolls-Royce plc, PO Box 31, Derby DE24 8BJ UK

Major alloying of the Ti-Al binary system, by adding refractory metals, has been applied to raise the liquidus temperatures for improving high-temperature strength, creep resistance and oxidation resistance. However, these additions exacerbate the already severe segregation problems facing cast ?x-TiAl alloys, and may cause pronounced partitioning of heavy elements from light elements in dendrites. As a result, significant microsegregation occurs during solidification. This study assesses the degree and causes of microsegregation. Work focuses on 100-mm diameter, 50-kg ingots produced by both plasma arc cold-hearth remelting and vacuum arc remelting. 20-mm diameter casting bars produced by induction skull melting are also assessed. The effects of subsequent HIPping and heat treatment on microsegregation have also been studied in detail. The influence of such microsegregation on tensile behaviour and fatigue strength is also considered in this paper.

9:50 AM Invited
Powder Metallurgy Processing of Gamma Titanium Aluminide: *C. F. Yolton*[1]; Young-Won Kim[2]; Ulrike Habel[1]; [1]Crucible Research, 6003 Campbells Run Rd., Pittsburgh, PA 15205-1022 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA

As gamma titanium aluminide alloys transition from research and development into production, processing issues become increasingly important. Gas atomization of prealloyed powder followed by hot isostatic pressing (HIP) to full density is a viable approach for the production of forging and rolling performs, and as well as as-HIP components. A distinct advantage of the powder metallurgy (PM) production route is the uniform and fine microstructure in preforms/components of any size. The first part of this paper will describe gamma titanium aluminide powder production and processing with emphasis on powder characterization and powder cleanliness. Microstructures of consolidated material will also be discussed. Boron is a common alloying element in gamma titanium aluminide alloys. Observations on the effect of boron on microstructure evolution as a function of processing will be presented. The effect of heat treatment on boride and carbide distribution will also be discussed. Thermally induced porosity (TIP) is a phenomenon, which can occur in all inert gas atomized powders. The effect of HIP temperature and time combinations and post HIP heat treatment conditions on the microstructure and occurrence of TIP in gamma titanium aluminide powders will also be discussed.

10:20 AM Invited
Powder Production Techniques and PM Processing Routes for Gamma Titanium Aluminides: *Rainer Gerling*[1]; Frank Peter Schimansky[1]; Helmut Clemens[1]; [1]GKSS Forschungszentrum, Inst. for Matls. Rsrch., Max Planck Strasse 1, Geesthacht D 21502 Germany

As a consequence of the reactivity of the melt, only distinct techniques can be used for the production of high quality TiAl alloy powders. Such techniques are presented and properties of the resulting alloy powders with respect to process peculiarities are discussed. The use of TiAl alloy powders within the following processing routes is described. (i) Forging of HIP-compacted alloy powder at 850∞C and characterization of the submicron microstructure with respect to superplastic behaviour. (ii) Sheet rolling of HIP-compacted high Niobium containing TiAl alloy powder and tensile testing of these high strength sheets between RT and 1000∞C, (iii) Metal injection moulding of alloy powder <45 micrometer and discussion of the present achievable strength and impurity levels. With respect to the feasibility to produce large TiAl preforms of high chemical and microstructural homogeneity, the results of laboratory scale spray forming experiments are presented and the process related characteristics are discussed.

10:50 AM Invited
The Development of Sheet Gamma TiAl Technology Under the Enabling Propulsion Materials/High Speed Civil Transport (EPM/HSCT) Program: *Gopal Das*[1]; P. A. Bartolotta[2]; H. Kestler[3]; H. Clemens[4]; [1]Pratt & Whitney, 400 Main St., E. Hartford, CT 06108 USA; [2]NASA GRC, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [3]Plansee Aktiengesellschaft, Reutte/Tirol A-6600 Austria; [4]GKSS Research Center, Max-Planck Strasse 1, Geesthacht D-21502 Germany

The sheet gamma TiAl technology is one of several new and challenging technologies successfully developed under the NASA sponsored EPM/HSCT program. The task was to design and fabricate the divergent flap of the nozzle that would meet the overall weight goal of the HSCT engine. The designers selected gamma TiAl for its lightweight, high modulus, and elevated temperature properties to design the divergent flap of an extremely large exhaust nozzle required to reduce exhaust and noise pollution. This presentation will review diverse technologies that were developed to fabricate the nozzle subelement using sheet gamma TiAl. These include: production of large sheets, hot-die forming of sheets into corrugations, joining of sheets by brazing and diffusion bonding, EB welding, as well as by rivets, evaluation of microstructure and mechanical properties of sheets and joints, fabrication of a nozzle subelement involving brazing of large corrugations to face sheets, non-destructive evaluation of bond quality, and successful bend test of the subelement. The sheet gamma TiAl technology was successfully transferred to BFGoodrich for manufacturing of industrial scale components. Since the termination of the EPM/HSCT program, use of sheet gamma TiAl is being explored for aerospace applications and critical technologies are being developed to support their needs. These are: nozzles for gas turbine engines and

helicopters, and thermal protection systems for the reusable launch vehicles (RLV), to name a few. Additional technologies such as superplastic forming/diffusion bonding, laser joining and drilling, waterjet machining, and fabrication of honeycomb structures are being developed in order to support these activities.

11:20 AM

An Innovative Method for Manufacturing Gamma-TiAl Foil: *Stephen John Hales*[1]; Mohammad Saqib[2]; Joel Alexis Alexa[2]; [1]NASA Langley Research Center, Struct. & Matls., MS 188A, 2 W. Reid St., Hampton, VA 23681 USA; [2]Lockheed Martin Engineering & Science Company, c/o NASA-LaRC, MS 188A, 2 W. Reid St., Hampton, VA 23681 USA

The manufacture and entrance into service of thin gage (~ 0.010 in.) gamma-TiAl product has been hampered by the inherent low room temperature ductility of the material. At NASA-LaRC a new approach is being explored for the efficient manufacture of gamma-TiAl foil with improved ductility. The objective is to produce a very clean material (low interstitial content) with a highly refined, homogeneous microstructure placed in a fully lamellar condition. The processing route involves the use of RF plasma spray deposition of pre-alloyed powders, followed by consolidation via vacuum hot pressing and heat treatment. The approach takes advantage of a deposition process which includes no electrodes, no binders and high cooling rates. Results and discussion of the work performed to-date will be presented.

11:40 AM Invited

Low-Temperature Sheet Rolling of Gamma-TiAl+Alpha2-Ti3Al Alloys: Approach and Implementation: *Renat Imayev*[1]; Valery Imayev[1]; Andrey Kuznetsov[1]; Marat Shagiev[1]; Gennady Salishchev[1]; [1]Institute for Metals Superplasticity Problems, Khalturin str. 39, Ufa 450001 Russia

The present work proposes a new approach for producing sheet material from gamma-TiAl+alpha2-Ti3Al alloys. This approach includes correct choice of alloy composition, effective thermomechanical processing for producing the most workable sheet prematerial and subsequent sheet rolling below the eutectoid temperature under near-superplastic conditions. The proposed low-temperature sheet rolling process was successfully demonstrated for wrought ingot-metallurgy Ti-45.2Al-3.5(Nb,Cr,B) and Ti-44.2Al-3.5(Nb,Cr,B) alloys. As was shown for the Ti-45.2Al-3.5(Nb,Cr,B) alloy, the proposed process provided sheet with excellent and isotropic superplastic properties in the temperature range of 1000-1100∞C. Using a strain rate 10-3 s-1 elongation of up to 330-550% were measured with flow stress at 50% true strain ranging between 60-140 MPa. It is thought that the developed processing route is more cost-effective when compared with current used processing routes, which include sheet rolling above the eutectoid temperature.

12:10 PM

Equal Channel Angular Extrusion (ECAE) Processing of Titanium Aluminides for Microstructural Refinement and Mechanical Property Improvements: *Shankar M.L. Sastry*[1]; [1]Washington University, Mechl. Eng., CB 1185, One Brookings Dr., St. Louis, MO 63130 USA

ECAE process consists of extruding a well-lubricated billet through two intersecting channels of identical cross section. Deformation is achieved at the plane formed by the crossing of the channels through simple shear. A large and uniform strain intensity per pass can be obtained through this process without a reduction in the cross-sectional area of the billet. If the process is carried out at high temperatures at certain extrusion rates, grain refinement by dynamic recrystallization and/or spheroidization of lamellar microstructure can be produced. ECAE experiments were carried out on several gamma-based titanium aluminides in a vacuum of 10í5 Torr at 1000 and 1100°C at extrusion rates of 0.01 to 0.05 in/min. Grain refinement down to 1 mm was observed in all the ECAE processed alloys. ECAE processing results in a 25-50% increase in yield strength and 50-90% increase in ductility, 20-40% increase in fracture toughness. At temperatures and stresses where creep is predominantly controlled by dislocation glide and climb, the creep rates are not adversely affected by the fine grained microstructure produced by ECAE processing.

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

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

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

Wednesday AM Room: 8
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Dongliang Lin, Shanghai Jiao Tong University, Sch. of Matls. Sci. & Eng. & Open Lab. of Edu. Ministry of China for High Temp. Matls. & Tests, Shanghai 200030 China; Fritz Appel, GKSS Research Centre, Inst. for Matls. Rsrch., Geesthacht D-21502 Germany

8:30 AM Invited

Development, Processing and Applications of High Strength, High Ductility Iron Aluminides: *S. C. Deevi*[1]; R. S. Sundar[1]; S. Gedervanishvili[1]; [1]Philip Morris USA, Rsrch. Ctr., Richmond, VA 23234 USA

Iron Aluminides based on Fe-40Al have been investigated as possible replacements for a wide variety of structural and functional applications due to their low density, high strength, and excellent oxidation and corrosion resistance. Several approaches were considered to enhance the strength, creep resistance and rupture life of Fe-40Al alloys while maintaining or enhancing the ductility. In this paper, we present an overview of our approaches on enhancing the creep strength by mechanical alloying, solid solution and precipitation strengthening mechanisms. In addition, we will discuss the cold rolling and powder processing characteristics of iron aluminides, and the advantages and applications offered by powder processing approaches towards commercialization of iron aluminides.

8:50 AM Invited

Strain-Induced Ferromagnetism in Plastically-Strained FeAl Single Crystals: *I. Baker*[1]; D. Wu[1]; P. R. Munroe[2]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755 USA; [2]University of New South Wales, Electron Microscope Unit, Sydney, NSW 2052 Australia

An overview of plastic strain-induced ferromagnetism in FeAl will be presented. Recent research has indicated that the effect arises mostly from the generation of anti-phase boundary (APB) tubes, where Fe atoms can have ≥3 like nearest neighbors (NNs). The resulting saturation magnetization, Ms, depends on both the Fe:Al ratio and the degree of deformation. A quantitative mode will be presented that describes the effects of both of these parameters in terms of the local environment theory applied to the atoms in APB tubes. The behavior of ternary alloys will be explained by considering the site preferences of ternary atoms on the FeAl sublattices, which change the probabilities of a Fe atom having like NNs in the APB tubes. Transmission electron microscopy observations of APB tubes in lightly-strained FeAl single crystals will be presented, and the annealing out of APB tubes and the associated activation energy will be discussed. Research sponsored by National Science Foundation through grant DMR 9973977.

9:10 AM
Effects of Lattice Vacancies on the Phonon DOS of FeAl: *Tabitha Liana Swan-Wood*[1]; [1]California Institute of Technology, Matls. Sci., MS 138-78, Pasadena, CA 91125 USA

FeAl alloys can have vacancy concentrations as high as 3-4%. The equilibrium thermal vacancy concentration is given by c(T)=a*exp(-E/kT) E being the energy of formation and a=exp[S(vib)/k] with S(vib) the vibrational entropy of formation. Prior experiments show S(vib) is between 5 and 6 k_B/vacancy. We expected vibrational entropy to be a significant component of the thermodynamics of vacancy formation in FeAl. A sample with 2% vacancies would have 16% of atoms with first-nearest neighbor vacancies, so we expected measurable changes in its phonon spectrum. Inelastic spectra were measured on LRMECS at IPNS from three Fe-50 at.% Al samples with vacancy concentrations approximately 0 to 2%. The phonon modes shifted slightly with vacancy concentration. Nevertheless, the data do not show any significant change in vibrational entropy between concentrations.

9:25 AM Invited
Interpretation of the Electrical Properties of Fe-Al Alloys from Electronic Structure Calculations: *P. Jena*[1]; S. C. Deevi[2]; G. P. Das[1]; B. K. Rao[1]; A. C. Lilly[2]; [1]Virginia Commonwealth University, Dept. of Physics, Richmond, VA 23284 USA; [2]Philip Morris USA, RD&E Ctr., Richmond, VA 23234 USA

Lilly et al (Mat. Sci. Engg. A258, p.42, 1998) have shown that the electrical resistivity of Fe-Al alloys increases from 0 at.% to about 33 at.% after which there is a steep decrease with further increase of Al concentration. A qualitative explanation was provided based on the phenomenological s-s and s-d scattering theory of Mott-Jones for AB alloys. In this paper, we carried out a systematic theoretical investigation of the electronic, magnetic, and cohesive properties of Fe1-XAlX alloys by successively replacing Fe atoms by Al atoms and studying the electronic structure within the density functional theory and generalized gradient approximation for exchange and correlation. We have shown that the stoichiometric FeAl exists in two nearly degenerate statesña non-magnetic and a ferromagnetic state, while Fe3Al is clearly ferromagnetic. The bonding between Fe and Al atoms is dominated by nearest neighbor interaction including hybridization of Fe 3d with Al 3p states. With increase of Al concentration, the total density of states at the Fermi energy increases, reaches a peak around X0.33, and then decreases. This behavior is very similar to the concentration dependence of electrical resistivity in Fe1-XAlX suggesting that the resistivity anomaly has an electronic origin. In addition, these results are consistent with model calculations where atomic clusters are used as a model of the bulk. In this paper, we review the electrical resistivities of Fe1-XAlX alloys and present our electronic structure calculations carried out during the last four years.

9:45 AM
Periodicities of Chemical Environments in Fe3Al Measured by Mossbauer Diffraction: *Jiao Y.Y. Lin*[1]; Ryan Douglas Monson[1]; Brent Fultz[1]; [1]California Institute of Technology, Matls. Sci., 1200 E California Blvd., MC 138-78, Pasadena, CA 91125 USA

Mossbauer diffractometry combines the capability of Mossbauer spectrometry to distinguish local chemical environments with the capability of diffractometry to measure long-range order (LRO) in materials. The energy spectra of intensities of fundamental and superlattice Bragg diffractions in partially-ordered Fe3Al were measured and compared to theoretical calculations. The energy spectra gave the expected results that Fe atoms with 4 Al first-nearest neighbor (1nn) are arranged as a simple cubic lattice, while Fe atoms with 0 Al 1nn atoms have an fcc periodicity. More interestingly, Fe atoms with 3 Al 1nn atoms have a simple cubic LRO, similar to that of Fe atoms with 4 Al 1nn atoms. This unexpected LRO was related to distributions of antisite defects.

10:00 AM Invited
Composite Based on Iron Aluminide Intermetallic Alloy and CrMo Steel: *Shuji Hanada*[1]; *Naoya Masahashi*[1]; [1]Tohoku University, Inst. for Matls. Rsrch., Katahira 2-1-1, Aoba-ku, Sendai, Miyagi 980-8577 Japan

Composite of iron aluminide intermetallic alloy and CrMo steel was prepared by solid state bonding to improve corrosion resistance of the steel. The microstructure observation reveals that a sound joint is achieved without producing any defects and columnar grains are evolved towards the steel matrix from the joint interface bonded at high temperatures above A3. Composition changes continuously near the joint interface, and the interdiffusion coefficient of Al depends on the content of Cr. The formation mechanism of the columnar microstructure is explained by a nucleation at the joint interface caused by Al diffusion to stabilize α, followed by grain growth to steel side. The composite demonstrates high bonding strength due to a sufficient interdiffusion between the constituents. A sound composite is also fabricated by clad-rolling. These results suggest that the composite has a potential for corrosion-resistant applications.

10:20 AM Break

10:35 AM Invited
Development of High Strength High Ductility and High Temperature Iron Aluminide: *David G. Morris*[1]; Maria A. MuÕoz-Morris[1]; J. Chao[1]; Carmen Garcia Oca[1]; [1]CENIM, CSIC, Dept. of Physl. Metall., Avenida Gregorio del Amo 8, Madrid E-28040 Spain

Significant research activities over the last ten years have led to the development of a high strength and high ductility Fe-40Al alloy produced by mechanical alloying of the intermetallic with dispersed oxides. Strength and ductility and high temperature behaviour of this material are analysed to deduce the important microstructural features that contribute to each aspect of behaviour. High strength is seen to be a consequence of solution hardening, particle strengthening, and grain size hardening, in that order of importance. High ductility is seen to depend mostly on a fine grain size which, together with dispersed particles, ensures slip homogenisation and delays failure crack nucleation to high strains. Strength at high temperature falls because of rapid diffusion in the open bcc base lattice. Ways for improving high temperature strength are based on including large volume fractions of rather coarse and stable second phase particles.

10:55 AM Invited
Thermal-Cycling Deformation of Superplastic Coarse-Grained Fe-24.5%Al-1.4%Ti alloy: *Jinn P. Chu*[1]; C. L. Chiang[1]; H. Y. Yasuda[2]; Y. Umakoshi[2]; K. Inoue[3]; T. Mahalingam[4]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2, Pei-Ning Rd., Keelung 20224 Taiwan; [2]Osaka University, Dept. of Matls. Sci. & Eng., Osaka 565-0871 Japan; [3]University of Washington, Dept. of Matls. Sci. & Eng., Seattle, WA 98195 USA; [4]Alagappa University, Dept. of Physics, Karaikudi 630 003 India

Many studies have demonstrated that coarse-grained Fe-Al based alloys show all the deformation characteristics that conventional fine-grained superplastic materials possess. Using an electron backscattered diffraction technique, we have identified several important crystallographic features of a superplastic coarse-grained Fe-27at.%Al alloy. As a result of dynamic recovery and recrystallization during deformation, major microstructural evolutions are summarized as a sequence of (1) subgrain-boundary formation at 600°C, (2) grain-boundary migration at 700°C, (3) formation of new grains resulting in grain refinement and hence greater superplastic elongation at 800°C, and (4) growth of recrystallized grains at 850°C and above. In the present study, effects of thermal-cycling deformation on superplastic properties of coarse-grained Fe-24.5%Al-1.4%Ti alloy have been examined in air under an initial strain rate of $1x10^{-4}$ s^{-1}. Thermal cycling deformation between 800-850°C results in refined grains as a dominant structure and evidently improves the superplastic elongation to 391%.

11:15 AM Invited
Microscopic and Macroscopic Deformation Observations in FeAl and Fe3Al Alloys: *Bimal Kad*[1]; Joe Horton[2]; Chain T. Liu[2]; [1]University of California-San Diego, Structl. Eng., 409 University Ctr., MC-0085, La Jolla, CA 92093-0085 USA; [2]Oak Ridge National Laboratory, M&C Div., MS-6115, Bldg. 4500S, PO Box 2008, Oak Ridge, TN 37831-6115 USA

Macroscopic texture measurements of FeAl, Fe_3Al-based alloys deformed at 925<T<1325K in the B2 structure regime by rolling, forging and extrusion, at deformation rates of 10^{-4}-10^{-1} sec^{-1}, agree well with the numerically predicted textures for the <111>{110}+<110>{112} slip system activation for each of the deformation histories. These results appear to be in disagreement with microscopic TEM observations, that overwhelmingly support the activation of <100>{011} and <100>{001} slip systems at high temperature. We revisit the issue of TEM observations within the framework of a deformation experiment in the Gleeble apparatus, where the high temperature deformation sub-structure is quenched-in by imposing cooling rates of about 10^3 $K.sec^{-1}$, thereby minimizing post deformation dislocation reorganization. TEM studies reveal that such quenched-in substructure is indeed dominated by <111> and not <100> dislocations. Furthermore, the <111> to <100> thermal reorganization is quite rapid, as observed via short annealing treatments, and this propensity increases with aluminum content.

11:30 AM Invited
Microstructures and Mechanical Properties of Fe-Al-C and Fe-Al-M-C (M = Ti, V, Nb, Ta) Alloys: *AndrÈ Schneider*[1]; Ladislav Falat[1]; Gerhard Sauthoff[1]; Georg Frommeyer[1]; [1]Max-Planck-Institut

f¸r Eisenforschung GmbH, Matls. Tech., Max-Planck-Str. 1, D¸sseldorf 40237 Germany

This paper presents results on constitution, microstructure and mechanical properties of a variety of Fe_3Al-based alloys. Alloys based on the systems Fe-Al-C and Fe-Al-M-C (M = Ti, V, Nb, Ta) with strengthening carbides and Laves phase were investigated. The Fe-Al-C materials contain 23 to 29 at% Al and 1 to 3 at% C. The Fe-Al-M-C alloys contain 26 at% Al, 2 at% Ti, V, Nb, or Ta, and 1 at% C, respectively. The alloys were processed by vacuum induction melting and cast into Cu-moulds. The alloys were investigated in the as-cast state and after various heat treatments. For evaluating the mechanical properties as a function of the temperature, compression tests were performed. Microstructural analysis was performed by means of light optical microscopy (LOM), scanning electron microscopy (SEM), electron probe micro-analysis (EPMA), and X-ray diffraction analysis (XRD). The experimentally determined phase equilibria are compared with thermodynamic calculations using Thermo-Calc.

11:50 AM

Improvement of the Creep Strength of FeAl-Based Alloys: *Wei-Jun Zhang*[1]; R. S. Sundar[1]; S. C. Deevi[1]; [1]Philip Morris USA, PD&T, PO Box 26603, Richmond, VA 23261 USA

Great attention has been paid to the environmental embrittleness, vacancy hardening and corrosion behavior in FeAl-based alloys during the last decade. However, the creep behavior of FeAl has not been well understood as yet. The relatively poor creep strength of FeAl limits the wide spread applications of FeAl alloys. In this paper, we discuss the major factors leading to the poor creep resistance of FeAl in terms of high diffusional coefficient, low activation energy, high dislocation mobility, high vacancy concentration and microstructural instability. We will demonstrate that creep resistance can be improved through precipitation hardening and solid solution strengthening. However, more studies are needed to enhance the microstructural stability of FeAl alloys during long-term service.

12:05 PM

Reactive Thermomechanical Processing of Ni3Al and FeAl: *K. Morsi*[1]; S. O. Moussa[1]; J. Wall[1]; J. Rodriguez[1]; [1]University of Missouri, Mechl. & Aeros. Eng., E3411 Eng. Bldg. E., Columbia, MO 65211 USA

Nickel aluminides (Ni3Al) and Iron aluminides (FeAl) are intermetallics of significant technological importance. Major drawbacks, which have so far restricted the use of these materials, are the high-energy usage in synthesis and processing and difficulty in fabrication into the final component shape. The proposed work explores a new near net shape, low-energy approach which can be used to process a range of important aluminide intermetallics and their composites (e.g. titanium aluminides, nickel aluminides, iron aluminides and niobium aluminides), with wide ranging applications. The work discusses the application of thermomechanical processing (extrusion & forging) during the high temperatures achieved in reaction synthesis to form and simultaneously shape Ni3Al and FeAl aluminide intermetallics. The effect of varying processing parameters on the developed microstructure and properties is presented. Results confirm the feasibility of this new approach to process these materials at operating temperatures ~400 C lower than conventionally used in the extrusion and forging of these materials with potentially considerable energy and cost savings.

International Symposium on Structures and Properties of Nanocrystalline Materials: Microstructure and Properties

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

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

Wednesday AM Room: 14B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Ian Baker, Dartmouth College, Thayer Sch. of Eng., Hanover, NH 03755 USA; Chandra S. Pande, Naval Research Laboratory, Div. of Matls. Sci. & Tech., Washington, DC 20375 USA

8:30 AM

An Electron Microscopy Study of the Structure and Deformation Behavior of Electrodeposited Nanocrystalline Nickel Alloys: *Sharvan Kumar*[1]; Subra Suresh[2]; Matthew F. Chisholm[3]; Joe A. Horton[4]; Ping Wang[1]; [1]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA; [2]Massachusetts Institute of Technology, DMSE, Cambridge, MA 02139 USA; [3]Oak Ridge National Laboratory, Solid State Div., Oak Ridge, TN 37831 USA; [4]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

The deformation of electrodeposited nanocrystalline nickel with a mean grain size in the range of 30-40 nm and a columnar grain morphology with column lengths being anywhere from 3 times to 8 times the in-plane grain diameter was studied by ex-situ and in-situ deformation experiments. Results of the in-situ experiments confirmed the presence of extensive dislocation activity at the crack tip in these specimens. Voids form in the region ahead of the crack tip at grain boundaries and triple junctions and partially relieve the constraints on the grain, permitting dislocation processes to occur more readily. Examination of the fracture surfaces of such specimens after the test in an SEM confirms failure by dimpled rupture. The scale of these dimples is significantly larger than the average grain size and fracture in the bulk does not appear to propagate along grain boundaries in this material.

9:00 AM

Analysis of Misfit Dislocations in Epitaxial Ni/Cu Bilayers: *David Mitlin*[1]; Amit Misra[1]; Richard G. Hoagland[1]; Mike Nastasi[1]; Harriet Kung[1]; John P. Hirth[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-8, Los Alamos, NM 87545 USA

Understanding bilayer behavior is a critical step in the successful development of ultra-high hardness nano-layer composites. We have analyzed misfit dislocations at the interface of Ni/Cu bilayers using plan-view and cross-sectional transmission electron microscopy (TEM). The bilayers consisted of varying thickness of Ni (5-1000 angstroms) deposited on 1000 angstroms of Cu. Conventional g-b analysis was used to determine the types of dislocations present at the interface, while high-resolution TEM performed on cross-sectional samples was used to analyze the interface in more detail. It is demonstrated that there is minimum Ni thickness below which interface dislocations do not nucleate. As the Ni layer thickness is increased, the dislocations spacing decreases. Agreements and discrepancies of the experimental results with the existing interface dislocation theory are discussed.

9:20 AM

Microstructures and Mechanical Properties of Nanoscale Copper-304 Stainless Steel Multilayers Synthesized by Magnetron Sputtering: *Xinghang Zhang*[1]; Amit Misra[1]; Harriet Kung[1]; John D. Embury[1]; Michael A. Nastasi[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MST-8, MS G755, Los Alamos, NM 87545 USA

Copper_304 stainless steel (SS) multilayers synthesized by magnetron sputtering were studied systematically in an attempt to understand the role of metastable crystal structures on the strength of nanolayered composite. The composites were composed of Cu and SS layers of equal thickness, with the layer thickness varying from 1 nm to 500 nm. Transmission electron microscopy analysis shows that the SS layers with thickness of greater than 10 nm have a mixture of metastable bcc and equilibrium fcc phases. The co-existence of the two phases offers a special opportunity to study stabilization mechanisms of unusual phases at the nanometer scale. Nanoindentation measurements show that the hardness of these nanolayered materials increases with the decreasing layer thickness, reaching a maximum value of about 5.5 GPa at the layer thickness of 5 nm. A decrease in hardness was noted with decreasing layer thickness below 5 nm. The effects of layer thickness, interfaces and the formation of metastable bcc phase in the SS layer on strength of Cu-SS multilayer are discussed.

9:40 AM

Structure-Property Studies of Bias Sputter Deposited Cu1-xNbx Alloys: *Guoyi Tang*[1]; [1]North Carolina State University, Matls. Sci. & Eng., CB 7907, Raleigh, NC 27695-7907 USA

Nanocrystalline, non-equilibrium Cu1-xNbx (x = 2 to 75) alloys have been deposited onto glass and Cu substrates using dual magnetron sputter deposition. In addition to the usual sputter deposition parameters of cathode power and Ar working pressure, these films were deposited under a range of temperature and substrate bias (low energy Ar+ ion bombardment) conditions. X-ray diffraction, scanning electron microscopy, transmission electron microscopy, and atomic force microscopy (AFM) were used to characterize film microstructure, tex-

ture, and surface morphology. The film microstructures consist of nanoscale, discrete Nb particles in a Cu matrix. Films with Nb concentration >5 atomic percent have strong [111] type textures. Increasing Nb concentrations altered the surface morphologies from irregular (<5% Nb) to spherical (>10% Nb).

10:00 AM Break

10:20 AM
Formation and Characterization of CuOx Nanowire Arrays: Chen Jin-Ming[1]; [1]Industrial Technology Research Institute, Matl. Rsrch. Labs., 240r, Bldg. 77, Chutung 310 Taiwan

We reported a novel method for preparing one-dimensional configuration and well-ordered nanowires of copper oxide by using electrodeposition followed by oxidation treatment. The morphology of CuOx nanowires were examined by scanning electron microscopy and transmission electron microscopy and showed that the diameters of nanowires were in range of 10-50 nm. The result of X-ray photoelectron spectroscopy indicated the presence of two stages of copper oxides, CuO and Cu2O in the nanowire array. The analysis results suggest that the CuOx nanowires are promising candidates for field-emission devices and Li-ion anode materials.

10:40 AM
The Interaction Between Nanocrystalline Grains in Cryomilled Nanocrystalline Al-Mg Alloys: *Zonghoon Lee*[1]; Bing Q. Han[2]; Farghalli A. Mohamed[2]; Enrique J. Lavernia[2]; Steven R. Nutt[1]; [1]University of Southern California, Matls. Sci., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA; [2]University of California-Irvine, Dept. of Cheml. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

The deformation of bulk nanocrystalline Al-Mg alloys was investigated using transmission electron microscopy and high-resolution electron microscopy. Grain refinement was achieved by cryomilling of elemental powders, and powders were consolidated by hot isostatic pressing (HIP) and extrusion to produce bulk nanocrystalline Al-Mg alloys. The microstructure of cryomilled Al-Mg alloys consisted of equiaxed and elongated grains, which were elongated along the extrusion direction. The nanocrystalline Al-Mg alloys exhibited unusual deformation characteristics involving complex interactions between nanocrystalline grains in contrast to a conventional Al-Mg alloy. Elongated grains were comprised of dislocation cells, sub-grains, and slip bands. Investigation of bulk tensile and compression fracture specimens revealed unusual failure mechanisms and interactions between nanocrystalline grains. Tensile behavior was characterized by high yield strength, high ductility, and low strain hardening. After the yield point, the alloy exhibited nearly perfectly plastic behavior and low strain hardening.

11:00 AM
Structure, Mechanical and Transport Properties of Nanocrystalline Nb and Ti Thin Films: *Rajarshi Banerjee*[1]; Evan Sperling[1]; Gregory B. Thompson[1]; Parinda Vasa[2]; Pushan Ayyub[2]; Hamish Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Tata Institute of Fundamental Research, Condensed Matter Physics & Matls. Sci., Homi Bhabha Rd., Mumbai, Maharashtra 400005 India

Nanocrystalline thin films of Nb and Ti with varying grain sizes have been deposited using high pressure magnetron sputtering. The structure of these films as a function of grain size is being characterized using x-ray diffraction and TEM. Initial results suggest that in case of Nb, there is a significant lattice expansion with reduction in grain size while in case of Ti, the c/a ratio changes with the grain size. Nanoindentation experiments are being conducted to evaluate the influence of these structural changes on the modulus and hardness of these nanocrystalline films. In addition to the mechanical properties, the electrical transport properties of these films are also being investigated. Thus, a systematic investigation of the influence of grain size and structure on the superconducting properties of the Nb and Ti films is being carried out through the measurement of the ac-Meissner effect using a SQUID magnetometer and the results will be presented in this paper.

11:20 AM
DC Magnetron Sputtered Fe Films for Growth of C Nanotubes: *Guoyi Tang*[1]; Yunyu Wang[2]; Robert J. Nemanich[2]; J. M. Rigsbee[1]; [1]North Carolina State University, Matls. Sci. & Eng., CB 7907, Raleigh, NC 27695-7907 USA; [2]North Carolina State University, Dept. of Physics, Raleigh, NC 27695 USA

Thin films of Fe have been DC magnetron sputter deposited onto Si (100) wafers using cathode powers from 30 to 150Watts and times up to 60 minutes. These processes led to four iron thin films with thicknesses between 40 and 90 nm. X-ray diffraction, transmission electron microscopy, scanning electron microscopy and atomic force microscopy were used to characterize film structure and surface morphology. It is shown that the surface morphology and roughness of the iron films varied with sputter cathode power density. Above 70 Watts cathode power, the film surfaces consisted of nearly spherical grains. The ability of these films to nucleate and grow carbon nanotubes were found to be strongly influenced by surface morphology and nanotube growth was optimum at 70 Watts. The results for film and carbon nanotube growth will be presented.

Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging: Mechanical Properties and Fatigue Behavior

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Srini Chada, Motorola, Department APTC, Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Jud Ready, MicroCoating Technologies, Atlanta, GA 30341 USA; Jin Yu, KAIST, Center for Electronic Packaging Materials 305-701 Korea

Wednesday AM Room: 15B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Kwang-Lung Lin, National Cheng Kung University, Dept. of Matls. Sci. & Eng., Tainan 70101 Taiwan; K. N. Subramanian, Michigan State University, Cheml. Eng. & Matl. Sci., E. Lansing, MI 48824 USA

8:30 AM Invited
Compression Stress Strain Behavior of Sn-Ag-Cu (Cu = 0.2, 0.6, and 0.7): *Paul T. Vianco*[1]; Jerome A. Rejent[1]; Joseph Martin[1]; [1]Sandia National Laboratories, MS0889, PO Box 5800, Albuquerque, NM 87185-0889 USA

Several Sn-Ag-Cu Pb-free solders have been identified to replace Sn-Pb eutectic solder in reflow process applications. The compositions differ by a few tenths of a percent of Cu. The sensitivity of the mechanical properties was investigated for Sn-Ag-Cu solders having nominal Cu contents of 0.2, 0.6 and 0.7 wt.% Cu. Compression stress strain tests were performed to measure the yield stress of the solders as a function of test temperature and strain rate. The test temperatures were 25C, 25C, 75C, 125C, and 160C. Two strain rates were used: 0.00043 1/s and 0.000082 1/s. Samples were tested in the as-cast condition and following an initial annealing treatment at 125C for 24 hours. The microstructure of the solders was surveilled after testing. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Dept. of Energy under contract DE-AC04-94AL85000.

8:55 AM
A Potential Drop-in-Replacement for Eutectic Sn-Pb Solderñ The Sn-Zn-Ag-Al-Ga Solder: *Kwang-Lung Lin*[1]; Kang-I Chen[1]; Po-Cheng Shi[1]; [1]National Cheng Kung University, Matls. Sci. & Eng., 1 Ta-Hsuey Rd., Tainan 701 Taiwan

Sn-Zn alloy exhibits the closest eutectic temperature to the eutectic Sn-Pb solder, although it suffers easy oxidation characteristic. A novel alloy basing on the Sn-Zn eutectic alloy was developed and found promising as a drop-in-replacement for the eutectic Sn-Pb solder. This alloy, consisting of Sn-Zn-Ag-Al-Ga combination, was developed showing promising properties. DSC (Differential Scanning Calorimetry) investigation reveals that the investigated solder exhibits eutectic temperature of around 199°C. The TGA (thermal gravimetric analysis) study shows it attains much better oxidation resistance than the eutectic Sn-Pb solder at 250°C. Stress-strain curve indicates that this solder also possesses a greater UTS (ultimate tensile strength) and a better ductility than the eutectic Sn-Pb solder. A wetting time of less than one second was found for this solder with a suitable flux. Ga was seen to enhance wetting behavior. Cost of this solder was also estimated and showing comparable with that of other solders.

9:15 AM

Effect of Sb Addition on Microstructure and Shear Strength of Sn-Ag Solder Joints: *Hwa-Teng Lee*[1]; *Chuan-Lien Yang*[1]; Ming-Hung Chen[1]; Cheng-Shyan Li[1]; [1]National Cheng Kung University, Mechl. Eng. Dept., No. 1, Dashiue Rd., Tainan 701 Taiwan

The effect of Sb addition on microstructure, intermetallic compound (IMC) and mechanical properties of Sn-Ag solder joints is investigated. The compositions of selected solders are Sn2.58Ag, Sn2.82Ag1.75Sb, Sn2.87Ag4.75Sb and Sn2.7Ag8.78Sb. Experimental results show that most of the added Sb are solved in beta-Sn matrix, and the rest react with the Ag3Sn to form Ag3(Sn,Sb) phase, which contributes to suppress the coarsening of Ag3Sn phase. SbSn phase can be observed in beta-Sn matrix as the Sb addition exceeds 4.75% and remains stable during the thermal storage test. The solder microhardness increases with increasing Sb. And the growth rate of interfacial IMC layer decreases as Sb addition increases. EPMA analysis indicates there are some Sb diffusing into the interfacial IMC layer. Shear strength of solder joints are raised by adding Sb. The shear strength by as-soldered condition are 27.8MPa(0%Sb), 29MPa(1.75%Sb), 30.4MPa(4.75% Sb) and 43.4MPa(8.78%Sb) respectively.

9:35 AM

Assessment of Microstructural Evolution and Change in Mechanical Properties of Sn-Pb and Pb-Free Chip ResistorsñSolder Joints in Response to Aging and Accelerated Thermal Cycling: Adam R. Zbrzezny[1]; Polina Snugovsky[2]; *Doug D. Perovic*[1]; [1]University of Toronto, Matls. Sci. & Eng., Wallberg Bldg., 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]Celestica, Matls. Lab., 844 Don Mills Rd., Toronto, Ontario M3C 1V7 Canada

Aging and Accelerated Thermal Cycling (ATC) are currently being performed on 2512 chip resistors assembled with the Sn-Pb eutectic and Sn95.5Ag3.8Cu0.7Cu solders. The test vehicles were divided into 12 cells comprised of 5 boards per cell, 16 components per board. The boards were finished with immersion Ag, ENIG, and HASL, and components terminations were 100% Sn and SnPb. The microstructure is being investigated by cross-sectioning with subsequent optical microscopy and SEM/EDX. The mechanical properties are being assessed by the shear tests. The cross-sections and shear tests are being performed every 500 hrs of aging and 250 cycles of ATC. The results from this study, which will be presented, will allow for a direct comparison between the solder joints of various metallurgies. In addition, the microstructure evolution and its influence on the mechanical properties will be studied in detail.

9:55 AM

Isothermal Aging of Near-Eutectic Sn-Ag-Cu Solder Alloys and its Effect on Electrical Resistivity: *Bruce Allan Cook*[1]; Iver E. Anderson[1]; Joel H. Harringa[1]; Robert L. Terpstra[1]; Sung K. Kang[2]; [1]Iowa State University, Ames Lab., Metal & Ceram. Scis. Prog., 47 Wilhelm, Ames, IA 50011-3020 USA; [2]IBM T. J. Watson Research Center, Yorktown Heights, NY 10598 USA

Solder joints were prepared from seven near-eutectic Sn-based alloys and characterized for electrical resistivity after 100 and 1000 hours of isothermal aging at 423K. The solder joint samples were prepared by hand soldering to copper substrates and the post-heat treatment resistivity was measured at room temperature in a specially-designed 4-point fixture. Compositions tested included Sn-3.5Ag, Sn-3.7Ag-0.9Cu, Sn-3.0Ag-0.5Cu, Sn-3.6Ag-1.0Cu, and Sn-3.9Ag-0.6Cu; moreover, the effect of minor addition of a fourth element, designed to improve high temperature shear strength, was also evaluated in the compositions Sn-3.7Ag-0.6Cu-0.3Co and Sn-3.7Ag-0.7Cu-0.2Fe. The observed changes in electrical transport are discussed in terms of microstructural coarsening, diffusional transport from the substrate, and nucleation of precipitate phases. Results are compared with previous studies conducted on cross-shaped specimens formed by joining two ìL-shapedî copper coupons.

10:15 AM

Influence of Adding High Sb Content into Sn-Ag Solder Joints on Microstructure and Shear Strength: *Hwa-Teng Lee*[1]; Chuan-Lien Yang[1]; [1]National Cheng Kung University, Mechl. Eng. Dept., Tainan 70101 Taiwan

The study investigates the effect of adding Sb into Sn-Ag solder joints on microstructure, intermetallic compound (IMC) and mechanical properties. The compositions of selected solders were Sn2.58Ag, Sn2.82Ag1.75Sb, Sn2.87Ag4.75Sb and Sn2.7Ag8.78Sb. FR-4 PCB is used as the substrate material and the single-lap specimen form is selected for shear test. And thermal storage test is also performed after soldering to estimate the varity of microstructure and shear strength. Experimental results show that most of added Sb are solved in ,-Sn matrix, and the rest react with the Ag3Sn to form Â-Ag3(Sn,Sb) phase, which contributes to suppress the coarseness of Ag3Sn phase. SbSn phase can be observed in ,-Sn matrix as the Sb addition exceeds 4.75%, and the coarseness of SbSn phase is not obvious during thermal storage. The solder microhardness increases with increasing Sb addition. And the growth rate of interfacial IMC layer decreases as Sb addition increases. EPMA analysis indicates there are some Sb atoms diffusing in to the interfacial IMC layer. Shear strength of solder joints are improved as adding Sb. The shear strength of as-soldered condition are 27.8 (0%Sb), 29 (1.75%Sb), 30.4 MPa(4.75% Sb) and 43.4(8.78%Sb) MPa respectively.

10:35 AM Break

10:50 AM Invited

Temperature Effects on Low Cycle Fatigue Behavior of Sn/3.5Ag/0.75Cu and 63Sn/37Pb Solder Joints: Tae-Sang Park[1]; *Soon-Bok Lee*[1]; [1]CARE Electronic Packing Laboratory, Dept. of Mechl. Eng., KAIST, 373-Kusong-dong, Yusong-gu, Daejeon 305-701 Korea

The demand of lead-free solder and high-density interconnection technology in modern microelectronic packaging has been increased. Due to this high degree of integration and component density, the modern advanced SMC assembly has imposed more stringent reliability requirements on packaging design. Service failure of solder interconnections generally arises due to thermomechanical fatigue (TMF) and usually occurs within the solder itself since it is more soft parts of the joint. The combination of temperature fluctuations, either due to power switching or the external environment, and materials in the joint which possesses different coefficients of thermal expansion, produce substantial cyclic strains within the solder. Therefore in most applications, solder joints are under thermo-mechanical loading condition. To understand this thermo-mechanical material behavior of solder joints, isothermal cyclic fatigue tests at several temperature levels are essential. In the present work, low cycle isothermal mechanical fatigue tests of lead-free solder (Sn/3.5Ag/0.75Cu) and lead-containing solder (63Sn/37Pb) were carried out in several temperature levels. The mechanical fatigue tests were performed under conditions of shear loading at the temperature of 25°C, 70°C, and 100°C. Constant displacement tests are performed using a computer controlled micro-mechanical test apparatus. The low cycle fatigue behavior of these solder alloys was found to be strongly dependents on test temperature. Failure patterns of the fatigue tests are observed and discussed. As a fatigue model, the Morrow energy model and Coffin-Manson model were examined. The material parameters in these models are found to be a function of temperature rather than constants.

11:15 AM

Effects of Mechanical Deformation and Annealing on the Microstructure and Hardness of Pb-Free Solders: *Sung K. Kang*[1]; Paul Lauro[1]; Won Kyoung Choi[1]; Da-Yuan Shih[1]; [1]IBM T. J. Watson Research Center, PO Box 218, Yorktown Heights, NY 10598 USA

The microstructure-property relations of several Pb-free solders have been investigated in order to understand the microstructure changes during thermal and mechanical processes of Pb-free solder joints. Pb-free solder alloys investigated include pure Sn, Sn-0.7%Cu, Sn-3.5%Ag and Sn-3.8%Ag-0.7%Cu (in weight). To reproduce a typical microstructure observed in solder joints, the cooling rate, ingot size and reflow conditions of cast alloys are carefully controlled. The cast alloy pellets are subjected to compressive deformation up to 50% and annealing at 150∞C, 48 h. The microstructure of Pb-free solders is evaluated as a function of alloy composition, plastic deformation and annealing. The changes in mechanical property are measured by microhardness test. The work hardening in Sn-based alloys is found to increase as the amount of alloying elements increases. The change in microhardness upon deformation and annealing has been correlated with the microstructural changes, such as recrystallization or grain growth in Pb-free solder alloys.

11:35 AM

Lead (Pb)-Free Ceramic Ball Grid Array (CBGA): Thermo-Mechanical Fatigue Reliability: *Mukta Farooq*[1]; Charles Goldsmith[1]; Ray Jackson[1]; Gregory Martin[1]; [1]IBM Microelectronics, 2070 Rte. 52, MS 87P, Hopewell Junction, NY 12533 USA

Flip-chip carriers have become the preferred solution for high-performance ASIC and microprocessor devices. Typically these are packaged in organic or ceramic Ball Grid Array (BGA) packages which cover a wide range of package I/O capabilities required for high-performance devices, typically between 300 to more than 1600 I/O. Recently, there has been a move towards Pb-free solders, as replacement alloys for standard eutectic Sn/Pb and other Pb-based BGAís. The leading solder that has emerged is an Sn/Ag/Cu (SAC) alloy. One of the primary issues with changing solders is the reliability of the joints when subjected to Thermo-Mechanical Fatigue (TMF). With the need

to shrink the I/O pitch to accommodate higher wiring density, it has become important to conduct reliability assessments in a 1.00mm pitch format. This paper describes such an evaluation conducted using SAC BGA assemblies. The results show that for a 1.00mm pitch, the Pb-free SAC CBGA solution provides superior reliability as compared to the standard Sn/Pb CBGA solutions. This finding is an added incentive for a new CBGA offering employing the new Pb-free SAC single alloy self-aligning system.

11:55 AM
Characteristics of Ni Bearing Sn-Ag Composite Solders: *Joo-Won Lee*[1]; Zin-Hyoung Lee[1]; [1]Korea Advanced Institute of Science and Technology, Matl. Sci. & Eng., 371-1 Guseong Dong, Taejeon 305-701 S. Korea

Ni bearing Lead Free Sn-Cu-Ag solders have been investigated by employing various experimental techniques such as conventional cross-sectional metallography, scanning electron microscopy (SEM), electron microprobe analysis, X-ray diffraction, differential scanning calorimetry (DSC) and others. Based on the thermodynamic calculation, compositions of eutectic and composite solders were determined. Ni bearing solder had lower melting temperature and better mechanical properties, compared with Sn-Ag, Sn-Ag-Cu eutectic solders. Structure and composition of reinforcing particle were varied with the change of Cu/Ni ratio. This variation affected the sedimentation of reinforcing particles. Small addition of Ni was found to retard IMC layer thickening and to enhance ball shear strength after reflow and aging. IMC growth model was also discussed.

12:15 PM
Isothermal Fatigue Properties of Lead-Free BGA Joints: Yoshiharu Kariya[1]; *Takuya Hosoi*[2]; Yasunori Tanaka[3]; Masahisa Ostuka[2]; [1]National Institute for Materials Science, Eco-Matls. Ctr., Namiki 1-1, Tsukuba, Ibaraki 3050044 Japan; [2]Shibaura Institute of Technology, Matls. Sci. & Eng. Dept., Shibaura 3-9-14, Minato-Ku, Tokyo 10888548 Japan; [3]NEC Corporation, Mobile Terminal Div., Ikebecho, Tsuzuki-Ku, Yokomaha, Kanagawa 224 Japan

The split board fatigue test has been performed at 298K and 358K to look for strain life relationship of the Sn-3.5Ag-0.75Cu and Sn-37Pb solder joints made between a 0.5mm pitch or a 0.8mm pitch CSP and a PCB. The fatigue life of Sn-3.5Ag-0.75Cu joint is not significantly different to that of Sn-37Pb joint at as soldered condition, although the Sn-37Pb joint exhibits poor fatigue when the joint is subjected to the aging at 393K for 20 days. The degradation of Sn-37Pb joint due to the aging is attributed to the formation of Au-Ni-Sn and Ni-Sn layers. As the fatigue life of the joints was correlated to the strain energy density regardless of the pitch, the energy based life prediction model well describes the fatigue life of CSP/PCB interconnects. A set of strain energy density versus fatigue life curves is provided which can be used to predict the joint life.

Magnesium Technology 2003: Magnesium Alloy Development-Mechanical Properties - I

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

Wednesday AM Room: 2
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Mihriban Pekguleryuz, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Robert R. Powell, General Motor Corporation, NAO R&D Ctr., Warren, MI 48090-9055 USA

8:30 AM
Enhanced Ductility and Strength Through RE Addition to Magnesium Die Casting Alloys: *Per Bakke*[1]; Ketil Pettersen[1]; HÂkon Westengen[1]; [1]Norsk Hydro ASA, Magnesium Matls. Tech., PO Box 2560, Porsgrunn N-3907 Norway

Development of new die casting alloys is a multifacetted task, where success depends upon the ability to control a chain of properties, where the weakest link determines the outcome. It is commonly experienced that optimizing one property by alloying comes at the expense of one or more other properties. A typical example is yield strength vs. ductility. In developing alloys for high pressure die casting, the peculiar aspects of the process must be considered. High injection speeds, high metal pressures and the lack of efficient thermal barriers lead to extremely high cooling rates. This is making high pressure diecasting unique, since the resulting refined microstructure provides excellent mechanical properties. In the current paper, the influence of alloy composition on mechanical properties is investigated, with special emphasis on strength and ductility.

8:50 AM
Dead Sea Magnesium Alloys Newly Developed for High Temperature Applications: *Eli Aghion*[1]; Boris Bronfin[1]; Frank von Buch[1]; Soehnke Schumann[1]; Horst Friedrich[2]; [1]Dead Sea Magnesium, Rsrch. Div., PO Box 1195, Beer-Sheva 84111 Israel; [2]Volkswagen AG, Vehicle Rsrch., Wolfsburg Germany

Recently several new magnesium alloys for high temperature applications have been developed with intention to obtain an adequate combination of different properties such as castability, creep resistance, mechanical properties, corrosion performance and affordable cost. Unfortunately, it is very difficult to achieve an optimal combination of properties and in fact, most of the new alloys can only address part of the required properties and performances. This paper aims at evaluating the current status of the newly developed alloys for powertrain applications. The paper also explains the complexity of magnesium alloy development and illustrates the effect of alloying elements on properties and cost. In addition, the paper presents an attempt to set the position of each alloy in the space of combined properties and cost.

9:10 AM
Effects of Ca Additions on Microstructures, Age Hardening Response and Creep Behaviour of Mg-8Zn-4Al Casting Alloy: *Chamini Mendis*[1]; Laure Bourgeois[1]; Barry Muddle[1]; Jian-Feng Nie[1]; [1]Monash University, Sch. of Physics & Matls. Eng., Melbourne, Victoria 3800 Australia

Increased applications of magnesium alloys for elevated temperature service in the automotive industry require the development of low-cost, high-strength and creep-resistant alloys. It has been demonstrated in recent years that Mg-Zn-Al casting alloys exhibit creep resistance superior to that of binary Mg-Al and Mg-Zn alloys, and that quaternary additions of small concentrations of Ca to these Mg-Zn-Al alloys further improve the creep resistance of the alloys. However, the microstructure and creep behaviour of the Mg-Zn-Al(-Ca) alloys have not been characterized in detail, and the role of Ca in improving the creep resistance remains to be elucidated. In this work, the age-hardening response of Mg-8Zn-4Al (wt%) alloys, with and without a quaternary addition of 0.5wt% Ca, in the temperature range 150-200∞C has been measured using Vickers hardness testing, and precipitate microstructures have been characterised using transmission electron microscopy. The peak-aged samples have been creep tested at selected stress levels in the temperature range 125-175∞C. It is found that additions of Ca result in significant change in the commence of over-aging, even though little change was observed in the maximum hardness achievable. Results from creep tests indicate that the Ca-containing alloy has higher creep strengths and lower minimum-creep rates than the Ca-free alloy. Examinations of microstructures of peak-aged samples reveal that the majority of retained intermetallic particles in both alloys are not the cubic T phase, Mg32(Al,Zn)49. The microstructure of peak-aged samples of the Ca-free alloy contains predominantly a distribution of diamond-shaped precipitates, while an additional distribution of rod-shaped precipitates was observed in the Ca-containing alloy.

9:30 AM
The Effect of Exposure to Elevated Temperature on the Microstructure and Hardness of Mg-Ca-Zn and Mg-Ca-Zn-Si Alloys: Amir Finkel[1]; Ludmila Shepeleva[1]; *Menachem Bamberger*[1]; Eugin Rabkin[1]; [1]Technion, Dept. of Matls. Eng., Technion City, Haifa 32000 Israel

Two alloys of Mg-5wt%Ca-6wt%Zn (MCZ) and Mg-5wt%Ca-6wt%Zn-2wt%Si (MCZS) were cast into steel mold and than exposed to 160°C for up to 40 days. The dependencies of microstructure, thermal behavior, microhardness and hardness on the exposure time were determined. This enabled us to monitor the thermal stability of the cast alloys. The as structure of MCZ alloy is composed mainly of α-Mg solid solution, and in the grain boundaries 2μm large precipitation of $CaMg_2$ and eutectic structure of Mg and $Ca_2Mg_6Zn_3$ were found. The $CaMg_2$ precipitates do not appear in as-cast MCZS, whereas coarse CaMgSi grains are scattered between the matrix grains. The α-Mg grain size in both alloys was approximately 10μm. The structure of both alloys did not change during the exposure to the elevated tem-

perature, although a small increase of the amount of inter-granular phases was observed. As-cast MCZ alloy contain less inter-granular precipitates, but the relative change in their amount during exposure to elevated temperature is higher compared to MCZS. Exposure to 160°C resulted in a decrease of micro-hardness of α-Mg grains in both alloys, but no change in the overall hardness of MCZ alloy was observed during this time, in contrast with the age hardening behavior of MCZS alloy. MCZ alloy is harder than MCZS alloy for all exposure times. Based on thermodynamic calculations utilizing the Thermo-Calc software package the composition-microstructure-hardness variations were studied and the effect of Si content in MCZS alloys on the microstructure was evaluated.

9:50 AM

Phase Identification and Microanalysis of Phases in Mg-Al-Ca Alloy System: *Koray Ozturk*[1]; Zi-Kui Liu[2]; Alan A. Luo[3]; [1]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., 107 Steidle Bldg., University Park, PA 16802 USA; [2]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., 209 Steidle Bldg., University Park, PA 16802 USA; [3]General Motors Research and Development Center, Matls. & Processes Lab., 30500 Mound Rd., Warren, MI 48090-9055 USA

A fast and efficient characterization route was employed to the Mg-Al-Ca system for the construction of the ternary phase diagram. Two alloys (Alloy A: Mg-4.5%Al-1.9%Ca and Ally B: Mg-4.5%Al-3.0%Ca) and one Mg-Al-Ca diffusion triple were used in the present investigation. The alloys were sealed under an inert atmosphere and heat treated at 290C and 370C for one week. The diffusion triple was assembled carefully using precisely machined pure elements of Mg, Al and Ca and heat treated at 370C under an inert atmosphere. Crystal structure identification of the phases was made by electron backscatter diffraction (EBD) technique. And, the chemical composition of the individual phases was determined using electron probe microanalysis (EPMA).

10:10 AM Break

10:20 AM

Magnesium Diecasting Alloy Aj62x with Superior Creep Resistance, Ductility and Diecastability: *Mihriban Ozden Pekguleryuz*[1]; Pierre Labelle[1]; Donald L. Argo[1]; Eric Baril[1]; [1]Noranda, Noranda Tech. Ctr., 240 Hymus, Montreal, Quebec H9R1G5 Canada

Magnesium diecasting alloys for elevated temperature applications are coming of age. Several research centers and companies have been working on alloy systems based on alkaline earth and rare earth alloying additions to push the limits for the creep performance of Mg-based diecasting alloys. Norandaís Mg-Al-Sr based alloys have shown superior creep performance and high-temperature performance at temperatures as high as 150-175C and stress levels as high as 50 MPa. The most recent alloy formulation AJ62x (Mg-6Al-2Sr) has in addition shown excellent castability, high ductility and superior hot-tear resistance. Based on these attributes AJ62x is positioned well for applications such as transmission cases and oil pans. In this paper, mechanical properties (creep and tensile) and microstructure of AJ62x are presented. High ductility is an added advantage for this alloy. Industrial trials indicating that the alloy is highly castable and lab-scale evaluation shows that the alloy is more resistance to hot-tearing and cracking than all other magnesium alloys and the A380 aluminum alloy.

10:40 AM

Die Cast Magnesium Alloys Ae42 and Aj52x for High Temperature Applications: *Yemi Fasoyinu*[2]; Terri Castles[2]; RÈal Bouchard[2]; Mahi Sahoo[2]; Mihriban Pekguleryuz[1]; Pierre Labelle[1]; [1]Noranda, Noranda Tech. Ctr., 240, Hymus, Montreal, Quebec H9R1G5 Canada; [2]CANMET, Matls. Tech. Lab., 568 Booth St., Ottawa, Ontario K1A 0G1 Canada

Several R&D laboratories and companies are working to develop new die cast magnesium alloys with high-temperature properties better than the AZ and AM series. These R&D initiatives were driven by the need for alloys that could be used in such applications as transmission cases and covers, and other structural parts where improved performance at higher operating temperatures (150-175C) is desirable. This paper provides a comparitive evaluation of diecast microstructures and mechanical properties of AE42 (Mg-4Al-2 Rare Earth) and AJ52x (Mg-5Al-2Sr) alloys. Second phases in the two alloys are discussed and their stability are discussed. Tensile, impact, creep and fatigue property data generated in an ongoing research program at Materials Technology Laboratory (MTL)-Canada Centre for Minerals and Energy Technology (CANMET) are compiled. The effects of test bar thickness and test temperature on mechanical properties are investigated. Alloy AJ52x exhibits significantly better creep properties than alloy AE42, especially at higher applied stress (50 MPa) and 200 hrs rupture time. The use of a multi-cavity die to produce test samples of different section thickness or geometry requires optimization of the die design and/or pressurization parameters to reduce turbulence and porosity formation during die casting operation. It is shown that less than optimum properties could occur in die cast test samples due to the presence of surface defects and centerline shrinkage and/or gas porosity.

11:00 AM

Zirconium Alloying and Grain Refinement of Magnesium Alloys: *Ma Qian*[1]; *David H. StJohn*[1]; M. T. Frost[2]; [1]The University of Queensland, CRC for Cast Metals Mfg. (CAST), Div. of Matls. Eng., Sch. of Eng., Brisbane, Queensland 4073 Australia; [2]Australian Magnesium Corporation, Ltd., PO Box 1364, QLD, Milton, BC 4064 Australia

Factors that influence alloying zirconium to magnesium with a Mg-33.3Zr master alloy and the subsequent grain refinement were discussed based on a large number of experiments conducted at the laboratory scale. Particular attention was given to the influence of alloying temperature, stirring pattern, Fe pick-up from steel crucibles and residence time prior to pouring on the soluble and total zirconium in the final alloy. It was shown that excellent grain refinement was dictated by both soluble and total zirconium in the melt immediately prior to pouring. An ideal zirconium alloying process should end up with both high soluble and high total zirconium in order to achieve the best grain refinement in the final alloy. The mechanism of grain refinement of magnesium by zirconium was discussed based on the grain structures observed in the final alloy and the role of soluble and total zirconium in the grain refinement of magnesium.

11:20 AM

Grain Refinement of Magnesium Alloys Using Rolled ZirmaxÆ Master Alloy (Mg-33.3Zr): *Ma Qian*[1]; *David H. StJohn*[1]; M. T. Frost[2]; R. M. Barnett[1]; [1]The University of Queensland, CRC for Cast Metals Mfg. (CAST), Div. of Matls. Eng., Sch. of Eng., Brisbane, Queensland 4072 Australia; [2]Australian Magnesium Corporation, Ltd., PO Box 1364, Milton, BC QLD 4064 Australia

Owing to the limited solubility of zirconium in magnesium, almost all of the zirconium contained in the ZirmaxÆ master alloy (Mg-33.3Zr) is present in the form of nearly pure zirconium particles. Of them, individual particles and solid particle clusters greater than 5 microns in dimension account for approximately 70% of the area of all zirconium particles observed in the microstructure. Rolling a ZirmaxÆ master alloy ingot can allow coarse zirconium particles and particle clusters to fragment. Comparisons were made of the grain refining ability between as-cast ZirmaxÆ and deformed ZirmaxÆ by adding both forms of master alloy to pure magnesium under the same conditions. It was found that the use of rolled ZirmaxÆ delivered higher total and soluble zirconium, therefore better grain refinement in the final alloy than the use of as-cast ZirmaxÆ. The improved performance of rolled ZirmaxÆ master alloy was primarily attributed to less intense zirconium particle settling.

Materials Lifetime Science and Engineering - III

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; D. Gary Harlow, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015-3085 USA; Dwaine L. Klarstrom, Haynes International, Inc., Kokomo, IN 46904-9013 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Robert P. Wei, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015 USA

Wednesday AM Room: 18
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Dwaine L. Klarstrom, Haynes International Inc., Kokomo, IN 46904-9013 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA

8:30 AM Invited

On the Development of Life Prediction Methodologies for the Failure of Human Teeth: R. K. Nalla[1]; V. Imbeni[1]; J. H. Kinney[2]; S. J. Marshall[2]; *R. O. Ritchie*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]University of California-San Francisco, Dept. of Preventive & Restorative Dental Scis., San Francisco, CA 94143 USA

Human dentin is known to be susceptible to failure under cyclic loading. However, there are few reports that quantify the effect of such loading, especially when viewed in conjunction with the fact that a typical tooth experiences typically a million loading cycles annually. In the present study, a systematic investigation is described of the effects of prolonged cyclic loading on human dentin in a simulated physiological environment. In vitro stress-life (S/N) data are discussed in the context of possible mechanisms of fatigue damage and failure. Stiffness loss data collected in situ during these tests are used to calculate crack-growth velocities and the fatigue thresholds, and are presented as plots of the crack-propagation rates (da/dN) as a function of the stress-intensity range (deltaK). The S/N and da/dN-deltaK data are discussed in light of a framework for a fracture mechanics-based methodology for the prediction of the fatigue life of human teeth.

9:00 AM Invited

Fatigue of Interfaces in Adhesive Bonds: *J. K. Shang*[1]; T. Du[1]; [1]University of Illinois at Urbana-Champaign, 1304 W. Green St., Urbana, IL 61801 USA

Polymer adhesives are widely used to bond advanced materials in structural applications where durability of the adhesive bond remains a major concern, especially under cyclic loading in an aggressive environment. In this paper, we will focus on durability of the interface in the adhesive bond. Three experimental techniques for characterizing durability of the interface will be introduced. Effects of mechanical and environmental variables on durability of polymer-metal interface will be examined. Efforts for improving durability of polymer-metal interfaces by using self-assembled monolayers will be described. Finite element models will be presented to predict fatigue lives of adhesive joints based on damage tolerant and total-life approaches.

9:30 AM

Predicting Material Consumption by Cyclic Oxidation Spalling Models: *James L. Smialek*[1]; [1]NASA Glenn Research Center, Matls. Div., 21000 Brookpark Rd., MS 106-1, Cleveland, OH 44135 USA

The cyclic oxidation process has been described by an iterative scale growth and spallation model. Inputs include the selection of an oxidation growth law and a spalling geometry, plus oxide phase, growth rate, spall constant, and cycle duration. Output includes weight change, the amounts of retained and spalled oxide, the total amount of oxygen and metal consumed, and the terminal rates of weight loss and metal consumption. A computer program, COSP for Windows, was used to run multiple families of curves demonstrating the functional behavior of the outputs for various input parameters. A simple summation series has also been developed for a special case of interfacial spallation, producing closed form equations and the expression of various descriptive parameters as direct functions of the inputs. By suitably normalizing the weight change and cycle number, all cyclic oxidation model curves can be represented by a dimensionless, universal expression. Comparison to actual data validates the model prediction.

9:50 AM Invited

Corrosion Damage Functions and Life Prediction: *Russell H. Jones*[1]; [1]Pacific Northwest National Laboratory, Matls. Sci., PO Box 999, MSIN P8-15, Richland, WA 99352 USA

Corrosion damage can lead to reduced operational lifetimes. Often this damage is not as obvious as general corrosion but takes the form of pits, intergranular corrosion, crevice corrosion and hydrogen absorption. These types of corrosion damage lead to stress corrosion cracking, hydrogen induced cracking and corrosion fatigue. A critical step in defining a corrosion damage function is determining the relationship between the corrosion damage, the resulting crack propagation mechanism and component lifetimes. The sequence of events is often some localized corrosion event such as pitting, transition of the pit to a planar crack, propagation of this short crack, transition of the short crack to long crack conditions and continued propagation through stage I, II and III of the long crack SCC regimes. A description of critical corrosion damage processes and examples of the transition to long crack SCC conditions will be discussed.

10:20 AM Invited

Deuterium-Induced Fracture of Beryllium Films from Exposure to Experimental Reactor Environments: *Neville R. Moody*[1]; Rion A. Causey[1]; David F. Bahr[2]; Kenneth L. Wilson[1]; [1]Sandia National Laboratories, PO Box 969, MS 9404, Livermore, CA 94551-0969 USA; [2]Washington State University, Pullman, WA 99164 USA

Beryllium is a prime candidate material in the design of the International Thermonuclear Experimental Reactor (ITER) where it is proposed for use in components exposed to the hot tritium plasma. It has excellent thermal conductivity and a lower affinity for tritium than other candidate materials. However, deposition of energetic tritium ions with beryllium atoms can lead to formation of films that delaminate and blister. We therefore studied blister formation in beryllium films by replacing tritium with deuterium and using Atomic Force Microscopy to characterize blister sizes and shapes. Mechanics-based models were then combined with these measurements to determine the effect of deuterium on interfacial fracture energies. The analysis showed that deuterium induced very high compressive residual stresses in beryllium films. More importantly, the results showed that deuterium markedly lowered the interfacial fracture strength of these films. The authors gratefully acknowledge the support of the USDOE through Contract DE-AC04-94AL85000.

10:50 AM

Quantitative Cyclic Oxidation Modelling on Alumina Former: *Dominique Poquillon*[1]; Daniel Monceau[1]; [1]CIRIMAT, INPT/ENSIACET, 118 Rt. de Narbonne, Toulouse 31077 France

Structural materials for high temperatures are often subjected to cyclic conditions of atmosphere, mechanical loading and temperature. Most components used over 900∞C are made of alumina formers alloys. Oxidation is an important source of degradation which may even control the time of life of these systems, especially over 1050∞C. Lifetime prediction then requires the quantitative assessment of the metal rate consumption. Modeling of the oxide scale growth and degradation kinetics, as well as in-situ direct measurements are developed simultaneously in order to provide a quantification of cyclic oxidation experiments, to allow extrapolations for long life prediction, to supply data in diffusion models, but also to test the physical models which describe oxide spallation.

11:10 AM

Effects of Test Sample Thickness, Coating and Environment on the Creep Rate and the Time to Rupture of Single Crystal Nickel Base Superalloy: *SÈbastien Dryepondt*[1]; Eric Andrieu[1]; Daniel Monceau[1]; Fabrice Crabos[2]; Cyril Vernault[2]; Pierre Monge-Cadet[2]; [1]ENSIACET/CIRIMAT, 118 rte. de Narbonne, Toulouse, Cedex 4 31077 France; [2]Turbomeca, DE/MTA/EAP, Bordes 64511 France

Creep tests were carried out on thin plates and cylindrical MC2 samples coated or not with NiCoCrAlYTa, at 1150∞C, 80 and 140 MPa. The creep curves obtained in laboratory air show an increase in the steady-state creep rate leading to a decrease in time to rupture in the case of uncoated thin samples compared with thick ones. The gain in term of life duration increases along with decreasing the creep stress. The coating improves the creep life duration for thin samples whereas it has no significant effect for thick samples. This observation suggests that environment has a deteriorating effect on uncoated MC2 creep properties, which can be erased with the use of a NiCoCrAlYTa coating. Further creep tests have been performed to estimate the influence of environment under different temperatures and loading conditions. Interactions between oxidation and creep properties are studied using experimental setups under controlled atmosphere.

11:30 AM

Influence of Environment on Mechanical Behavior of Alloy 718 at 650∞C: *Veronique Garat*[1]; Bernard Viguier[2]; Jean Marc Cloue[3]; Eric Andrieu[2]; [1]FRAMATOME-ANP, 10, rue Juliette Recamier, Lyon 69456 France; [2]ENSIACET, CIRIMAT, 118, rte de Narbonne, Toulouse 31077 France; [3]Framatome-ANP, 1, rue Baptiste Marcet, Le Creusot 71205 France

The 718 alloy is widely used in aeronautical industry for its good creep and corrosion resistance. To study the influence of oxidation on the flow rules, creep tests were carried out at 650∞C on thin specimens (0.27 mm), under vacuum and under laboratory air testing conditions. The results of these tests showed that the steady-state creep strain rate is higher under laboratory air than under vacuum, whereas no damage, in terms of intergranular cracking, is observed on the surface specimen. In parallel, SIMS analysis were performed on specimens aged from 1 to 100 hours, at 650 or 700∞C under air and vacuum conditions. This characterization allows us to link the type of oxide formed to the modification of creep behavior of 718 alloy. The consequences of those observations on both flow rules and in the field of oxidation assisted cracking are addressed.

11:50 AM
The Behavior of HaynesÆ C-2000Æ Superalloy Subjected to Strain-Controlled Fatigue Loading: *R. L. McDaniels*[1]; L. Chen[1]; R. Steward[1]; P. K. Liaw[1]; R. A. Buchanan[1]; D. L. Klarstrom[2]; [1]The University of Tennessee, Dept. of Matls. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

The strain-controlled fatigue behavior of the new HaynesÆ Hastelloy C-2000 nickel-chromium-molybdenum superalloy was investigated at total strain ranges from 0.4% to 2.0% and at temperatures from 24∞C to 927∞C. The test specimens were subjected to fully reversed, push-pull, strain-controlled fatigue tests under axial strain range control. The results indicated that both test temperature and total strain range had a significant effect on the strain-controlled fatigue properties of the alloy. The alloy exhibited cyclic hardening, cyclic stability, or cyclic softening, depending on the temperature and strain to which the individual specimens were subjected. Coffin-Manson and Holloman parameters were found and plotted. Microstructural analysis of the as-received and tested specimens was also conducted using optical and scanning electron microscopy to provide a mechanistic understanding of the fatigue behavior. The present work is supported by the NSF Integrative Graduate Education and Research Training (IGERT) program, with Dr. W. Jennings and Dr. L. Goldberg as contract monitors and the Haynes International, Inc., with Dr. D. Klarstrom as the contract monitor.

Materials Processing Fundamentals: Heat and Fluid Flow: Modeling and Property Effects

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee, EPD-Process Fundamentals Committee

Program Organizers: Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA; Princewill N. Anyalebechi, Grand Valley State University, L. V. Eberhard Center, Grand Rapids, MI 49504-6495 USA

Wednesday AM Room: 1A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Rodney L. Williamson, Sandia National Laboratories, Liquid Metal Procg. Lab., Albuquerque, NM 87185-1134 USA; Uday Pal, Boston University, Dept. of Mfg. Eng., MA 02446 USA

8:30 AM
Model-Based Control of the Vacuum Arc Remelting Process: *Rodney L. Williamson*[1]; Joseph J. Beaman[2]; David K. Melgaard[1]; [1]Sandia National Laboratories, Dept. 1835, Liquid Metal Procg. Lab., PO Box 5800, MS 1134, Albuquerque, NM 87185-1134 USA; [2]University of Texas, Mechl. Eng., 700 Texas Ave., Austin, TX 78705 USA

A difficult control problem arises when a critical process variable responds in a nonlinear, history dependent fashion to a control input variable. Such a problem arises in vacuum arc remelting (VAR), an industrial metallurgical process used to cast large alloy ingots. Part of the VAR control problem consists of adjusting arc power to control melt rate. However, melt rate also depends on the electrode temperature distribution (ETD) causing the melt rate response to be history dependent and nonlinear. The situation is complicated further because the ETD cannot be measured, and electrode weight measurements are very noisy. To address this problem, a melting model was used to estimate the ETD. The model was incorporated into a control system that has been successfully used in VAR of nickel-base superalloys and aerospace titanium alloys. Data from laboratory and industrial tests show that the goal of accurate, ìinstantaneousî melt rate control has been achieved.

9:00 AM
An Investigation of Particulate Flows by Particle Image Velocimetry and Mathematical Modeling: Daniel Steingart[1]; *James W. Evans*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94610 USA

The flow of large dense particles has been investigated experimentally and by mathematical modeling. The experiments entail the measurements of the flow of beds of particles through orifices of various sizes by particle image velocimetry (PIV). This technique provides both the flow rate through the orifice and the distribution of particle velocities within the bed. Measurements have been made in air and in water. In some cases the water measurements were carried out with simultaneous flow of water through the orifice. In a few cases a boroscope was used to examine the details of movement of particles within the bed. The model of Hong and Caram has been adapted to simulate the dynamics of bed flow and shows a good fit to the experimental data.

9:20 AM
Flow of Steel in the Mold Region During the Continuous Casting of Steel: X. G. Zhang[1]; D. Steingart[1]; C. D. Seybert[2]; *James W. Evans*[1]; [1]University of California-Berkeley, Matls. Sci. & Eng., Berkeley, CA 94720 USA

Particle image velocimetry and a water model, with size of 1840 by 280mm, were used to study the fluid flow phenomena happening in continuous casting of steel. Two types of solidified shell, smooth and rough, were employed to simulate different liquid-solid interfacial condition. The effect on flow behavior of nozzle angle and submergence, as well as casting speed, were investigated quantitatively. The results show that: 1) There exist two large separate re-circulation loops above and below the fluid jet in the mold under the smooth interface condition. However, in the case of a rough surface, representing a coarse dendritic structure at the interface, it was found that not only was the velocity of fluid decreased, but that there were additional small vortices in the upper region of the mold. 2) The angle and submergence depth of the nozzle are two important factors that affect the flow pattern; they can even change the flow direction, 3) The higher the casting speed, the higher the velocity of the jets emerging from the nozzle ports (and traversing the liquid pool to the narrow face of the steel shell) and the higher impacting point on that face. Higher casting speed gives rise to large fluctuations of the meniscus with resulting defects. A Large Eddy Simulation (LES) model was used to compute the fluid flow.

9:40 AM
Effect of Initial Sample Temperature on the Boiling Water Heat Transfer During Quenching of a Stainless Steel Plate: *Dianfeng Li*[1]; Mary A. Wells[1]; Steve L. Cockcroft[1]; Gary T. Lockhart[1]; [1]The University of British Columbia, Dept. of Metals & Matls. Eng., Frank A. Forward Bldg., 6350 Stores Rd., Vancouver, BC V6T1Z4 Canada

A 2-D Inverse Heat Conduction (IHC) model was developed and verified to quantitatively determine the boiling water heat transfer during water quenching. Using a custom-built rig, with a single nozzle above the sample, a series of tests to examine the effect of initial sample temperature on the boiling water heat transfer during quenching of an AISI 316 plate were done. Sample starting temperatures ranged from 1000∞C-600∞C and the study demonstrated that when the starting temperature of the sample was above the Leidenfrost point no effect of temperature on the boiling water curve was seen. However, when the starting temperature of the sample was below the Leidenfrost point the temperature had a strong effect on the calculated boiling curve. This work shows that boiling water heat transfer is not only a function of the sample surface temperature but is also dependent on the thermal history experienced by the sample.

10:00 AM
Experimental Study of the Bubble-Burst Phenomenon at the Surface of a Liquid Steel Bath: Anne-GwÈnaÎlle GuÈzennec[1]; Jean-Christophe Huber[2]; *Fabrice Patisson*[1]; Philippe Sessiecq[1]; Jean-Pierre Birat[2]; Denis Ablitzer[1]; [1]Ecole des Mines, LSG2M, UMR 7584 CNRS-INPL, Parc de Saurupt, Nancy 54042 France; [2]IRSID, voie romaine, BP 30320, MaiziËres-lËs-Metz 57283 France

We have developed an experimental device for studying the main mechanism of dust formation in electric arc furnace steelmaking: the burst of gas bubbles at the liquid steel surface. As in the case of the air-water system, the bubble-burst process takes place in three steps: breaking of the film cap, projection of film drops, and projection of jet drops. The film breaking and the jet drop formation are observed with high-speed video. The film drop aerosol enters a particle counter, which characterizes the drops in size and number. Results are presented and discussed. The quantification of both types of projections leads to the conclusion that the film drop projections represent the major source of dust. We currently are investigating the influence of the bubble size on the amount of film drop projections, with the aim of checking the theoretical existence of an opimal bubble size that minimizes dust emission.

10:20 AM Break

10:40 AM Cancelled
Energy Efficiency of Fluidized Systems Used for Charge Preheating Before Electric Furnaces: *Kamal Adham*[1]; Cassandra Lee[1]; [1]Hatch Associates, 2800 Speakman Dr., Sheridan Sci. & Tech. Park, Mississauga, Ontario L5K 2R7 Canada

11:00 AM
Fundamental Study of the Interaction of Liquids with Rotating Disks: *Panos Tsakiropoulos*[1]; Yawei Wang[1]; [1]University of Surrey, Sch. of Eng. (H6), Mechl., Matls. & Aeros. Eng., Guildford, Surrey GU2 7XH England

The flow of a liquid on a stationary or rotating disk is widely encountered in many engineering practices. For example, the semiconductor industry uses this process to produce very thin and uniform coatings of photo resist films. In optical and magnetic recording media a thin layer of lubricant is used to prevent the slider from excessive wear during the start up and turnoff. TV manufacturers coat the TV screen with a thin film of coating. A thin film produced by a high speed-rotating disk provides a very important means for evaporation and drying of milk, soaps, detergents etc. The space industry exploits the flow of liquids on rotating disks in vapour absorption refrigeration systems. The metallurgical industry produces elemental or alloyed powders using centrifugal atomisation (CA). Near net shape metallic components can be manufactured by combining CA and spray forming (SF). Clean processing of alloy melts, which is a requirement of the aerospace and power plant generation industries, is also possible by combining cold hearth clean melting with CA or CA/SF. The paper will discuss the interaction of liquids with rotating disks and the characteristics of the spray as a function of flow rate, disk speed, and distance from injector to the point of impact on rotating disk. The outlines of centrifugal atomised sprays, studied using digital camera, will be compared. Results on the distribution of droplet size and droplet velocity in the spray, obtained using a Laser Phase Doppler Particle Analyser, will be presented. A theoretical analysis of the dependence of droplet formation mechanisms and spray characteristics on flow rate, disk speed and height from injector to disk will also be presented.

11:20 AM
Modeling Chip Formation of an Aluminum Alloy Using a Continuum Hydro-Dynamic Code: *Jaton Nakia Wince*[1]; Judy Schneider[2]; [1]Mississippi State University, Mechl. Eng., PO Box 1942, Mississippi State, MS 39762 USA; [2]Mississippi State University, Mechl. Eng., MS 9552, 210 Carpenter Bldg., Mississippi State, MS 39762 USA

This paper presents the simulation of chip formation in orthogonal metal cutting utilizing the predictive capabilities of DYNA 3D. The Johnson and Cook constitutive model for materials, Aluminum 6061 T6 alloy, were incorporated into the simulation to account for the effects of strain hardening, strain rate hardening, and thermal softening effects of the materials being machined. The model was compared to experimentally measured metal cutting plasticity parameters: shear plane angle and shear front angle, to verify the simulations accuracy.

11:40 AM
High Temperature Fatigue and Mechanical Properties of a Die Steel H3: *Eun-Gu Yoh*[1]; Y.-S. Lee[2]; [1]Kookmin University, Auto. Eng., 861-1 Chungneung-Dong, Sungbuk-Gu, Seoul, Seoul 136-702 Korea; [2]Kookmin University, Mechl. Eng., 861-1 Chungneung-Dong, Sungbuk-Gu, Seoul 136-702 Korea

The temperature in hot forming of metallic materials, such as hot extrusion and hot forging, ranges from 300 to 1200°C. Correspondingly, the die also exhibits very high temperatures very close to that of a work piece and its life is limited generally by high temperature fatigue. Thus, the analysis of high temperature fatigue would need the mechanical properties over the wide ranges of temperature. However, very few studies on the high temperature fatigue of brittle materials have been reported. Especially, the study on the fatigue behavior over such transition temperature regime is very rare. In this paper, the stress-strain curves and stress-life curves of a die steel such as H3 are experimentally obtained. The wide ranges of temperature from 300 to 1200°C are considered in experiments. And the transition temperature zone is carefully examined.

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

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee

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

Wednesday AM Room: 14A
March 5, 2003 Location: San Diego Convention Center

Session Chair: Nagy H. El-Kaddah, University of Alabama, Dept. of Metallurgl. & Matls. Eng., Tuscallosa, AL 35487-0202 USA

8:30 AM Opening Remarks

8:40 AM
Solid Ceramic Particulate Behavior into Molten Steel Bath Heated by Induced Currents: *Ignacio Erauskin*[1]; IÒigo Agote[2]; Ibon Mitxelena[2]; Mikel Asensio[2]; [1]Fundacion Inasmet, Matls. & Processes, Paseo Mikeletegi, 2 Parque Tecnologico., San Sebastian Spain; [2]Fundacion Inasmet, Paseo Mikeletegi, 2. Parque TecnolÛgico, San Sebasti·n Spain

The improvements that could be obtained in the properties of cast structural alloys through the reinforcing its matrix with hard, microscopic and dispersed ceramic particles as TiC type carbides, are accepted and recognized. In this sense, the development of a liquid metallurgy process enabling the reinforcement by means of the addition of the ceramic material to the molten metal in the melting furnace would become an important advance in the metallic materials field. Nevertheless, these ceramic products are prone to the coalescence and have poor wettability into molten baths as steel, superalloys, etc. A factor that minimizes these problems is the bath stirring during the ceramic particle addition, this stirring being produced, for instance, by the Foucault currents in a induction melting furnace. We have empirically verified, however, that the stirring magnitude, as function of the furnace power and frequency, and material density and resistivity, has a high influence in the ceramic behavior, so that, the working out of a numerical simulation software about this matter would allow the development of structural materials with improved properties.

9:00 AM Invited
Magnetic Field Alignment of Polymers to Enhance Material Properties: *Mark E. Smith*[1]; Brian C. Benicewicz[2]; Elliot P. Douglas[3]; Jim D. Earls[4]; Ralph D. Priester[4]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., Polymers & Coatings, MS E 549, Los Alamos, NM 87545 USA; [2]Rensselaer Polytechnic Institute, Troy, NY 12180 USA; [3]University of Florida, Gainsville, FL 32611 USA; [4]The Dow Chemical Company, Freeport, TX 77541 USA

Liquid crystalline polymers can be orientated by use of electrical or magnetic fields; however the majority of high molecular weight thermoplastic resins typically suffer from high melt viscosities, which limits the utility of this process method for materials production. Thermoset liquid crystals allow the viable application of electrical and magnetic orientation for solid part production. This research is focused on the curing of a liquid crystalline epoxy with a diamine crosslinker in the presence of magnetic fields. The results of statistical design experiments will be presented on the three main processing variables: magnetic field strength, time in field, and extent of B-staging. The magnetic processing method allows a substantial manufacturing window in which material of tailored properties can be produced. Underlying principles of the orientation mechanism and the nature of the liquid crystalline formation and stability are also presented.

9:30 AM
The Aligned Solidification Structure of MnBi in Semi-Solidified Bi-Mn Alloy with a Static Magnetic Field: *Zhongming Ren*[1]; Hui Wang[1]; Kang Deng[1]; Kuangdi Xu[1]; [1]Shanghai University, Dept. Matls., 149 Yan Chang Rd., Shanghai 200072 China

The macrostructures of the Bi-3%Mn, 6%Mn, and 20%Mn alloys solidified under the influence of a high static magnetic field (up to 10T) have been investigated experimentally. It is shown that the magnetic field influenced behavior of the primary phase MnBi crystals significantly. The morphology and alignment direction of the MnBi crystals were depended on the melting temperature and magnetic field. In the case of semi-melting temperature, the MnBi crystals were aligned along with the direction of the magnetic field, and in the case of whole melting temperature, the MnBi crystals were aligned perpendicularly to the direction of the field. The alignment tendency and the average length of elongated MnBi crystals of MnBi increased with the increase of the applied field and the solidification time. A model was proposed to explain the alignment and orientation growth of MnBi crystals in a magnetic field in terms of the magnetic anisotropy of the crystals and the interaction between them.

9:50 AM
Directional Solidification of Immiscible Alloys Under an Applied Electromagnetic Force Field: *Nagy El-Kaddah*[1]; Lirong Tong[1]; [1]The University of Alabama, Dept. of Metallurgl. Eng., Box 870202, Tuscaloosa, AL 35487 USA

This paper describes an electromagnetic approach for solidification processing of immiscible alloy composites. It is based on the use of the electromagnetic force generated by passing electric current through the melt and crossing it by a uniform magnetic field to offset and modulate the buoyancy force on dispersed droplets during solidification. The technique was used to study the macrostructure development of Zn-10 wt% Bi immiscible alloy over a wide range of effective gravity values in a vertical unidirectional solidification system. A mathematical model for describing the growth of nucleated dispersed phase in the two-liquid phase region ahead of the solidification front and the entrapment of these droplets by the moving solid-liquid interface in vertical unidirectional solidification systems has been developed. The model is used to study the particle size distribution in unidirectional solidified Zn-Bi hypermonotectic alloys at reduced gravity conditions. It has been found that the particle size and distribution in the solidified alloy depends on solidification rate and the ratio of effective gravity to thermocapillary forces. The model was found to reasonably predict the experimentally measured particle size and distribution over the entire range of effective gravity investigated.

10:10 AM
Magnetized Fiber Orientation and Concentration Control in Solidifying Composites: *George S. Dulikravich*[1]; Marcelo J. Colaco[1]; Thomas J. Martin[2]; [1]University of Texas at Arlington, Mech. & Aeros. Eng. Dept., Multidisciplinary Analy., Inverse Design & Optimization (MAIDO) Prog., UTA Box 19018, Arlington, TX 76018 USA; [2]Pratt & Whitney Engine Company, Turbine Discipline Eng. & Optimization Grp., 400 Main St., MS 165-16, E. Hartford, CT 06108 USA

If short carbon fibers (5-10 microns in diameter and 200 microns long) are vapor-coated with a thin layer (2-3 microns) of a ferromagnetic material like nickel, the fibers will respond to the applied magnetic fields by rapidly rotating and translating so that they become aligned with the magnetic lines of force. We have developed an improved analytical model and a numerical algorithm for the prediction of magnetic force lines inside a flowing solidifying melt. This computer code was combined with our hybrid constrained optimizer that minimized a normalized sum of least square differences between the user-specified and the predicted local magnetic lines of force geometric patterns. We have utilized this software package and the interactive graphics software to evaluate the strengths, locations, and orientations of magnets needed for generate specified magnetic field lines in the solidifying composite thus verifying the feasibility of this manufacturing process.

10:30 AM Break

10:50 AM
Microstructure Formation During BiMn/Bi Eutectic Growth with Applied Alternative Electric Fields: *Lili Zheng*[1]; *Yuan Ma*[1]; David J. Larson[2]; [1]University at Stony Brook, Dept. of Mechl. Eng., Stony Brook, NY 11794-2300 USA; [2]University at Stony Brook, Dept. of Matls. Sci. & Eng., Stony Brook, NY 11794-2275 USA

This paper is to demonstrate that the presence of an electric field can be used to control materials microstructure formation. Special efforts have been made to identify the foremost process control parameters that affect the interface dynamics, and thermoelectric effects on materials microstructure formation during directional solidification. A computational model that integrates microscopic analysis to macroscopic model has been developed and applied to directional solidification of BiMn/Bi eutectic in the presence of electric fields. Numerical results demonstrated that in addition to process parameters, microstructure formation strongly depends on intensity, and frequency of applied current, and it changes spontaneously as an electric field is applied. Predicted patterns of microstructures have qualitatively agreed with the experiments. The results indicate the feasibility of utilizing electric fields to control microstructure formation during eutectic growth.

11:10 AM
Generating Strange Interactions in Particle Suspensions: Classical Molecules and Particle Foams: *James E. Martin*[1]; Robert A. Anderson[1]; Rodney L. Williamson[1]; [1]Sandia National Laboratories, Organization 1120, MS 1421, PO Box 5800, Albuquerque, NM 87185-1421 USA

We have discovered a method of generating strange interactions in magnetic particle suspensions. These interactions can be attractive or repulsive, and lead to a variety of unexpected effects, including stable particle clusters with molecular geometries, the emergence of a particle foam phase, and amusing collective particle dynamics. Through this interaction novel isotropic particle structures can be made that cannot be produced by any other known means, leading to a new class of composite materials with highly optimized properties.

11:30 AM
Optimization of Wall Electrodes for Electro-Hydrodynamic Control of Charged Particle Depositions During Solidification: *George S. Dulikravich*[1]; Marcelo J. Colaco[1]; [1]University of Texas at Arlington, Mech. & Aeros. Eng. Dept., Multidisciplinary Analysis, Inverse Design & Optimization (MAIDO) Program, UTA Box 19018, Arlington, TX 76019 USA

In the case of electro-hydrodynamics (EHD), flow-field of electrically charged particles in a melt is influenced by an externally applied electric field in the absence of a magnetic field. Solidification front shape, distribution of the charged particles in the accrued solid, and the amount of accrued solid phase in such processes can be influenced by an appropriate distribution and orientation of the electric field. We have developed numerical analysis software for EHD solidification that is based on a finite volume method. This algorithm was then combined with a constrained optimization algorithm to determine the best locations of wall electrodes that will provide the most uniform generation and distribution of electrically charged particles in the accrued solid phase.

Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction: Macroscale Modeling & Simulation

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

Wednesday AM — Room: 16A
March 5, 2003 — Location: San Diego Convention Center

Session Chairs: Jay R. Jira, US Air Force, AF Rsrch. Lab. Matls & Mfg Direct., WPAFB, OH 45433-7817 USA; Robert A. Brockman, University of Dayton Research Institute, Dayton, OH 45469-0110 USA

8:30 AM
A USN Strategy for Mechanical and Propulsion Systems Prognostics with Demonstration Results: *William J. Hardman*[1]; [1]NAVAIR, AIR 4.4.2, B106, Unit 4, 22195 Elmer Rd., Patuxent River, MD 20670-1534 USA

A US Navy strategy has been generated to develop, integrate and demonstrate diagnostics, prognostics, health monitoring and life management for propulsion and mechanical systems. How this overall strategy has evolved and its current status will be presented. The SH-60 program was initiated as the first proof-of-concept effort to develop, demonstrate, and integrate available and advanced mechanical diagnostic technologies for propulsion and power drive system monitoring. Included in these technologies were various rule and model based analysis techniques that were applied to demonstrate and validate various levels of diagnostic and trending capabilities. Recently there has been increased emphasis on prognostic capabilities. As used in this paper, prognostics is the capability to provide early detection of the precursor and/or incipient fault condition to a component or sub-element failure condition; and to have the technology and means to manage and predict the progression of this fault condition to component failure. The benefit of this prognostic approach is increased safety and significantly reduced supportability costs over the aircraft life cycle; enabling better management of both existing and potential aircraft system faults. This prognostic philosophy, its benefits, and envisioned implementation will be further embellished. These will be discussed and updated with particular attention to recent gear fault detection results and capability demonstrations.

9:00 AM Invited
Calibration of Failure Mechanism-Based Prognosis with Vibratory State Awareness Applied to the H-60 Gearbox: *Gregory J. Kacprzynski*[1]; Avinash Sarlaskar[1]; Andrew J. Hess[2]; [1]Impact Technologies, LLC, 125 Tech Park Dr., Rochester, NY 14623 USA; [2]Naval Air Systems Command, 22195 Elmer Rd., Patuxent River, MD 20670 USA

This paper describes a generic prognostic module architeture configured for use on the H-60 IGB spiral bevel pinion gear. The software module integrates advanced stochastic failure mode modeling, failure progression information from vibration features, and run-to-failure experience bases to enable IGB pinion gear failure predictions in the H-60 critical drive train. The state-of-the-art structural/material level modeling aspects of the module includes the utilization of both a stochastic sub-zone crack initiation model and a 3D linear fracture mechanics model. Secondly, the module is engrained with an adaptive model updating techniques for ìtuningî key failure mode variables at a local material/damage-site based on fused vibration features. The failure rate prediction strategies are implemented within a probabilistic framework to directly identify confidence bounds associated with IGB pinion failure progression. The overall modeling scheme is aimed at minimizing inherent modeling and operational uncertainties by updating material/fatigue properties, crack propagation rates etc. via sensed system measurements that evolve as damage progresses. The results of seeded fault, run-to-failure tests on the IGB pinion gear will be provided and compared to prognostic module predictions.

9:25 AM Invited
Modeling of a Gear with EDM Crack: *Alan C. Leung*[1]; Virginia G. DeGiorgi[2]; [1]Nova Research (Naval Research Laboratory), Multifunctl. Matls.-6353, 1900 Elkin St., Ste. 230, Alexandria, VA 22308 USA; [2]Naval Research Laboratory, Multifunctl. Matls.-6353, 4555 Overlook Ave. SW, Washington, DC 20375 USA

Comparison of damage states during a structureís operation and prediction of the effects structural performance is a necessary first step in the process known as prognostics. The process developed must provide meaningful engineering data and be sensitive to small changes in material performance tracked by the material state awareness metric that monitors damage at the micro-structural scale. Such structures of interest are power trains. Gears, a key component to power trains, are the primary focus of this work. A 2-D FE model of a gear set was developed. A crack is placed on one gear and varied in length. The gears are subjected to varying amounts of torque. Displacements between gear teeth during gear contact are calculated. Relations between the displacement of gear teeth and the increasing crack length allow a means of assessing and monitoring gear damage with associated deformations. Comparisons are made with experimental data from a gear.

9:50 AM Break

10:20 AM Invited
Integrating Structural Analysis and Experimental Methods: *Robert A. Brockman*[1]; [1]University of Dayton Research Institute, 300 College Park Ave., MS 0110, Dayton, OH 45469-0110 USA

Analytical models of turbine engine components are reused in the event of changes in mission requirements, analysis and life prediction methodology, or fleet management philosophy. However, existing models of fielded systems contain no means of integrating state awareness information into the reanalysis process. Achieving the goal of comprehensive damage prognosis requires that measured data from numerous sources be assembled and used continually to sharpen analytical predictions of performance and potential failure. The problems involved in integrating state awareness data into structural analysis methods are similar to those encountered in integrating laboratory experiments and analytical models. Both require some reorganization of the analytical models and processes. This paper focuses on the role of structural analysis methods and software in materials prognosis, and how selected methods may need to be restructured to integrate known diagnostic data into refined predictions of component performance and life.

10:45 AM Invited
Prediction of Elevated Temperature Crack Growth in Spin Pit Tests: *Robert H. Van Stone*[1]; [1]GE, Aircraft Engines, 1 Neumann Way, MD K105, Cincinnati, OH 45215 USA

Accurate crack growth prediction methods are a necessary element in the use of advanced crack detection prognostic methods to extend the lives of rotating gas turbine components. Fracture mechanics methods used to predict the cyclic crack growth lives (without time dependent or surface enhancement effects) are well established. Linear elastic fracture mechanics methods have now been developed which accurately predict the acceleration associated with hold times at elevated temperatures using a linear superposition of cyclic and static crack growth rates. The beneficial effects of surface residual compressive stress such as those induced by shot peening can also be predicted using the superposition of the stress intensities calculated from the applied stress and a portion of the residual stress. The accuracy of these methods are assessed by comparing predicted and experimentally measured crack propagation lives for laboratory tests. These same methods will be used to predict the growth of cracks in spit pit tests planned to demonstrate the utility of advanced crack detection prognostics.

11:10 AM Invited
Materials Prognosis for Legacy Engines: *Bryon J. Wicks*[1]; [1]Department of Defence, DSTO-PSL-AVD, Box 4331, GPO, Melbourne, Victoria 3001 Australia

The main factor affecting the airworthiness, availability and through life support cost of legacy engines is the removal and replacement of critical rotating components. A safe life method is conventionally used to determine this life for components subject to low cycle fatigue, based on a statistical database. Subsequently, during engine service, unanticipated flaws develop which were not included in the data set, or the materials themselves degrade in a manner which was not originally expected, or alternatively additional data is generated and subsequently included in the design data set following service experience which frequently leads to life reductions which can have a dramatic effect on fleet availability. This paper examines several examples where the use of a safe life for determining component replacement intervals could be seen to be inappropriate and often unduly conservative. An alternative method for determining component lives is proposed, involving an assessment of the deterioration of materials during service from all mechanisms.

Measurement and Interpretation of Internal/ Residual Stresses: Neutron and SXRD

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

Wednesday AM Room: 17B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Aaron Krawitz, University of Missouri, Dept. of Mech. & Aeros. Eng., Columbia, MO 65211 USA; Hahn Choo, University of Tennessee, Matls. Sci. & Eng., Knoxville, TN 37996 USA

8:30 AM Invited
X-Ray Microbeam Measurements of Subgrain Stress Distributions in Polycrystalline Materials: *Gene E. Ice*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., One Bethel Valley Rd., Oak Ridge, TN 37831-6118 USA

The behavior of polycrystalline materials depends on many factors, including the grain size/morphology, elastic anisotropy of misoriented crystal grains, non-uniform elastic stress tensor distributions and deformation induced stored energy. We describe the worldís first dedicated polychromatic 3-dimensional (3D) scanning x-ray microscope: the 3D X-ray Crystal Microscope. This new class of x-ray instrument nondestructively measures intra-granular distributions of strain, crystallographic phase, local orientation and deformation with submicron spatial resolution in all three dimensions. The microscope simultaneously collects Laue patterns from every grain along the incident beam. With a beam size smaller than the typical grain size in most materials, only a few grains are simultaneously illuminated and special techniques can be used to disentangle the overlapping Laue patterns. Early results indicate the promise of this emerging class of instrumentation. New initiatives to improve the spatial resolution by an additional order of magnitude and to build a neutron analog are discussed.

9:00 AM Invited
Diffraction Measurement of Load Partitioning in Aluminum-Ceramic Microsphere Syntactic Foams: Dorian K. Balch[1]; *David C. Dunand*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

Liquid aluminum was infiltrated into packed alumina-mullite microspheres, resulting in a syntactic foam consisting of hollow ceramic spheres embedded in a pure aluminum matrix. Load transfer during uniaxial compression was studied by neutron and synchrotron x-ray diffraction. As for composites with solid reinforcement, load transfer was observed between the continuous metallic matrix and the hollow microspheres. The extent of load transfer is modelled and discussed in both the elastic and plastic regimes.

9:30 AM Invited
Differentiating Between Beams and Plates Using Microbeam X-Ray Diffraction: *Cevdet I. Noyan*[1]; [1]IBM, T. J. Watson Lab., Rt. 134, Kitchawan Rd., Yorktown Heights, NY 10598 USA

Pure(4-point) bending of beams and plates are fundamental topics in elementary mechanics. In the case of beams, transverse strains are generally neglected and pure bending results in a uniform stress distribution between the two inner supports. In the case of plates, such an assumption is not strong, and non-uniform stress distributions are theoretically predicted. In addition, anticlastic bending, which requires the plate to assume a ìsaddleî shape, becomes less important. In this study, we present microbeam curvature mapping of the surface of homogeneous Si single crystal strips subjected to pure bending. These measurements show that beams and plates can not be differentiated on the basis of their dimensions alone. We provide experimental proof of the Searle/Ashwell analysis (1908/1952) which should be used for such cases.

10:00 AM
Smarts: A Neutron Spectrometer for Studies of Engineering Materials: *Mark A.M. Bourke*[1]; Ersan Ustundag[3]; David C. Dunand[2]; Bjorn Clausen[1]; Sven Vogel[1]; Thomas Sisneros[1]; [1]Los Alamos National Laboratory, MST, MS H805, Los Alamos, NM 87545 USA; [2]Northwestern University, Dept. Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [3]California Institute of Technology, MC 138-78, Pasadena, CA 91125 USA

In August of 2001 a new neutron scattering instrument called SMARTS (Spectrometer for Materials Research at Temperature and Strain) took first beam. This instrument is the first of a new generation of instruments at pulsed neutron sources dedicated for the study of engineering materials problems. It was designed with two goals in mind i) to make spatially resolved measurements on engineered components and ii) to perform in situ loading studies at both ambient and elevated temperatures. In doing so it can address both macroscopic residual strain measurements and fundamental polycrystalline deformation problems. Funded by DOEís Office of Basic Energy Sciences, SMARTS was built by a development team that included but was not limited to the authors of this paper. Capabilities include 1mm3 spatial resolution, 230KN loading capacity, 1500C furnace and a translator that can manipulate loads up to 1500Kg. This paper will illustrate, by example, the range of problems that have been addressed to date.

10:20 AM Break

10:35 AM Invited
On the Effects of Residual Stresses on Fatigue Behavior in Deep-Rolled Ti-6Al-4V Alloys at Ambient and Elevated Temperatures: I. Altenberger[2]; R. K. Nalla[1]; U. Noster[2]; B. Scholtes[2]; *R. O. Ritchie*[1]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720-1760 USA; [2]University of Gh Kassel, Inst. of Matls. Tech., Kassel 34125 Germany

Mechanical surface treatments, such as deep rolling, shot peening and laser shock peening, can significantly improve the fatigue behavior of highly stressed metallic components commonly used in engineering applications. However, the utility of these treatments for higher temperature service is often brought into question. In the present study, we examine the effect of deep rolling and laser shock peening on the fatigue properties of a wrought Ti-6Al-4V at ambient and 450°C. Near-surface residual stresses were characterized using synchronous x-ray diffraction, and microstructural gradients using transmission electron microscopy, before and after mechanical/thermal exposure. It was found that despite the almost complete relaxation of residual stresses at the higher temperature, there was still a significant benefit for fatigue resistance from the surface treatments. This was attributed to the formation of a stable, near-surface work-hardened layer, with a nano-scale grain size, which acts to diminish the plastic strain amplitude in fatigue, thereby inhibiting both fatigue crack initiation and propagation and hence improving lifetimes.

11:05 AM Invited
The Role of Residual Stress Distributions in Engineering Applications by Synchrotron Probe: *Thomas Tsakalakos*[1]; [1]Rutgers University, Dept. of Ceram. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854-8065 USA

Quantitative understanding of the internal stresses field distribution under load is fundamental in the design engineering of static/cyclic load-bearing components. The manufacture/processing or duty cycle of such components can lead a residual stress distribution which can dramatically alter (for good or ill) a components load capacity, and resistance to failure. For example, compressive (tensile) surface stresses tend to retard (accelerate) the surface-initiation and growth of cracks. To compound the problem the stress distribution is extremely difficult to experimentally characterize, offers little or no external evidence of its existence, and is often recognized only a posteriori after failure. In order to make reliable estimates of component performance it is necessary to have an accurate knowledge of these stresses. In this paper, two powerful synchrotron x-ray scattering techniques for residual strain depth-profiling and tomography-like scatter-intensity profiling of materials are presented. The techniques utilize energy dispersive x-ray scattering, from a fixed micro-volume, with micro-scanning of the specimen being used to profile its interior. The tomography-like profiles exploit scattering-cross-section variations, and can be contrast-enhanced by separately monitoring scattering from different crystal structures. The strain profiling technique is shown to finely chronicle the internal strain variation over several mm of steel. Detailed strain profiling for a cantilever spring demonstrates the interplay of residual and external stresses in elastic/plastic deformation. Since surface compression, by shot peening, is a classic method to fortify against fatigue failure, the strain profile for shot-peened, surface-toughened material is determined and discussed in terms of a simple elastic-plastic stress/strain model. The residual stress profiling of fatigue deformation processes on WC/Co and Al2O3/TiO2 nanocoatings showed different internal stress relaxations and internal fatigue damages suggesting different mechanisms. Other residual stress distributions by surface treatment processes such as laser shot peening, low plasticity burnishing, and friction welding will be also reviewed. The correlation of the internal stresses (stress gradients) with both abrasive and sliding wear properties in Nanostructured and microsize-grain coatings will be presented. These results could explain the observed enhancement of wear resistance of nanocoatings vs. conventional. Finally, macro and micro stress distributions of super strength (2.5- 4 GPa) nano steel pearlitic wires will be presented along with modeling and theory. A fresh approach to the fatigue crack retardation by overload and other transient effects will be also presented. Overload effects, underload effects and fatigue crack growth resistance with increasing K are all relatable to the internal stresses arising from the dislocations in the plastic zone. Preliminary experiments have shown that the probing sensitivity of the EDXRD method is sufficient to elucidate the origin of this internal stress Kint by mapping the internal stress tensor in and around the plastic zone of a crack in steels and other alloys. Thus, it is demonstrated that, provided an accurate knowledge of the residual stress profiles generated is available and allowance is made for stress redistribution and the multiaxial nature of residual stresses, reliable predictions of fatigue damage and performance can be made. We gratefully acknowledge the support of the Office of Naval Research under grant N000149910424. Part of this work is also supported by the Defense University Research Initiative on NanoTechnology (DURINT), which is funded by a subcontract to Rutgers University through the Massachusetts Institute of Technology by the Office of Naval Research under Grant.

11:35 AM
Strain and Texture Analysis of Pb(Zr,Ti)O3 Under Compression: *Robert C. Rogan*[1]; Ersan Ustundag[1]; Bjorn Clausen[1]; Mark R. Daymond[2]; Volker Knoblauch[3]; [1]California Institute of Technology, Matls. Sci., 1200 E. California, MC 138-78, Pasadena, CA 91125 USA; [2]Rutherford-Appleton Laboratory, ISIS Neutron Scattering Fac., Chilton, Didcot OX11 0QX UK; [3]Robert Bosch GmbH, Rsrch. 1-Matls., PO Box 10 60 50, Stuttgart D-70049 Germany

In-situ neutron diffraction studies were performed on various piezoelectric Pb(Zr,Ti)O3, PZT, samples under compressive stress. Single phase tetragonal and rhombohedral systems were studied, as well as morphotropic compositions containing both phases. Rietveld analysis of the diffraction patterns allowed for observation of the onset and culmination of domain switching through modeling of the sample texture using the March-Dollase model. Calculated lattice parameters indicated significant residual stresses due to locked in domains, as well as significant strain anisotropy indicative of a complicated internal stress state. The effectiveness of electrical poling and mechanical depoling was evaluated.

Mercury Management: Treatment Technologies

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizer: Larry Twidwell, Montana Tech of the University of Montana, Metallurgical and Materials Engineering, Butte, MT 59701 USA

Wednesday AM Room: 1B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Larry Twidwell, Montana Tech of University of Montana, Metallurgl. & Matls. Eng. Dept., Butte, MT 59701 USA; Corby G. Anderson, Montana Tech of University of Montana, Ctr. for Adv. Minl. & Metallurgl. Procg. (CAMP), Butte, MT 59701 USA

8:30 AM Welcome

8:40 AM Cancelled
Evaluation of Treatment of Bulk Mercury and High Mercury Surrogate Waste: *Mary Cunningham*[1]; [1]US Environmental Protection Agency, Washington, DC USA

9:05 AM Invited
Toward Mercury Management on US Department of Energy Sites: Development and Selection of Technologies: *Michael I. Morris*[1]; Greg A. Hulet[2]; [1]Oak Ridge National Laboratory, Nucl. Sci. & Tech. Div., Oak Ridge, TN 37831-6179 USA; [2]Idaho Nuclear Environmental Engineering Laboratory, Idaho Falls, ID 83415-3875 USA

The US Department of Energy (DOE) TRU and Mixed Waste Focus Area (TMFA) is tasked with finding solutions for the mixed waste treatment problems of the DOE complex. During TMFAís initial technical baseline development process, three of the top four technology deficiencies identified were the need for amalgamation, stabilization, and separation/removal technologies for the treatment of mercury-contaminated mixed waste. The Mercury Working Group (HgWG), a selected group of representatives from DOE sites with significant mercury waste inventories, is assisting TMFA in soliciting, identifying, initiating, and managing efforts to address these areas. Solicitations and contract awards have been made to the private sector to demonstrate both the amalgamation and stabilization processes using actual mixed wastes. Development efforts that will address DOEís needs for separation and removal processes are currently being funded. This paper discusses the technology selection process, development activities, and the accomplishments of TMFA to date through these various activities.

9:30 AM Invited
Mercury Recovery from Chlor-Alkali Wastewater Processes Using REMERC: *Berndt Moeller*[1]; [1]Pioneer Americas, LLC, PO Box 23, St. Gabriel, LA 70776 USA

Mercury-cell chlor-alkali production plants generate mercury contaminated wastewater streams from which mercury must be precipitated and filtered, resulting in a sludge containing 2% mercury (designated K106) that cannot be landfilled in the United States without treatment. REMERC is a chemical treatment process used to recover 99% of the mercury from wastewater sludge generated at these plants. The process uses leaching to oxidize mercury compounds to mercuric chloride, washing and filtration to separate mercuric chloride from the treated sludge, and cementation to reduce the mercuric chloride to metallic mercury. Mercury not recovered in cementation is recycled to the wastewater process for precipitation and filtration. Treated sludge contains less than 260 ppm total mercury and 0.2 ppm TCLP, meeting the USEPA low mercury subcategory specifications that allow landfill disposal. REMERC uses readily available chemicals, is straightforward to control and inexpensive to operate, has no mercury emissions to air, and recovers metallic mercury. REMERC enabled significant cost savings including lower solids disposal costs, recovery and reuse of metallic mercury in the chlor-alkali process, and ability to treat non-wastewater sludge. No new EPA permits were required. REMERC was shown to be a viable process for recovery of metallic mercury from K106 designated sludge.

9:55 AM Break

10:10 AM Invited
Thermodynamic Investigation of the Stability of HgTe with Subsequent Recovery of Mercury from Copper Smelter Acid-Plant Sludge Using a Vacuum Retort Reactor: John P. Hager[1]; *Devon A. Harman*[1]; [1]Colorado School of Mines, Dept. of Metallurgl./ Matls. Eng., 1500 Illinois St., Golden, CO 80401 USA

Thermodynamic modeling calculations were undertaken for the recovery of mercury from HgTe with a vacuum retort reactor. Considerable discrepancy was noted in the available thermodynamic data. A series of transpiration experiments were undertaken on the thermal decomposition of HgTe. From the measured Gibbs Free Energy of decomposition, a 2nd law analysis was conducted to yield the Heat of Formation and Entropy at 298K, which provided for the development of a new database for HgTe in HSC Chemistry format. Modeling calculations were then conducted with HSC Chemistry. The minimum temperature for 100% volatilization of the mercury was determined as a function of retort pressure and the molar ratio of flush gas to HgTe. It was found that HgTe is more stable than was indicated from earlier references and that higher temperatures are required for the recovery of the mercury. It was determined that the temperature for 100% volatilization could be decreased from over 1180K to less than 790K by the use of a low residual pressure and the use of a high ratio of flush gas to HgTe.

10:35 AM Invited
Industrially Proven Methods for Mercury Removal from Gases: *Klas G. Hultbom*[1]; [1]Boliden Contech AB, Environml. Tech., PO Box 5097, Helsingborg, Boliden SE-250 05 Sweden

Mercury is present as a trace contaminant in many raw materials from metallurgical plants. When such materials are treated with pyrometallurgical processes the mercury is volatilised. Mercury has a high vapour pressure, even at low temperatures, and therefore it is removed only partly by normal gas cleaning processes. The Boliden Norzink scrubbing process removes elemental mercury by converting it to calomel. It is widely used in the base metals industry. This paper gives examples of the application, and also outlines the background for the development of the process. Another technology developed by Boliden for mercury removal is the Selenium filter. This is a fixed bed filter based on the reaction between elemental gaseous mercury and selenium, by which mercury selenide, HgSe, is formed. The mercury selenide is a highly stable compound with extremely low solubility in water and very low vapour pressure. Mercury is removed selectively without any side reactions. Special applications for the selenium filter technology include mercury removal from gases containing sulphur dioxide and geothermal off-gases.

11:00 AM Invited
Extraction and Recovery of Mercury from Concentrated Sulfuric Acid Streams Using Molecular Recognition Technology: Steven R. Izatt[1]; Neil E. Izatt[1]; Ronald L. Bruening[1]; *John B. Dale*[1]; Petrus J. Cilliers[2]; [1]IBC Advanced Technologies, Inc., 856 E. Utah Valley Dr., American Fork, UT 84003 USA; [2]Molecular Recognition Technology Africa (Pty), Ltd., Park Place E., 104 N. Rand Rd., Hughes, Boksburg 1460 S. Africa

IBC Advanced Technologies, Inc. (IBC) has developed a mercury selective product, which can be used by sulfuric acid producers as either a polisher, or as the primary technology for mercury removal from sulfuric acid. The Molecular Recognition Technology (MRT) separations material selectively binds Hg2+, and to a lesser extent Hg+, out of concentrated sulfuric acid. Mercury output levels of less than 0.1 ppm can be attained by the MRT system. A number of pilot plant tests have been successfully conducted at a number of base metal smelting operations. A large scale commercial plant is due for commissioning in 2003. Results from these projects will be discussed.

11:25 AM Invited
Treatment of Smelter Acid Plant Blow-Down Sludge: Ron Knott[1]; *Roshan Bhappu*[1]; [1]Mountain States R&D International, 13801 E. Benson Hwy., Vail, AZ 85641 USA

This paper describes a novel and proprietary treatment for the recovery of mercury, lead and associated by-products from the acid plant blow-down sludge. The proposed flow sheet includes a hydrometallurgical process for first recovering the contained mercury by an oxygen pre-treatment step in conjunction with the chlorine leach. The mercury is then recovered from the pregnant solution by cementation, first with iron to precipitate copper and then with aluminum powder to precipitate Hg, Au, Ag, etc. The leach residue, after Hg removal, is then leached with NaSH to convert lead oxides and sulfates to synthetic galena which is then floated off into a marketable product. The proposed process appears to be economically attractive and environmentally friendly.

Microstructural Processes in Irradiated Materials: Stainless Steels, Radiation Induced Segregation, Ion Irradiation and Other Materials I

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

Wednesday AM Room: 11A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Maria Jose Caturla, Lawrence Livermore National Laboratory, Chem. & Matls. Sci., Livermore, CA 94550 USA; Rodney Ewing, University of Michigan, Nucl. Eng. & Radiologl. Scis., Ann Arbor, MI 48109 USA

8:30 AM Invited
Radiation Effects in Nuclear Waste Glasses: *Rodney C. Ewing*[1]; Kai Sun[1]; Lumin Wang[1]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., Cooley Bldg., Ann Arbor, MI 48109 USA

Glasses used as nuclear waste forms will suffer radiation damage from the beta decay of fission products and the alpha decay of actinide elements. Since the ultimate radiation damage of a crystalline material is amorphization and glasses are in amorphous state, there is a misconception that glasses are radiation ìresistantî. However, experiments with neutrons, ions and electrons have demonstrated various radiation damages in glasses of various compositions. The typical radiation damage observed in glasses include: swelling associated with bubble formation, secondary phase precipitation due to radiation-induced chemical segregation, and increased leach rates of damage cascades. This talk will summarize our knowledge in radiation effects in glasses and present our most recent experiment results on this subject. In our experiments, the alpha and beta decay events in phosphate and silicate glasses are simulated with electron and ion beam irradiations at various temperatures. Advanced analytical and high resolution transmission electron microscopy (TEM) techniques are used to study the microstructural-structural and microchemical evolution of the glasses. These techniques include in situ TEM during energetic beam bombardment at various temperatures, electron energy loss spectroscopy (EELS), high angle annular dark field z-contrast scanning TEM (HAADF STEM), energy filtered and fluctuation TEM which provides additional information beyond the first-order pair statistics obtained from diffraction techniques. The results will be compared to radiation effects observed in crystalline ceramics.

9:15 AM
Ion-Irradiation of Electronic Materials: Defects and Microstructures: *Jim S. Williams*[1]; [1]Autralian National University, Canberra, ACT 0020 Australia

After a brief review of ion disordering processes in semiconductors, this presentation focuses on some specific defect and microstructural issues of importance in electronic and optoelectronic applications. Two aspects of silicon irradiation are treated. Firstly, ion irradiation and consequential lattice displacements can lead to a spatial separation of excess interstitials and vacancies. In some cases, annealing of ion implanted silicon can result in the coalescence of such point defects into both extended defects of interstitial character and voids. Secondly, larger voids, or nanocavities, can be produced by hydrogen or helium ion irradiation of silicon and these metastable defects can exhibit some interesting properties, including the trapping of defects and fast diffusing impurities. In addition, the disordering of compound semiconductors is also treated since ion disorder can influence and prohibit the application of ion implantation in these materials for the fabrication of devices. Selected disordering behaviour in GaN and ZnO is examined, particularly the ability of these materials to dynamically anneal displacement damage during irradiation. Some intriguing microstructures (e.g. aligned defect arrays and porous structures) are also observed in these materials. Finally, ion-disordered semiconductors can exhibit unusual deformation behaviour during nanoindentation measurements. Some phase transformation effects in silicon and dramatic structural changes in GaN are also illustrated.

10:00 AM Break

10:30 AM
Microstructure of Alumina Irradiated with Energetic Ions Under Applied Electric Fields: *Toru Higuchi*[1]; Kazuhiro Yasuda[1]; Kuninori Tanaka[1]; Kenichi Shiiyama[1]; Chiken Kinoshita[1]; [1]Kyushu University, Dept. of Appl. Quantum Physics & Nucl. Eng., Hakozaki 6-10-1, Higashi-ku, Fukuoka Japan

Insulating ceramics for fusion reactors are expected to be irradiated with neutrons and ions under electric fields. The electric field influences the kinetics of point defects in those materials. In the present study, we have irradiated alumina with 100 keV He-ions at 760 and 870 K under electric fields of 100 and 300 kV/m to a displacement damage of around 0.5 dpa and observed microstructure changes by using transmission electron microscopy. The electric field suppresses the formation of interstitial-type dislocation loops and enhances their growth in thinner specimens than 300 nm. A higher fraction of interstitials are found to annihirate at the surface under the electric fields, comparing those without the field. The effect of electric fields on the kinetics of point defects is discussed in terms of the interaction between charged defects and electric fields.

10:50 AM
Annealing Stages in Neutron-Irradiated Austenitic Stainless Steels: *Edward Paul Simonen*[1]; Dan J. Edwards[1]; Bruce W. Arey[1]; Stephen M. Bruemmer[1]; Jeremy T. Busby[2]; Gary S. Was[2]; [1]Pacific Northwest National Laboratory, Matls., PO Box 999, MS P8-15, Richland, WA 99352 USA; [2]University of Michigan, Nucl. Eng. & Rad. Scis., 2355 Bonistal Blvd., Ann Arbor, MI 48108 USA

Post-irradiation annealing kinetics in neutron-irradiated austenitic stainless steels exhibit distinct stages in spite of the fact that the detectable microstructure consists dominantly of a distribution of Frank loops and no other significant component of damage. Three distinct annealing stages measured in microstructure and hardness recovery and comparison with model predictions suggest that neutron and proton radiation damage is more complex than is evident in characterization of Frank loop size distributions. Identified stages include a component susceptible to short-term annealing, a component consistent with predicted Frank loop dissolution and a component resistant to longer-term annealing. Specimens of solution-annealed 304SS and 316SS were irradiated at 275C in a light-water reactor to 1 and 5 dpa. Transmission electron microscopy established distributions of Frank loops that extended down to 0.5 nm in diameter. This work was supported by the Materials Sciences Branch, BES, US Department of Energy, under Contract DE-AC06-76RLO 1830.

11:10 AM
Radiation Damage: What Can We Learn from Nanocrystalline Metals: *Max Victoria*[1]; [1]EPFL-CRPP, Fusion Tech. Matls., CH-5232 Villigen-PSI 5232 Switzerland

The volume density of grain boundaries in nanocrystalline (nc) materials is high and defects produced by displacement cascades are expected to be easily absorbed into such sinks. In the present contribution we analyze results from molecular dynamics simulations (MD) in Ni of different grain sizes and for different PKA energies and compare them to experimental observations, in terms of the type of defect produced, their density and the mode of absorption at the grain boundaries. The original microstructure of the nc Ni has been well characterized, particularly in terms of the presence of dislocations, so that the observed radiation hardening will be discussed both in terms of possible dislocation mechanisms or the relaxation at the grain boundaries (A. Hasnaoui, H. Van Swygenhoven and P. Derlet, accepted for publication Acta Mater. (2002)) introduced by the absorbed defects.

11:30 AM Cancelled
Perspective on Experimental and Computer Simulation Studies of Radiation Effects in Ceramics: *William J. Weber*[1]; Fei Gao[1]; L. Rene Corrales[1]; Weilin Jiang[1]; Ram Devanathan[1]; Constantin Meis[2]; Alain Chartier[2]; Yanwen Zhang[3]; [1]Pacific Northwest National Laboratory, Fundl. Sci. Direct., PO Box 999, MS K8-93, Richland, WA 99352 USA; [2]CEA-Saclay, Direction de liEnergie NuclÈaire, DPC/SCPA B,t 450 Sud., Gif-sur-Yvette 91191 France; [3]Uppsala University, Div. of Ion Physics, Angstrom Lab., Uppsala Sweden

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

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

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

Wednesday AM Room: 12
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Michael R. Notis, Lehigh University, Dept. of Matls. Sci. & Eng., Bethlehem, PA 18015 USA; Sinn-Wen Chen, National Tsing-Hua University, Dept. of Cheml. Eng., Hsinchu 300 Taiwan

8:30 AM Invited
Calculated Phase Equilibria for Solder Alloys: *Ursula R. Kattner*[1]; [1]NIST, Metall. Div., Stop 8555, 100 Bureau Dr., Gaithersburg, MD 20899 USA

Phase equilibria information is needed for the evaluation of melting behavior and possible effects of contamination by other elements for candidate solder alloys. Parameterization of phase equilibria data by thermodynamic modeling allows compact storage and retrieval of this information for any composition or temperature of interest. A thermodynamic database for solder alloys has been assembled for 7 components: Sn, Ag, Bi, Cu, In, Pb, Sb. For the construction of this database, available thermodynamic assessments of the constituent binary and ternary systems were reviewed for consistency and, when necessary, individual phase descriptions were revised. One important criterion for the evaluation of these descriptions is the requirement that calculated reaction temperatures need to be known with relatively high accuracy. A summary of the evaluation of available thermodynamic descriptions and results from assembling this database will be discussed.

8:50 AM Invited
Development of Thermodynamic Database on Micro-Solders and Cu Base Alloy Systems: Xing Jun Liu[1]; Ikuo Ohnuma[1]; Cui Ping Wang[1]; Ryosuke Kainuma[1]; *Kiyohito Ishida*[1]; [1]Tohoku University, Matls. Sci., Grad. Sch. of Eng., Aoba-yama 02, Sendai 980-8579 Japan

Recent progress on the database of calculated phase diagrams in micro-solders and Cu-base alloy systems, useful for the development of Pb-free solders and the interfacial phenomena between solder and Cu substrate in electronics packaging technology, is presented. A thermodynamic tool, ADAMIS (Alloy Database for Micro-Solders), is based on comprehensive experimental and thermodynamic data accumulated with the CALPHAD (Calculation of Phase Diagrams) method and contains 8 elements of Ag, Bi, Cu, In, Sb, Sn, Zn and Pb. It can handle all combinations of these elements and all composition ranges. The element of Al is also added to ADAMIS within a limited range of compositions. Furthermore, the database of Cu-base alloys including the Cu-X binary system and Cu-Fe, Cu-Ni, Cu-Cr base ternary systems, has also been developed. Typical examples of the calculation and application of these databases will be presented.

9:10 AM Invited
Assessment of Phase Equilibria in Ag-Cu-Sn Near Eutectic System: *Choong-Un Kim*[1]; Jaeyong Park[1]; Ted Carper[2]; Puligandla Viswam[2]; [1]The University of Texas at Arlington, Matls. Sci. & Eng., Woolf Hall, #325C, Arlington, TX 76019-0031 USA; [2]Nokia Mobile Phones, Rsrch. & Tech. Access, 6000 Connection Dr., Irving, TX 75039 USA

Accurate assessment of phase equilibria is one of the most critical tasks in developing and applying new engineering alloys. Phase equlibria of the Ag-Cu-Sn (ACS) alloy system, a potential replacement for Pb-Sn solder alloys, however, is not well known, especially in near-eutectic compositions. The difficulty in assessing phase equilibria of the near-eutectic ACS system stems from the fact that multiple phase fields exist within a very narrow range of composition and temperature, making them difficult to resolve by ordinary experimental techniques. This study presents the near-eutectic phase equilibria of the ACS system as determined by a selective cooling method in conjunction with theoretical calculation. Selective cooling induces growth of the equilibrium solid phase at a given temperature and forces the remaining liquid phase solidify into a finer microstructure by rapid cooling for better identification of phase equilibria.

9:30 AM
Soldering-Induced Cu Diffusion and Intermetallic Compound Formation Between Ni/Cu UBM and SnPb Flip Chip Solder Bumps: *Chien Sheng Huang*[1]; Jenq Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 101 Sec., 2 Kuang Fu Rd., Hsinchu 300 Taiwan

Ni-based under bump metallization has been widely used as a diffusion barrier to prevent the rapid reaction between Cu conductors and Sn-based solders. In this study, electroplated Ni with different thickness was employed to evaluate the interfacial reaction after reflow, annealing and aging between Ni/Cu UBM and eutectic Sn-Pb solders in the 63Sn-37Pb/Ni/Cu/Ti/Si3N4/Si multiplayer structure. In the as-reflowed sample, an 1-mm (Ni1-x,Cux)3Sn4 IMC was found between solders and Ni/Cu UBM. During multiple reflow or annealing at 240∞C for a period of time, another type of IMC with the form (Cu1-y,Niy)6Sn5 was found between solders and (Ni1-x,Cux)3Sn4, whereas the thickness of (Ni1-x,Cux)3Sn4 was still around 1 mm. However, after aging at 150∞C for 1000 hours, the thickness of (Ni1-x,Cux)3Sn4 IMC increased to 3mm, yet no (Cu1-y,Niy)6Sn5 was revealed. It is argued that formation of (Cu1-y,Niy)6Sn5 IMC was due to the Cu diffusion through (Ni1-x,Cux)3Sn4 IMC into solders along with the further reaction with Ni and Sn. The degrees of Cu diffusion could be correlated to the heat treatment conditions and morphological evolution of (Ni1-x,Cux)3Sn4 IMC.

9:45 AM
Computational Model for Multi-Component Base Metal Dissolution and Intermetallic Compound Formation with Molten Solder Alloys: *Kenneth L. Erickson*[1]; Polly L. Hopkins[1]; Paul T. Vianco[1]; Jerome A. Rejent[1]; [1]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185 USA

Results from experiments examining multi-component base metal dissolution by molten solder alloys were reported previously. Preferential dissolution of base metal constituents appeared to influence short-term base metal erosion and long-term inter-metallic compound (IMC) growth. Experiments with 76Au-21Pt-3Pd (wt%) alloy sheet and molten 63Sn-37Pb solder indicated that preferential dissolution of Au produced Pt-rich IMC layers that could limit base metal erosion and cause induction periods observed during subsequent IMC growth. To examine these issues quantitatively and develop predictive tools for process and long-term reliability analyses, a general model describing multi-component dissolution and IMC growth was developed based on solid-solid and liquid-solid reactions coupled with solid-phase and liquid-phase multi-component diffusion. The resulting equations now require phase-boundary conditions involving time-dependent concentration and flux discontinuities, rather than the constant-concentration ìjumpsî used with binary systems. This paper summarizes the multi-component model, its implementation in previously developed codes, and comparisons between calculated and experimental results.

10:00 AM Break

10:25 AM Invited
Directional Solidification of AgCuSn Eutectic Alloys: *Daniel Joseph Lewis*[1]; [1]NIST, Metall. Div., 100 Bureau Dr., Stop 8555, Gaithersburg, MD 20899 USA

Directional solidification has been used to examine microstructures in AgCuSn, CuSn, and AgSn eutectic alloys. The eutectic alloy in this system is of industrial importance as a replacement solder alloy for tin-lead. Using directional solidification it is possible to study solidifi-

cation rates relevant to soldering processes as well as solidification rates on the order of one micrometer per second. Microstructures collected over a range of solidification rates will help to understand the presence of a large volume fraction of tin dendrites seen in industrially assembled solder joints as well as provide information on the solidification velocities where three phase coupled growth is possible. Microstructures and quantitative image analysis will be presented for the three alloys considered.

10:45 AM Invited
Fluxless Plasma Soldering of Pb-Free Solders on Si-Wafer: *Jae-Pil Jung*[1]; Joon-Kwon Moon[1]; Soon-Min Hong[2]; [1]University of Seoul, Dept. of Matls. Sci. & Eng., 90 Cheonnong-dong, Dongdaemun-gu, Seoul 130-743 Korea; [2]Samsung Electronics Company, Ltd., Micro-Joining Lab., Inst. of Intelligent Sys., Mechatronics Ctr., 416 Maetan-3dong, Paldal-gu, Suwon City, Kyungki-do 442-742 Korea

In the field of electronic industry, a soldering flux is inevitable to get a reliable solder joint. However, with growing concerns about environment, the flux is avoided occasionally for environmental or technical problems. Flux residue on the fine-pitch device cannot be removed easily and may cause corrosion on the joint. For this reason many researchers have interests in fluxless soldering process. On this study fluxless solderability of Sn-3.5Ag, Sn-3.5Ag-0.7Cu and Sn-37Pb alloys were investigated. The solder balls were set on a UBM-coated Si-wafer to achieve fluxless reflow soldering in plasma. Ar-10%H2 plasma was used for the experiment. In the fluxless plasma reflow, the self-alignment of solder bumps and intermetallic compound on the interface were observed.

11:05 AM
Electromigration of Sn95/Sb5 Flip Chip Solder Bumps on Cr/Cr-Cu/Cu Under Bump Metallization: *T. L. Shao*[1]; Chih Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Eng., 1001 Ta Hsueh Rd., Hsinchu 30050 Taiwan

Increasing awareness of environmental and health impact of electronic materials has highly demanded the lead-free product of IC package recently. Lead-free solder of Sn95/Sb5 with low α particle property was used to avoid the soft error of memory IC flip chip package. The electromigration behavior of Sn95/Sb5 flip chip solder bumps was studied. The under bump metallization (UBM) on the chip side was Cr/Cr-Cu/Cu, and the metallurgy layer on the substrate side was Ni/Au. The chip was mounted on a BT substrate and then filled with underfill. The flip chip package was stressed at the current density of 2 * 10E4 A/cm^2 at 100°C. The current crowding effect and the stressing polarity effect were examined. Void formation on the cathode side was observed. The failure of the rerouting and thin film structure on chip side were examined.

11:20 AM
Relationship Between Mechanical Properties and Intermetallic Compound Formation at the Sn-0.7(0.5, 0.3, 0.1)wt%Cu/Ni Joints: *Sinn-Wen Chen*[1]; Shou-Wei Lee[1]; Chao-Hong Wang[1]; Ming-Chuen Yip[2]; [1]National Tsing Hua University, Dept. of Cheml. Eng., #101 Sec., 2 Kuang-Fu Rd., Hsin-Chu 300 Taiwan; [2]National Tsing Hua University, Dept. of Power Mechl. Eng., Hsin-Chu 300 Taiwan

Sn-0.7wt%Cu is one of the most promising Pb-free solders. However, the compound formation at the solder/Ni joint is very sensitive to the Cu amounts in the solders. This study determined intermetallic compound formation in the Sn-0.7(0.5, 0.3, 0.1)wt%Cu/Ni joints at 200°C and 250°C and also the tensile properties of these joints. At 250°C, only Ni3Sn4 phase was found in the Sn/Ni, Sn-0.1wt%Cu/Ni, Sn-0.3wt%Cu/Ni couples; while Cu6Sn5 was found in the Sn-0.5wt%Cu/Ni and Sn-0.7wt%Cu/Ni couples. The critical compositions for the Cu6Sn5 formation were determined to be 0.4wt%Cu and 0.1wt%Cu at 250°C and 200°C, respectively. Both Ni3Sn4 and Cu6Sn5 phases were found in the solid/solid couples (200°C), while the Ni3Sn4 was hardly detected in the solid/liquid couples (250°C) when the Cu6Sn5 existed. No significant differences of tensile properties were observed for these Sn-Cu/Ni joints made with solders of various Cu amounts, even if the intermetallic compounds formed at the interfaces were different.

11:35 AM
Interfacial Segregation of Bismuth in Pb-Free Solder Interconnects on Copper Metallization: P. L. Liu[1]; C. Z. Liu[2]; A. P. Xian[2]; *J. K. Shang*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA; [2]Chinese Academy of Sciences, Inst. of Metals Rsrch., Shenyang Natl. Lab. for Matls. Sci., 72 Wenhua Rd., Shenyang 110016 China

Bismuth is used as a major alloying element in Pb-free solder alloys because it stabilizes tetragonal tin and reduces the melting temperature of tin-base alloys. In this study, microchemistry of the interface in a tin-bismuth solder interconnect was examined by scanning Auger microscope equipped with an in-situ fracture stage. Segregation of bismuth was found on the interface between copper and copper-tin intermetallic phase when a copper/tin-bismuth solder interconnect was thermally aged. The segregation was confined to about a couple of atomic layers from the interface, resulting in embrittlement of the interface. Aging conditions for Bi-induced interfacial embrittlement were systematically determined.

Products, Applications, and Services Showcase: Furnaces & Casting Technology

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: David V. Neff, Metaullics Systems Company, Solon, OH 44139 USA

Wednesday AM Room: 5A
March 5, 2003 Location: San Diego Convention Center

Session Chair: TBA

8:30 AM
Furnaces for the New Business Trends: *Jarda Urbanek*[1]; Dirk Menzler[2]; [1]Junker, Inc., 2015B Rte. 34, Oswego, IL 60543 USA; [2]Otto Junker GmbH, PO Box 1180, Simmerath 52417 Germany

Brief intoduction of our company and services provided,followed by an overview of several heat treating furnace designs for various semifinished products. Furnace design emphasis is on varying lot size and product dimensions for either the strips, plates, coils, ingots or billets. Design features to support this business trends will be pointed out. Presentation of typical cycle times, efficiencies and temperature tolerances from actual process data for various products will be discussed.

8:50 AM
Heat Treating and Thermal Processing for the Aluminum Industry: *Thomas Zamanski*[1]; [1]LOI, Inc., 333 Technology Dr. S. 109, Canonsburg, PA 15317 USA

Today, LOI, Inc. continues to be a leader in heat-treating and thermal processing equipment with a large selection of equipment designs. Long recognized in the Steel Industry, LOI is making its presence felt in the Aluminum Industry as well, with its melting and heat-treating technology. Currently, the company is marketing technology in the following areas: -A Solution Anneal and Age Hardening Line for Aluminum wheels -Single coil Annealing Furnaces for Aluminum Strip or Foil -Twin Chamber Melting Furnace for Recycling Aluminum Scrap. LOI is able to perform projects that are on the cutting edge of technology because of the research and development team that is supported by the parent of the LOI Group, LOI Thermprocess. This relationship has allowed LOI, Inc. to remain technically strong enough to meet the demands of the North American market, yet flexible enough to handle both large and small projects.

9:10 AM
Recent Technological Advancements in Electro-Magnetic Pumping Solutions for the Aluminium Industry: *Alan Peel*[1]; [1]EMP Technologies Ltd., Beedes House Easton Ave., Stretton Burton-on-Trent, Staffordshire VE13 OVB UK

The presentation will discuss new enhancements to the EMP System particularly focusing on the new generation of vortex system based on the LOTUSS principles for ultra light gauge scrap submergence at very high charging rates in excess of 20,000 lbs/hour. Results of the improvements gained by clients following installation of the new vortex will be discussed. Combining metal transfer and de-gassing as an integral part of the circulation and charging system within the EMP System will also be discussed. In addition new developments based around the electro-magnetic pump for different applications from small die casting operations with furnace capacities less than 20,000 lbs allowing these operations to recycle in house their own scrap arisings and carry out their own efficient alloying.

9:30 AM
Advanced Monolithic Refractories for Lining Aluminium Melting and Holding Furnaces: *Duncan Jones*[1]; [1]Thermal Ceramics, Tembay Rd., Bromborough, Wirral CH62 3PH UK

Monolithic refractories are now becoming more widely used in aluminium melting and holding furnaces. In particular, specialist high strength castables with non wetting additives have been developed which are now replacing traditional brick linings in these furnaces. Thermal Ceramics, have focused their efforts on developing a range of

specialist monolithic refractories to meet the necessary demands placed on refractory linings in modern aluminium furnaces. This paper reviews the various Tri-Mor monolithic refractories available for a complete aluminium furnace lining and in particular details the properties and application of some aluminium contact castables. These materials have been designed to be non-wetted by aluminium and resistant to corundum growth even under extreme furnace operating conditions. Additionally a range of maintenance materials suitable for both hot and cold repairs is discussed.

9:50 AM

High Temperature Alternatives to Refractory Ceramic Fiber (RCF): *Lance J. Caspersen*[1]; [1]Thermal Ceramics, 2102 Old Savannah Rd., Augusta, GA 30906 USA

In recent years there have been some discussion about the health and safety aspects of refractory ceramic fiber or RCF, particularly among aluminum companies. Although Thermal Ceramics, the largest producer of RCF material in the world, does not take the stance that these products are harmful if handled properly, we recognized the desire by some companies for alternative materials. Through a research project that began in the early 80ís, our company introduced a high temperature, non-RCF insulating product called Superwool 607 with low biopersistance, or capability to persist in the body. Thermal Ceramics has since developed many other innovations in product chemistries, capabilities and forms in these class of amorphous wool materials. This presentation gives a broad overview of the health and safety matters related to RCF and Superwool products, the various high temperature Superwool products available and the many applications that theyíve been used for.

10:10 AM

The Real Cost of Alloying in the Casthouse: *Edgar C. Burhop*[1]; Paul Cooper[2]; [1]Metallurg Aluminium, PO Box 768, Newfield, NJ 08344 USA; [2]Metallurg Aluminium, Fullerton Rd., Rotherham, S. Yorkshire S60 1DL UK

The cost of alloying in the casthouse is generally poorly defined, and the process of alloying is not always given the consideration that it deserves. Too much focus is usually placed on the price of an alloying additive, and not enough on other factors that affect the true cost of alloying. This paper presents these issues and gives an insight into the dissolution processes occurring in the furnace, and problems associated with different addition methods. A review is made of why poor elemental recoveries are sometimes observed. Recommendations for best alloying practice are also provided. The report concludes that the real cost of alloying is very complicated and not readily defined. Decisions on which alloying method to adopt, therefore, require studied consideration, and must be based on a close study and understanding of the melting and casting operations and the alloying methods available.

10:30 AM Break

10:50 AM

Recent Developments in the Application of Brochotís Casting Wheel in Ingot Production: *Jean-Jacques Grunspan*[1]; [1]Brochot SA, 52 Ave. Marcel Paul, Tremblay en France, Cedex 93297 France

Tomago has retro-fitted Brochotís advanced casting wheel to achieve both increased throughput and high quality, dross free ingots. The wheel eliminates the oxidation that causes dross by reducing turbulence at all critical points. This means that dross is not just hidden, but really absent. To meet Tomagoís desire of increasing capacity, the wheel has been slightly redesigned to ensure operation at 27 tonnes per hour. Other advantages include constant casting level in the moulds and the automatic lifting of both wheel and launder to avoid spillage of molten metal in the event of a stoppage downstream or an unstripped ingot. Other development already achieved in ingot production will also be detailed.

11:10 AM

The Manufacturing, Use and Plant Test Results of TF Combo Bags for DC Sheet Ingot Casting: *Sylvain Tremblay*[1]; M. Ruel[2]; [1]Pyrotek High Temperature Industrial Products, Inc., 1623 Manic St., Chicoutimi, Quebec G7K 1G8 Canada; [2]Pyrotek High-Temperature Industrial Products, Inc., 2400 Blvd. Lemire, Drummondville, Quebec J2B 6X9 Canada

In the last decade, the molten aluminum distribution in the ingot heat of dc sheet casting ingots has been achieved using mostly a combo bag made of fiberglass fabrics. Some of these fabrics are open-weave materials while others are solid fiberglass fabrics sewn together. This bag is usually soft and flexible and extensive sewing operations are required to manufacture it. Usually the bag deforms and this can affect not only the distribution but also the molten metal temperature profile around the mold and at the end, the final ingot quality. This paper will review the production and use of a new combo bag for dc sheet ingot casting. The paper is divided in two parts. The first part will deal with the manufacturing of the TF combo bag (acronym for thermal formed). Description of the fabrics used as well as the new production techniques to manufacture that bag will complete this first part. Part two (2) will present the test results of the TF combo bag used at different plants to cast AA-1045, AA-3003 and AA-5052 alloys. The following variables and measures will be discussed: Cast start-up, actual metal flow observations, ingot surface finish, temperature profile around the mold, and finally, results from the rolling plant. Comments will also be presented from an operation point of view using this new molten aluminum distributor.

11:30 AM

Conform TechnologyñDevelopments in Feedstock Materials: *Phillip M. Thomas*[1]; [1]Holton Conform, Business Dvlp. & Tech., Albany House Elliott Rd., W. Howe, Bournemouth, Dorset BH11 8JH UK

The Conform process is an established technology for the production of a wide range of end products from a range of non ferrous materials. The feedstock material has traditionally been rod which has been continuously cast and rolled although for some products - notably grain refining rod - the feedstock has been continuously cast bar. Over the past few years the range of alloys that can be processed has been extended and 2xxx and 7xxx alloys have been successfully processed as well as a number of composite materials. The type of feedstock materials has also been increased and now includes powders, granules and a variety of other particulate materials. The presentation will describe these developments by means of a series of case studies.

11:50 AM

Wagstaff Direct Chill Casting Update: *Craig Johnson*[1]; [1]Wagstaff, Inc., 3910 N. Flora Rd., Spokane, WA 99216 USA

Wagstaff will provide an update on the a latest developments on our ingot casting technology, billet casting technology, and automated casting controls.

12:10 PM

Advanced Welding Techniques for Aluminum Alloys: *Menachem Kimchi*[1]; [1]Edison Welding Institute, Resistance & Solid-State Welding, 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

This paper describes two new joining techniques for joining aluminum. One technique, called magnetic pulse welding (MPW), is applicable to joining of aluminum sheet. These processes both offer numerous advantages over conventional arc welding processes especially in automotive and aerospace applications. MPW is a very high-speed process which produces solid-state (cold) welds with minimal distortion and degradation to mechanical properties, offers potential for significant manufacturing cost reduction through increased weld speed, and is very good for welding dissimilar metal combinations (i.e., aluminum to steel). Conductive heat seam welding (CHRSEW) is a resistance welding process that offers the potential to reduce welding cost through increased welding speed, reduced joint preparation and consumable cost, the ability to successfully weld difficult-to-weld aluminum alloys such as the 7xxx series alloys, the ability to join dissimilar sheet thickness, and to produce good mechanical properties compared to arc welding. This paper will highlight process details and variables, mechanical properties, and potential applications for both welding techniques.

Recycling - General Sessions: Aluminum

Sponsored by: Light Metals Division, Jt. LMD/EPD-Recycling Committee

Program Organizers: Han Spoel, Spalco Metals Inc., Toronto, Ontario M5R 1W8 Canada; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

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

Session Chair: Han Spoel, Spalco Metals Inc., Toronto, Ontario M5R 1W8 Canada

8:30 AM Announcements

8:45 AM

Determining Recyclability, A Working Method: *W. Bryan Steverson*[1]; Donald L. Stewart[2]; [1]Alcoa Inc, 2300 N. Wright Rd., N290, Knoxville, TN 37701-3141 USA; [2]Alcoa Inc., Casting Tech. Div., Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

How do you determine recyclability? When a customer approaches and asks if their change, addition, coating modification, etc. affects the recyclability of your product, how do you answer? How are concerns about safety, hygiene, hazardous contaminants, melt loss, cost, metal quality, and operational issues reviewed and answered? How are these ongoing questions answered in a thorough manner which both protects the recycler while providing the supplier the necessary flexibility to grow and improve their product. The purpose of this paper is to identify the concerns and discuss one current method in place within a UBC recycling environment.

9:15 AM

The Secondary Metal Supplier and Foundry Metal Quality: *Ray D. Peterson*[1]; [1]IMCO Recycling, Inc., 397 Black Hollow Rd., Rockwood, TN 37854-4021 USA

Foundry customers can purchase ingot or molten metal from many metal supplier sources. The primary concern of the customer should be receiving an alloy with a composition within the specified limits. If the chemical limits are correct, the source of the elements should not matter. Overly tight compositional specifications can lead to higher material costs. Once compositional concerns are met, other customer concerns should include hydrogen and alkali metals concentrations, and inclusions (both the type of inclusion and the quantity). Foundry metal from secondary metal sources can be equivalent to or superior to primary metal sources on the basis of both chemistry and impurities. The melting equipment used for processing the secondary metal sources can have a significant impact on the metal quality. Care must also be taken at the customerís plant not to degrade metal quality once it is received. Every plant producing castings should practice proper handling and treatment of molten metal to ensure quality and reliability.

9:45 AM

Controlled Melting of Secondary Aluminum in Rotary Furnaces: *Henrik Gripenberg*[1]; [1]Linde Gas Division, Lidingˆ S-181 81 Sweden

Secondary plants strive to optimize the cost of scrap raw materials. However lower cost scraps contain more organic contaminations like lacquer and oils. These cause emissions that have a negative influence on productivity and environmental conditions thus limiting the amount of organic components that can be allowed in the charge. By using controlled melting and optimized hardware solutions the negative impact of organic contaminations can be reduced and the economy of the melting is improved. The new WASTOX process involves controlled oxygen lancing and burner management for combustion of the emissions inside the rotary furnace. The composition of the flue-gas is analyzed using a new laser technology concept and is together with other parameters used for on-line process control. The WASTOX process was installed at the new Universal Rotary Tiltable Furnace (URTF) plant at Stena Aluminium AB. The paper will discuss theory, hardware solutions and industrial results from the WASTOX concept and the URTF plant.

10:15 AM Break

10:30 AM

Gases Evolved During Decoating of Aluminium Scrap in Inert and Oxidizing Atmospheres: *Anne Kvithyld*[1]; Thorvald Abel Engh[1]; Piotr Kowalewski[3]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., Trondheim N-7491 Norway; [3]Warsaw University of Technology, The Fac. of Matls. Sci. & Eng., Warsaw 00-661 Poland

Thermal removal of coatings on aluminium scrap in a delacquering unit prior to melting makes it possible to lower the emissions and utilize the energy released by the combustion process. Polyester coated on aluminium was studied using both inert and oxidizing atmospheres. The evolved gases were registered using quadrupole mass spectroscopy (MS) during the coat decomposition in a thermogravimetric/differential thermal analysis (TG/DTA) furnace. Ion gas intensity scans collected during the main weight losses are presented, together with the enthalpy changes. The losses, degradation products and enthalpy produced depend both upon the atmospheres and the heating rates. The data have been used to develop kinetic models of pyrolysis and combustion of the coating. These can be used to choose suitable atmosphere and temperature profiles in industrial decoating units so as to reduce and control emissions.

11:00 AM

A Study on Titanium Hydride Formation of Used Titanium Scrap: *Bo Young Hur*[1]; *Duck Kyu Ahn*[1]; *Sang Youl Kim*[1]; *Hiroshi Arai*[1]; *Soo Han Park*[1]; [1]Gyeongsang National University, ULSFoM-NRL, ReCAPT, Div. of Matl. Eng., 900, Kajoa-Dong, Chinju, Kyungnam 660-701 S. Korea

Metallic hydride is often used as foaming agent during fabrication of porous metal. Most of the titanium hydride made from sponge Ti, but in this study, It made from scrap Ti to reduce the cost. Scrap Ti with various shapes such as plate, chip, and bulk etc. was hydride in self-made apparatus and compared with the standard sample made from sponge Ti. The temperature of hydrogenation was about 400° in the case of pure sponge titanium, but return scrap titanium alloy were increased. The hydrides of titanium alloy were crushed by ball mill for 3 hours. It was determined by heating and cooling curve in reaction chamber. The result of XRD was titanium hydride peak only that it was similar to pure titanium.

11:30 AM

In Situ, Real Time Measurment of Aluminum Melt Chemistry: *Robert De Saro*[1]; Arel Weisberg[1]; Joseph Craparo[1]; [1]Energy Research Company, 2571-A Arthur Kill Rd., Staten Island, NY 10309 USA

Energy Research Company is developing an instrument to measure the chemistry of molten aluminum. Termed LIBS, for Laser Induced Breakdown Spectroscopy, the probe can measure in situ and in real time the elemental concentrations of aluminum. The laser, operating in the visible spectrum, is repetitively fired through a fiber optic cable, which is placed in the melt via a proprietary probe. A small amount of melt absorbs the laser light and is rapidly ionized. The returning light is resolved by a spectrometer, which uniquely identifies the elements in the melt. The light amplitude determines the concentration. Laboratory experimental results have confirmed that the concept works. Full-scale field tests at commercially operating plants will begin this year. This work was funded, in part, by the Department of Energyís Office of Industrial Technology.

Science and Technology of Magnetic and Electronic Nanostructures: Nanostructures in Functional Materials: Magnetic

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee

Program Organizers: Ramamoorthy Ramesh, University of Maryland, Department of Materials and Nuclear Engineering, College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Wednesday AM Room: 15A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Robert D. Shull, NIST, Magnetic Matls., Gaithersburg, MD 20899-0001 USA; Xiaoqing Pan, University of Michigan, Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109 USA

8:30 AM

Multi Component Functional Nanostructures: Fabrication and Local Properties: *Dawn A. Bonnell*[1]; [1]University of Pennsylvania, Matls. Sci., 3231 Walnut St., Philadelphia, PA 19104 USA

The potential to assemble dissimilar molecular or nanostructural elements into structures with complex functionality has motivated considerable activity in several scientific disciplines. The library of new molecules with electrical, optical or chemical activity grows quickly and now includes synthetic polypeptides, conjugated organic molecules, nanotubes, metallic and semiconducting nanowires, etc. However, functionality has been achieved in only a few select systems. In order to realize the potential inherent in nanodevices, methods of assembling a wide range of dissimilar elements and connecting the resulting complex structures must be developed. Furthermore, the processes must be informed by a knowledge of the properties of individual components. We report here a novel approach that utilizes local reactivity of ferroelectric surfaces due to variations in atomic polarization. It will be demonstrated that chemical reactivity is domain specific based on control of local electronic structure. When combined with chemical assembly nanostructures consisting of oxide substrates, metal nanoparticles, and organic/biological molecules can be fabricated. The procedure can be iterated to develop structures in which multiple types of metal particles, and electronically or optically active molecules can be assembled in predetermined configurations. New techniques for quantifying the local properties of nanostructures and even individual defects will be illustrated.

9:15 AM
Future Data Storage and Nanoscience: *Joachim Walter Ahner*[1]; [1]Seagate Technology, 1251 Waterfront Pl., Pittsburgh, PA 15222 USA

It is well appreciated, that as the size of material objects approaches nanometer dimensions, the materials structural and electronic properties change. This is related to a number of factors including quantum size effects and the enhancement of the surface/volume ratio with diminishing size. The investigation of these effects and the impact in future recording media will become crucial in the next future, when recording approaches Terabit/sq.inch densities. I will report about the development of two novel probing instruments for imaging, manipulation and analyzing nanometer scaled materials. I will propose concepts of applications for future data storage devices and present first results on ferromagnetic and ferroelectric recording media.

10:00 AM
Magnetic Properties of Lithographically Defined Nanomagnets: *Fernando J. Castano*[1]; Yaowu Hao[1]; Caroline A. Ross[1]; Bernhard Vogeli[2]; Henry I. Smith[2]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Cambridge, MA 02139 USA; [2]Massachusetts Institute of Technology, Dept. of Electl. Eng. & Compu. Sci., 77 Massachusetts Ave., Cambridge, MA 02139 USA

The present work reviews progress in the fabrication and characterization of sub-100 nm nanomagnets made using lithography techniques, assessing the possibility of integrating these small structures in magneto-electronic devices. The starting materials are evaporated or sputtered multilayer structures and arrays of nanomagnets, with a wide variety of different shapes, are defined using either subtractive or additive fabrication approaches. The magnetization reversal in compositionally modulated rectangular and ring shaped elements will be discussed. Issues such as the element variability, as well as the interaction among nanomagnets will be addressed. Furthermore, the magnetoresistance exhibited by these small magnets will be examined as a function of their shape.

10:45 AM
Magnetism, Structure and the Characterization of Antisite Disorder in the Predicted Half-Metals Co_2MnSi and Co_2MnGe: *J. A. Christodoulides*[1]; M. P. Raphael[1]; B. Ravel[1]; S. F. Cheng[1]; B. N. Das[1]; Q. Huang[2]; R. Ramesh[3]; G. A. Prinz[1]; V. G. Harris[1]; [1]Naval Research Laboratory, Washington, DC 20375 USA; [2]National Institute for Standards and Technology, Ctr. for Neutron Rsrch., Gaithersburg, MD 20899 USA; [3]University of Maryland, College Park, MD 20742 USA

Magnetoelectronic devices rely upon an imbalance in the number of majority and minority carriers to add additional degrees of freedom to traditional logic devices. The ideal magnetoelectronic device consists of ferromagnetic materials that exhibit complete spin polarization at the Fermi surface (i.e. half-metallic ferromagnets). One promising class of materials is the Heusler alloys, some of which have been predicted by first principles band theory to be half-metallic. Here we report on the magnetic, structural and electrical transport properties of Co_2MnSi and Co_2MnGe. We have studied single crystals, poly-crystalline arc-melted buttons and thin films grown by d.c. and r.f. magnetron sputter deposition. Samples have been characterized for antisite disorder by neutron diffraction and anomalous x-ray diffraction techniques. For Co_2MnSi, the thin films and arc-melted buttons have lattice constants, magnetic properties, and room temperature resistivities that approach those of the single crystal. On the other hand, the residual resistivity ratio, ρ_{300K} / ρ_{5K}, of these samples shows sharp contrasts with the single crystal; the crystal, the arc-melted button and the best thin films having ratios of 6.5, 2.7 and 1.4, respectively. Disorder studies on thin film and arc-melted samples show that the antisite disorder is typically zero for Mn-Si antisite disorder, but between 10% and 14% for Co-Mn antisite disorder. We postulate this to be the cause for the increased amount of defect-induced scattering in the electrical transport measurements of these samples. The results of a similar study on Co_2MnGe will also be presented.

Surface Engineering in Materials Science - II: Manufacturing via Surface Engineering

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

Wednesday AM Room: 7A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: John J. Moore, Colorado School of Mines, Dept. of Matls. Sci. & Eng., Golden, CO 80401 USA; R. Mishra, University of Missouri-Rolla, MSE, Rolla, MO 65409-0340 USA

8:30 AM Invited
Laser Surface Modification of Aluminum for Automobile Application: *J. Mazumder*[1]; J. Caroll[1]; Y. Lu[1]; J. Kelly[1]; [1]University of Michigan, Dept. of Mechl. Eng., & Matls. Sci. & Eng., 2350 Hayward St., Ann Arbor, MI 48109-2125 USA

There is a considerable interest in using Aluminum in automobile for reducing the weight of a car for improved fuel efficiency. This paper reports various laser surface modification applications for possible use in various engine and power train components. Laser Cladded Mn-Al Bronze coatings on cast alloy AA 333 were successfully produced using a 6kw continuous wave CO2 laser with Oscillating Beam. The coatings were dense, well bonded, free of cracks and relatively much harder with sufficient toughness. Copper based alloys can be used to improve resistance for pump and valve seats for Aluminum engine heads. Laser surface alloying of Iron on Aluminum cast alloys were also investigated for improved wear properties within the Aluminum engine bore. Preliminary evaluation of the selected coatings microstructures was carried out using optical and scanning microscopy, x-ray energy dispersive spectroscopy and x-ray diffractometry. It revealed columnar type grains of varying sizes, homogenous distribution of the alloying elements and FCC Cu-based solid solution with enhanced lattice parameters, depending on the processing condition. The coating/substrate interface microstructure was found to contain a-Al (FCC) and tetragonal Cu-Al2. The dry sliding tribological tests revealed encouraging results of 1/3 of the wear rate for coatings compared to the cast aluminum AA333 substrate. The coating was much less prone to seizure under identical test conditions of 10 lbs normal load at 600 rpm and 30.3 mm radius of sliding. Significant improvements of thermal diffusivity were reported for valve seats cladding compared to inserts. For Al-Fe alloys predominant phase were a-Aluminum solid solution with a range of distribution of Al-Fe-Si intermetallics. Improved wear resistance was also observed for this alloy.

9:00 AM
Formation of Metal-Ceramic Layer on Aluminiun Alloy (Al-12Si) by Laser Processing: *Dillibabu Sastikumar*[1]; M. Jamal Mohamed Jaffar[2]; R. Jagdheesh[1]; A. K. Nath[3]; [1]Regional Engineering College, Dept. of Physics, Tiruchirappalli, Tamil Nadu 620 015 India; [2]Jamal Mohamed College, Dept. of Physics, Tirichirappalli, Tamil Nadu 620 020 India; [3]Center for Advanced Technology, Industl. CO2 Laser Sect., Indore 452 013 India

The hardness and wear resistance of various engineering materials have been reported to be greatly enhanced by laser surface treatment with different metal-ceramic powder mixtures (Ti-SiC, Ni-WC, Ni-TiC and Ni-Cr3C2/(Ni-Cr). The observed increase in the surface hardness was in the range of 600-1500 HV. Al-12Si is widely used in automobile industry for manufacturing engine pistons. However, its hardness and wear resistance are found to be insufficient for many engineering applications. The fundamental cause of poor wear characteristics of Al-12Si is its low hardness (100 HV). In the present investigation, attempts are made to form metal-ceramic layers over Al-12Si with various compositions of Ni-SiC using high power CW CO2 laser to improve its hardness and wear resistance. Different laser powers and scan speeds were used. The sample laser surface treated with 25Ni-75SiC (% wt) composition exhibited formation of metal-ceramic layers. The layers exhibited appreciable increase in the microhardness

(500-1200 HV). Microstructure of laser melted zone exhibited dendrite structures. Different phases formed in the laser treated region were identified by XRD.

9:20 AM

Growth of TiN/AlN Superlattice by Pulsed Laser Deposition: *Haiyan Wang*[1]; Ashutosh Tiwari[1]; Abhishek Gupta[1]; Xinghang Zhang[1]; Jagdish Narayan[1]; [1]North Carolina State University, Matls. Sci. & Eng., 2141 Burlington Lab., CB 7916, Raleigh, NC 27695-7916 USA

TiN-AlN binary-components have attracted a lot of interests in coatings of high speed cutting tools, due to their higher oxidation resistance, higher hardness, lower internal stresses and better adhesion. Especially, nanometer-scale multilayer structures of AlN/TiN show superior structural and mechanical properties due to their tremendous interface area and become one of the promising candidates for superhard coatings. Here we present a novel method to grow highly aligned TiN/AlN superlattice by pulsed laser deposition. In this method TiN and AlN targets are arranged in a special configuration that they can be ablated in sequence, giving alternate layer by layer growth of TiN(1nm)/AlN(4nm). X-ray diffraction and transmission electron microscopy (TEM) analysis showed the structure to be cubic for both TiN and AlN in the nanoscale multilayers. Microstructure and uniformity for the superlattice structure were studied by TEM and Scanning transmission electron microscopy with Z-contrast (STEM). Nanoindentation results indicated a higher hardness for this new structure than pure TiN and AlN and four point probe electrical resistivity measurements showed overall insulating behavior.

9:40 AM

Deposition of Yttria Stabilized Zirconia with Ion-Plating Apparatus: *Guo Chun Xu*[1]; Kazuhisa Fujita[1]; Taiji Torigoe[2]; Yutaka Hibino[1]; [1]Ion Engineering Research Institute Corporation, The 2nd Rsrch. Dept., Tsuda-Yamate 2-8-1, Hirakata, Osaka 573-0128 Japan; [2]Mitsubishi Heavy Industries, Ltd., Matls. & Strength Lab., 2-1-1 Shinhama, Arai-Cho, Takasago, Hyogo 676-8686 Japan

Currently air plasma spraying (APS) and electron-beam physical vapor deposition (EB-PVD) are two prominent processes to fabricate the thermal barrier coating (typically yttria stabilized zirconia (YSZ)) on turbine engine blades. The APS process results in a porous coating with lower thermal conductivity, and the EB-PVD process provides a columnar microstructure with superior tolerance against thermal expansion mismatch. Because the formation of the columnar microstructure requires the substrate temperature over 1000°, a low temperature process is proposed using an ion-plating apparatus equipped with ionization and bias power supplies for the deposition of YSZ. In this presentation we discuss the structural influences of process parameters including the bias voltage, the ionization power as well as annealing after the deposition. The microstructure of the YSZ formed are characterized with Raman spectrometer, X-ray diffraction detector, and scanning electronic microscopy.

10:00 AM

Development of Grain Size Functionally Gradient Materials-Application to Alumina: H. Keshavan[1]; S. Bal[2]; *K. Morsi*[1]; [1]University of Missouri, Mechl. & Aeros. Eng., E3411 Eng. Bldg. E., Columbia, MO 65211 USA; [2]University of Missouri, Dept. of Orthopedic Surgery, Columbia, MO 65211 USA

Nanoceramics have emerged as materials of high technological importance owing to their enhanced properties compared to conventional ceramics. A major draw back for the application of such materials is the higher cost of the precursor nano-powders used compared to conventional ceramic powders. Most mechanical and wear properties of materials are however dictated by the material condition at the surface rather than the bulk. The work presented covers sequential slip casting and optimized hot pressing experiments to produce a continuously decreasing grain size from the bulk to the surface. In other words functionally gradient materials with respect to grain size. The effect of processing parameters on the product microstructure are discussed. The applications for such a technology would be wide, ranging from cutting tools to orthopedic implants.

10:20 AM Break

10:40 AM Invited

Friction Stir Surface Composite Fabrication: *R. S. Mishra*[1]; Z. Y. Ma[1]; [1]University of Missouri, Dept. of Metallurgl. Eng., 218 McNutt Hall, Rolla, MO 65409 USA

Friction stir processing is a new solid state technique for microstructural modification in metallic materials. This has been used to develop a new method for surface composite fabrication. Examples of various SiC reinforced aluminum matrix surface composite will be shown. This relatively fast solid state process avoids any phase stability issues. The transition interface from unreinforced to reinforced region shows excellent bonding. The effects of processing parameters on microstructure and hardness will be presented.

11:10 AM

The Influence of Pulsing and Helium Additions on Particle Energy Distributions on the Structure and Properties of Reactively Sputtered Titanium Oxide and Titanium Nitride Thin Films: Chris Muratore[1]; J. A. Rees[2]; Brajendra Mishra[1]; *John J. Moore*[1]; [1]Colorado School of Mines, Adv. Coatings & Surface Eng. Lab., 1500 Illinois St., Golden, CO 80401 USA; [2]Hiden Analytical, Ltd., 420 Europa Blvd., Warrington, WA5 5UN UK

A plasma probe consisting of an electrostatic ion energy analyzer in series with a quadrupole mass spectrometer was used to study the effect of pulsing the power supply when using inductively coupled plasma (ICP) enhanced reactive deposition of titanium nitride and titanium oxide thin films. The deposition processes for both materials employed an unbalanced magnetron to generate the metal flux, and utilized the ICP as a remote plasma source to excite the reactive gas before it entered the processing chamber. The relative abundances of various oxygen and nitrogen species arriving at the substrate were manipulated by adding small amounts (less than 20% total reactive gas flow) of helium to the reactive gas before excitation by the ICP source. Additionally, higher energy molecular and atomic ions of nitrogen were observed in the presence of helium gas. The variation of plasma chemistry and/or energy distributions associated with helium additions resulted in a 44% increase in hardness and a 22% increase in the smoothness of titanium nitride films. It was also possible to increase the transmission of titanium oxide films in the range of visible light wavelengths by adding helium. Pulsing the plasma source also resulted in denser films, increased hardness and decreased resisistivity of the films. Such modifications of material properties are explained in terms of measured plasma properties.

11:30 AM

Direct Laser Deposition of In Situ Boride Reinforced Titanium Alloy Composites: *Rajarshi Banerjee*[1]; Peter C. Collins[1]; Sean Connors[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Due to their wide applicability, there is considerable interest in the development of metal-matrix composites consisting of hard precipitates, such as transition-metal borides, dispersed in a metallic/alloy matrix. One such system that has generated considerable interest in recent years is the titanium boride in titanium alloy matrix system. Despite the development of a variety of different processing routes for these composites, including some in situ ones, there are relatively few technologies capable of processing a fully dense, near-net shape component with a relatively fine dispersion of boride precipitates. A recent advancement in the field of near-net shape manufacturing techniques is laser engineered net-shaping (LENSô) which falls in the class of direct laser deposition processes using powder feedstock. Using a feedstock consisting of a blend of Ti (or Ti-6Al-4V) and elemental boron powders, TiB reinforced Ti-alloy composites have been deposited via the LENSô process. These as-deposited composites exhibit a refined homogeneous distribution of TiB precipitates within the alloy matrix, a consequence of the rapid solidification rates inherent to the LENSô process. The TiB precipitates are thermodynamically stable and exhibit minimal coarsening even after prolonged exposures to high temperatures. The ability to deposit these composites in situ at precise locations using the LENSô process affords the possibility of using this technology for surface engineering and repairing expensive components of intricate geometry, such as turbine blades used in aircraft jet engines.

The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research: Amorphous Materials, Radiation Effects, Composites, Quasicrystals

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

Wednesday AM Room: 9
March 5, 2003 Location: San Diego Convention Center

Session Chairs: M. Yamamoto, Osaka University, Matls. Sci. & Eng. Dept., Osaka 565 Japan; Wayne King, Lawrence Livermore National Laboratory, San Ramon, CA 94583 USA

8:30 AM Invited
Microstructure Evolution During Irradiation: *Michio Kiritani*[1]; [1]Hiroshima Institute of Technology, Sch. of Eng., Miyake 2-1-1, Saeki-ku, Hiroshima 731-5193 Japan

Microstructural evolution in metals and alloys during energetic particle irradiation is reviewed with emphasis on the underlying defect reaction processes. The microstructures produced by electon irradiation with a high-voltage electron microscope, fusion-neutron irradiation with a D-T fusion neutron source, fission-neutron irradiation with reactors, and ion irradiation with accelerators are compared and contrasted. By electron irradiation, analyses of defect microstructure developments extend to include the measurement of point defect properties, and the detection of a variety of point defect processes such as radiation-induced diffusion, stochastic fluctuation of point defect reactions, and one dimensional easy motion of small interstitial clusters. By neutron irradiation, analysis of defects produced by cascades and subcascades leads to the recoil energy spectrum analysis. A comparison is made of the damage produced by fission- and fusion-neutron irradiation, to find differences depending on the defect processes involved.

9:00 AM
Nucleation of Vacancy Loops in Quenched Pure Aluminum: *Kuan Yeul Victor Chen*[1]; [1]Formerly of Northwestern University, Matls. Rsrch. Ctr. & Dept. of Matls. Sci. & Eng., Evanston, IL, Presently at 2715 Cazadero Dr., Carlsbad, CA 92009 USA

The nucleation of vacancy loops has been studied in nominally 99.999% pure aluminum using transmission electron microscopy and electrical resistivity measurements. The clustering of supersaturated vacancies after quenching was examined with a two-step aging treatment. The temperatures characterizing the nucleation of vacancy loops in aluminum are analyzed. For aluminum quenched from 610°C into liquid nitrogen and preaged at temperature -60°C or lower, the rate controlling process for the nucleation of vacancy loops is the migration of single vacancies at the beginning of preaging. Toward the end of preaging, the nucleation rate is dominated by the migration of divacancies. At temperature higher than -40°C, dissociation of subcritical clusters is noticeable. The nucleation rate is found to vary with the concentration of quenched-in vacancies and the aging temperature. This investigation suggests that the migration energy, E_m^2, of a divacancy is $E_m^2 = 0.47 \pm 0.02$ eV.

9:20 AM
In-Situ Observation of Brownian Motion and Dynamical Response to Irradiation of Helium Bubbles in Al and Cu: *Kotaro Ono*[1]; Kazuto Arakawa[1]; Robert C. Birtcher[2]; [1]Shimane University, Dept. of Matls. Sci., 1060 Nishi-Kawatsu, Matsue, Shimane Prefecture 690-8504 Japan; [2]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA

The dynamical response to irradiation of helium bubbles in pure Al has been studied in-situ using high-energy self -ions irradiation in an electron microscope. Helium bubbles were introduced in pure Al by pre-irradiation of He+ ions. TEM specimens were then warmed up stepwise to higher temperatures. The dynamical behaviors of the bubbles during beam on and beam off periods were continuously monitored by TEM and recorded with a video recording system. At enough high temperature, Brownian type motion of helium bubbles was quantitatively demonstrated during the beam off periods. While, most of helium bubbles were retarded and a few bubbles were accelerated during the beam on period. At low temperatures when bubbles are thermally immobile, irradiation of high energy self-ions caused intermittent bubble motion. These results are interpreted in terms of competitive point defects absorption to bubbles and easy migration of bubbles along interstitial type dislocation loops during the irradiation with high energy self-ions.

9:40 AM Invited
Is Segregation-Induced Grain Boundary Embrittlement a Kinetically-Constrained Melting Process?: *Paul R. Okamoto*[1]; Jonas K. Heuer[2]; Nghi Q. Lam[1]; [1]Argonne National Laboratory, Matls. Sci. Div., 9700 S. Cass Ave., Argonne, IL 60540 USA; [2]Bettis Atomic Power Laboratory, Matls. Tech., PO Box 79, W. Miffin, PA 15122 USA

The kinetics of intergranular embrittlement of dilute polycrystalline Ni-S alloys and that of S-implantation-induced amorphization of single crystal Ni were investigated. Plots of % intergranular fracture versus S-grain boundary concentration and that of the amorphous volume fraction versus S-implant concentration were found to be virtually identical, implying that (1) the implanted S-atoms and those segregating to grain boundary sites are subject to the same kinetic-constraint (i.e. drastically reduced S-diffusivity), and (2) that their distributions obey the same Poisson statistics. Hence, polymorphous melting may be the physical origin of both amorphization and segregation-induced embrittlement, an interpretation which is supported by the fact that the critical S-concentration for 50% intergranular fracture and 50% amorphization are equal to the maximum nonequilibrium solubility limit of 17 at. % S defined by the polymorphous melting curve on the Ni-S phase diagram. The extension of the polymorphous melting concept to two co-segregating impurities provides a simple explanation for the well-known synergistic effects of hydrogen and sulfur co-segregation on embrittlement.

10:10 AM
Friction-Induced Solid-State Amorphization from Non-Equilibrium Solid Solution Phase in Fe-Cr-B-Ni-Mo Spray Coatings: *C. G. Park*[1]; H. W. Jin[1]; M. C. Kim[2]; [1]Pohang University of Science & Technology, Ctr. for Adv. Aeros. Matls., Pohang 790-784 Korea; [2]Research Institute of Industrial Science & Technology, Pohang 790-600 Korea

Experimental evidence of friction-induced amorphization is presented from cross-sectional TEM investigations. Rapid quenching of Fe-Cr-B-Ni-Mo alloys resulted in the non-equilibrium microstructure composed of micro-crystalline $(Cr,Fe)_2B$ borides and nanocrystalline α-(Fe,Cr) solid solution phase supersaturated with B and Si. Cross-sectional TEM observations revealed that the crystalline-to-amorphous phase transition occurred in the outer surface layer of the α-(Fe,Cr) phase. The spray coatings exhibited significantly enhanced wear resistance with a significantly low friction coefficient (~0.04) with a presence of thin (~150 nm) amorphous surface layer. Theoretical calculations based on thermodynamic instability of crystalline phase suggest that the super-saturation of the α-(Fe,Cr) solid solution phase and the introduction of lattice defects due to frictional work are the major factors to trigger the amorphization reaction.

10:30 AM
A High-Resolution TEM Study of Deformation-Induced Shear-Band Formation and Crystallization in Amorphous $Al_{90}Fe_5Gd_5$: Wenhui Jiang[1]; *Michael Atzmon*[1]; [1]University of Michigan, Cooley Bldg./N. Campus, NERS, Ann Arbor, MI 48109-2104 USA

In several amorphous, Al-based, alloys, Al-rich nanocrystallites have been reported to precipitate at shear bands formed by plastic deformation. The mechanism of this room-temperature process is still a subject of debate. While large, local, temperature spikes are expected at shear bands, the cooling rates are very high, suggesting that the crystallization cannot be explained by a temperature rise. In an effort toward explaining mechanically assisted nanocrystalliztion in these alloys, we have investigated the nanoscale structure using images constructed from the small-angle signal in reciprocal space, excluding thickness effects. We observe nanoscale voids to form at shear bands in the tensile, but not in the compressive region. On the other hand, Al nanocrystallites at shear bands are observed only in the compressive region. These results support a crystallization mechanism based on defect production in the solid state.

10:50 AM
Sea Urchin Mineralized Tissue: *Stuart R. Stock*[1]; [1]Northwestern University, IBNAM, Tarry 16-717, 303 E. Chicago Ave., Chicago, IL 60611-3008 USA

Sea urchin ossicles are structural analogs of mammalian bones and a model biomineral system. Relatively new x-ray methods complement electron microscopy and allow study of mineralized tissue with voxels (volume elements) approaching 1 μm3 in millimeter-sized samples. This talk focuses on a multi-mode x-ray investigation of mineralized tissue using microCT, both synchrotron and laboratory sources, phase contrast radiography and transmission microbeam diffraction mapping. Sea urchins employ as wide a range of composite reinforcement strategies as are seen in engineering composites, and, studied as materials, teeth (and other ossicles) from different echinoid families illustrate combinations of reinforcement parameters and toughening mechanisms providing good functionality. This probe of the design space available to sea urchins offers important guidance for engineering of structural tissue. The results on sea urchin teeth and pyramids (jaws) also illustrate what might be accomplished by such an integrated approach in bone.

11:10 AM
Microstructures in an Al-Cu-Fe Quasi-Crystal After Deformation at Room Temperature: *S. Miyazaki*[1]; C. Hirose[2]; S. Kumai[1]; A. Sato[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Eng., 4259 Nagasuta, Midori-ku, Yokohama 226-8502 Japan; [2]LSI Logic Japan Semiconductor Corporation, 10 Kitahara, Tsukuba-shi, Ibaraki 300-0032 Japan

Plastic deformation was given to a specimen containing Al-Cu-Fe quasi-crystals. The specimen thickness as well as strain rate were altered in order to seek a deformable condition at room temperature. It is found that the quasi-crystals embedded in Al do not deform in the usual compression test of a bulk specimen but in a thin foil specimen of the order of the quasi-crystal size they deform plastically under the high stress acting on them directly. A large number of line contrasts, presumably planar defects across a foil specimen, were introduced by a compressive stress applied on the foil. A high density of dislocations was introduced simultaneously by the compression test. Burgers vectors of the glide dislocations were determined by visibility criterion. The dislocation type was different from those reported in the literature after high temperature deformation.

11:30 AM
Interactive Effect of Temperature, Stress, Moisture, and Physical Aging on Creep and Creep Rupture of a Polymer Composite Matrix: *Raghavan Jayaraman*[1]; [1]University of Manitoba, Mech. & Indl. Eng., 348D, Eng. Bldg., 15, Gillson St., Winnipeg, MB R3T 5V6 Canada

Individual and interactive effect of temperature, stress, moisture, and physical aging on creep and creep rupture of Hexcel F263 epoxy were experimentally evaluated and modeled. Individually, increase in temperature, stress, and moisture accelerated creep and creep rupture of the epoxy and increase in physical aging retarded them. Under combined loading conditions, there was no interaction among temperature, stress, and moisture; however, there was significant interaction among physical aging, moisture, and stress. A creep model based on superposition principle and a creep rupture model based on a critical fracture energy criterion have been used to model the experimental data. Fractographs are presented and discussed, in support of the critical fracture energy criterion used in this study.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Iron and Steel Making Fundamentals I

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday AM — Room: Leucadia
March 5, 2003 — Location: San Diego Marriott Hotel

Session Chairs: V. Sahajwalla, The University of New South Wales, Sch. of Matls. Sci. & Eng., Sydney, NSW Australia; Takashi Nakamura, Tohoku University, Dept. of Metall. Grad. Sch. of Eng., Sendai Japan

8:30 AM Invited
A Basic Study on the Effective Utilization of Chromium-Containing Steel Slag: *Etsuro Shibata*[1]; Takashi Nakamura[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 1,1 Katahira, 2-Chome, Aobaku, Sendai 980-8577 Japan

The solubility of chromium oxide and valance of chromium in slag are important to consider how to utilize effectively the chromium-containing slag. Those experimental data of various types of slag cited in literatures were arranged using optical basicity, and the effects of oxygen potential and slag composition were investigated. The saturated solubility of chromium oxide and the ratio of Cr2+ to Cr3+ tend to decrease with increase in optical basicity of slag at the oxygen potential below 10(-5) atm. On the other hand, at the oxygen potential of 0.21 atm, the saturated solubility and the ratio of Cr6+ to Cr3+ tend to increase with increase in optical basicity. For a practical approach to reduce chromium in stainless steel slag and simultaneously recover chromium as a ferroalloy, the direct smelting reduction of chromium oxide in molten slag using some reductant was also investigated using a small furnace.

8:55 AM Invited
Kinetics of Carbon Injection into Metallurgical Slag: F. Ji[1]; M. Barati[1]; *K. S. Coley*[1]; G. A. Irons[1]; [1]McMaster University, McMaster Steel Rsrch. Ctr., Hamilton, Ontario L8S 4L6 Canada

Carbon injection into metallurgical slags is important in EAF steelmaking for slag foaming and in nonferrous slag cleaning. The current publication presents kinetic data for coal injection into electric arc furnace slags to draw some general conclusions about the interaction between injected carbon and metallurgical slags. Coal particles were injected into EAF slags and the kinetics were followed by gas and slag analysis. The results were analyzed, in terms of fundamental rate equations for each possible reaction step, as a function of temperature and slag composition.

9:20 AM
Semi-Stochastic Optimization of Chemical Composition of High-Temperature Austenitic Steels for Desired Mechanical Properties: *George S. Dulikravich*[1]; Igor N. Egorov[2]; Vinod K. Sikka[3]; Govindarjan Muralidharan[3]; [1]University of Texas at Arlington, Mech. & Aeros. Eng. Dept., Multidisciplinary Analy., Inverse Design & Optimization (MAIDO) Prog., UTA Box 19018, Arlington, TX 76019 USA; [2]IOSO Technology Center, Milashenkova 10-201, Moscow 127322 Russia; [3]Oak Ridge National Laboratory, Matls. Procg. Grp., PO Box 2008, MS6083, Metals & Ceram. Div., Oak Ridge, TN 37831-6083 USA

The methodology for steel optimization subject to several simultaneous objectives consists in organization an iterative optimized experiment. The result of these studies is the Pareto-optimal set of steel compositions that simultaneously optimizes the chosen objectives. The multi-objective optimization algorithm is based upon the use of a response surface that is created from the available experimental data with the help of radial-basis functions and artificial neural networks.

During the conduct of research the information is being stored concerning the properties of steel in the vicinity of the Pareto set. This allows us to improve the accuracy of the results. As the independent design variables we considered the percentage of following components: C, Mn, Si, Ni, Cr, N. Ranges of their variation were set in accordance with lower and upper bounds of the available set of experimental data. As the main optimization objective we considered the strength of the H-type steel after 100 hours under the temperature of 1800F. Other objectives have been chosen based on the necessity to reduce the cost of the steel. In this work, the additional three objectives were to simultaneously minimize the percentages of Mn, Ni, Cr. Thus, the multi-objective optimization problem had 6 independent design variables and 4 objectives. We defined the desirable number of Pareto optimal solutions as 10 points. Every iteration of this methodology results in a formulation of a set of steel compositions, which are Pareto optimal and need experimental evaluations to obtain the true values of the objectives.

9:45 AM
ìIn-Situî Observation of Iron Carburization During Smelting Reduction: *Ko-ichiro Ohno*[1]; Tetsuya Nagasaka[2]; Mitsutaka Hino[2]; [1]Tohoku University, Dept. of Metall., Aza-Aoba 02, Aramaki, Aoba-ku, Sendai 980-8579 Japan; [2]Tohoku University, Dept. of Metall., Grad. Sch. of Eng., Aoba-yama 02, Sendai 980-8579 Japan

It was recently recognized that the reduction, melting of iron and slag separation in the composite of granular iron ore and coal are completed in a very short period of about 10 minutes when the composite is heated rapidly up to approximately 1673K. Utilization of these phenomena is attempted for new iron-making process. The mechanism of iron ore reduction and iron-slag separation during rapid heating has not yet been realized, and the fundamental research has just been started. Carburization of the reduced iron is regarded as an important factor in this process. In the present work, the composite was prepared from four kinds of coal or graphite and electrolytic iron powder. The temperature at which liquid were formed and iron was carburized was monitored at various heating speeds. Direct observation of smelting reduction behavior was made by a laser microscope combined with the infrared image heating furnace to clarify the effect of molten ash on iron carburization. The meltdown temperature of composite fell as melting temperature of coal ash fell. From the observed result, it was presumed that liquid formation in composite triggered carburization of iron. Carburization mechanism during smelting reduction was considered as follows. When slag containing iron oxide melted down and contacted with carbon, iron oxide in slag was reduced and metallic iron particle was formed and carburization of the iron particle occurred simultaneously at slag-carbon interface. Carburized iron particle was carried from slag-carbon to slag-iron interfaces due to slag convection flow caused by the difference of interface tension between carbon-slag and iron-slag interfaces. Repeating the above-mentioned process continuously carburized the reduced iron.

10:10 AM Break

10:20 AM Invited
Fundamental Investigation of Basic Mechanisms of Carbon Dissolution in Molten Iron: *V. Sahajwalla*[1]; R. Khanna[1]; [1]The University of New South Wales, Sch. of Matls. Sci. & Eng., Sydney, NSW 2052 Australia

Based on experimental and computer simulation results from our group, we report a systematic, atomic level analysis of carbon dissolution from carbonaceous materials in molten iron. Experimental results were obtained using carbon dissolution studies, wetting, XRD and FESEM investigations, and Monte Carlo simulations on Graphite/Fe-C-S system. Three basic mechanisms, namely carbon atom dissociation rate, interfacial phenomena and mass transfer in the melt were found to be responsible for the overall dissolution process. While mass transfer in the melt is governed by the concentration gradients, impurities such as sulphur and temperature, sulphur and oxide impurities present in ash play a significant role in the interfacial phenomena. Carbon atom dissociation kinetics depends strongly on the degree of long-range order of carbonaceous materials and is an important rate controlling mechanism in the overall dissolution kinetics of less ordered forms of carbon. A systematic comparison of dissolution rates for a var iety of coals, cokes, chars, natural and synthetic graphite has been carried out in an attempt to identify rate-controlling mechanisms. The structure of the carbonaceous material appears to be an important underlying factor in carbon dissolution.

10:45 AM Invited
Dissolution of Alumina Particles in CaO-Based Fluxes: *W. D. Cho*[1]; Peter Fan[1]; [1]University of Utah, Dept. of Metallurgl. Eng., Salt Lake City, UT 84112 USA

The slag chemistry in steel processing plays an important role in terms of the removal of impurities including alumina particles. In the present study, the dissolution of solid alumina particles in CaO-based fluxes at the temperatures between 1450 and 1550°C has been investigated to determine dissolution kinetics and mechanism. Alumina particles were added directly in the slags and the size of alumina particles was measured as a function of time using optical and scanning electron microscopes. The dissolution rate has also been obtained as a function of SiO2, CaF2, MgO and MnO contents in the slags. The diffusion boundary layer between alumina and bulk slag phase has been observed and analyzed using SEM and EDX. In addition, some experiments have been performed for the dissolution of alumina in BaO-based fluxes.

11:10 AM
The Effect of MgO and Al2O3 Additions on the Liquidus for the CaO-SiO2-FeOx Systems at Various Partial Pressure of Oxygen: Hisao Kimura[1]; *Fumitaka Tsukihashi*[2]; [1]The University of Tokyo, Grad. Sch. of Eng., 7-3-1 Hongo, Bunkyo, Tokyo 113-8656 Japan; [2]The University of Tokyo, Grad. Sch. of Frontier Scis., 7-3-1 Hongo, Bunkyo, Tokyo 113-0033 Japan

Phase diagrams for the CaO-SiO2-FeOx-MgO, Al2O3 systems are necessary for the analysis of copper smelting reaction and sintering process of iron ore. The effect of MgO and Al2O3 addition on the liquidus of phase diagram for the CaO-SiO2-FeOx systems at low oxygen partial pressure was observed. The liquid phase area was changed by the addition of MgO and Al2O3 with changing oxygen partial pressure from 10^{-8} to 10^{-3} atm at 1573K. The effect of Fe2+/Fe3+ ratio on the melting mechanism is discussed.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Elemental Losses and Distributions

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday AM	Room: Solana
March 5, 2003	Location: San Diego Marriott Hotel

Session Chairs: Yoichi Takeda, Iwate University, Fac. of Eng., Morioka 020-8551 Japan; Kimio Itagaki, Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Sendai, Miyagi 980-8577 Japan

8:30 AM Keynote
Distribution of Minor Elements in Sulfide Smelting: *Kimio Itagaki*[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi 980-8577 Japan

Various kinds of molten sulfide systems of Cu2S-FeS, Ni3S2-FeS, Ni3S2-Cu2S-FeS, ZnS-Cu2S, PbS-Cu2S, Ni-S and Pb-S were equilibrated with molten FeOx-SiO2, FeOx-CaO and Al2O3-CaO based slags in a magnesia crucible under controlled partial pressures of SO2, O2 and S2 to determine the distribution ratios of some valuable(Ag, Au, Co, Ni etc.) and detrimental(Pb, As, Sb, Bi etc.) minor elements in smelting various sulfide concentrates. The experimental results were analyzed on the basis of thermodynamics and the effects of partial pressures of oxygen and sulfur as well as slag basicity were discussed in this study.

9:00 AM
Thermodynamic Evaluation of Arsenic and Antimony on the Bessemer Matte and Calcium Ferrite Slag: *Jonkion M. Font*[1]; Mitsuhisa Hino[2]; Kimio Itagaki[2]; [1]The University of Alabama, Dept. of Metallurgl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487-0202 USA; [2]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Sendai, Miyagi 980-8577 Japan

Due to the known disadvantages of the use of iron silicate slag in the nickel converting stage, i.e., high metal losses, high magnetite

content, and low fixation of impurities in the slag, the development of the new proposed iferrous calcium silicateî slag for smelting and converting processes of nickel is very promising. Hence, considering the successful experimental results of using CaO base slag for the nickel smelting process, a study based on a detailed comparison for the phase equilibrium, and the minor elements distribution between the Ni_3S_2-FeS matte with either FeO_X-CaO or FeO_X-SiO_2 base slag was investigated. The main results are summarized as: no difference in the nickel losses between both slags was found at high nickel content in the matte, and the fractional distribution analysis for arsenic and antimony pointed out their preferential fixation in the calcium ferrite slag rather than in the iron silicate slag. The technical feasibility of using calcium ferrite slag in a converting process of the Bessemer matte will have a prominent future for the nickel industry when the energy and pollution issues are taken into account.

9:25 AM

Thermodynamics of PbO, ZnO and CuO0.5 in CaO-SiO2-Al2O3 Melts: *Takaaki Ishikawa*[2]; Kenji Matshuzaki[3]; Takayuki Tsukada[1]; Kimihisa Ito[1]; [1]Waseda University, Matls. Sci. & Eng., 3-4-1, Okubo, Shinjuku-ku, Tokyo 169-8555 Japan; [2]Chiba Prefectural Machinery & Metallurgy Research Institute, Chiba 263-0016 Japan; [3]Mitsui Mining & Smelting, Rsrch. Ctr., Saitama 362-0021 Japan

The distribution equilibria of lead, zinc and copper between CaO-SiO2-Al2O3 melts and liquid copper were measured at 1623K under a controlled H2-CO2 atmosphere. The distribution ratios were plotted against the oxygen partial pressure, and reasonable oxide forms dissolved in the melts were estimated from the slopes of the plots. The activity coefficients of lead oxide (PbO), zinc oxide (ZnO) and cuprous oxide (CuO0.5) increased with increasing slag basicity, defined by XCaO/XSiO2. The temperature dependence of the activity coefficients was also measured.

9:50 AM

Distribution of Minor Elements Between Ni-S Melt and Al_2O_3-CaO-MgO Slag at 1873 K: *Hector Mario Henao*[1]; Mitsuhisa Hino[1]; Kimio Itagaki[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls. (IMRAM), 1,1 Katahira, 2-Chome, Sendai, Miyagi 980-8577 Japan

To provide thermodynamic data for converting the nickel matte to liquid nickel, distribution ratios of some minor elements (Au, Ag, Cu, Co, Fe, P) between the Ni-S alloy and the Al_2O_3-CaO-MgO slag in a magnesia crucible were determined at 1873 K under controlled PSO_2 of 10.1 kPa and PO_2 in a range between 8.0 and 0.1 Pa by using CO-CO_2-SO_2 gas mixtures. It was found that the distribution ratios, $L_X^{S/Ni}$, defined by (mass%X in slag)/{mass%X in alloy}, for P, Fe are larger than unity, while less that unity for Au, Ag, Cu, Co.

10:15 AM Break

10:25 AM Keynote

Thermodynamic Evaluation of Copper Loss in Slag Equilibrated with Matte: *Yoichi Takeda*[1]; [1]Iwate University, Fac. of Eng., Ueda 4-3-5, Morioka 020-8551 Japan

Main focus is the determination of copper solubility in SiO2-CaO-FeOx slag equilibrated with matte under specified SO2 atmosphere. A classical thermodynamic model postulating CuO0.5 and CuS0.5 molecules applies to calculate copper solubility in slag. A large number of experiments provide proper parameters: activity coefficients of copper oxide and sulfide, oxygen and sulfur potentials and activity of copper in the slag-matte system. Copper loss , that is function of copper solubility and slag volume, in the silica saturated SiO2-FeOx binary slag is minimally equilibrated with less than 66% copper matte while copper loss in the 50% FeOx-SiO2-CaO is minimally equilibrated with over 66% copper matte. This slag composition has potential for continuous copper converting, direct copper production or high-grade matte production.

10:55 AM Invited

Copper Losses in Copper Smelting Slags: *Ivan Imris*[1]; [1]Technical University of Kosice, Dept. of Power Eng., Fac. of Mechl. Eng., Letna 9, Kosice 041 87 Slovak Republic

The common problem of new and conventional technology of copper production is the copper losses in the slags, which may be divided into mechanical and physico-chemical losses. In the first group can be included particles of mechanically entrained or floated unsedimented matte particles which coexist with slag. On the other hand, the physico-chemical losses are caused by solubility of copper in the slag in the form of sulphide and oxide. The copper losses in the slags from different processes has been predicted by calculation from thermodynamic data and compared with those determined by microscopic examination and quantitative electron microprobe analysis. Depending on the forms of copper losses in the slags the reduction of copper losses in the slag or the slag cleaning process could be suggested.

11:20 AM

Distribution Behavior of Arsenic, Antimony and Bismuth: *Supachai Surapunt*[1]; Nozomu Hasegawa[2]; [1]Thammasat University, Fac. of Eng., 99 Phaholyothin Rd., Klong-Luang, Pathumthani 12121 Thailand; [2]Naoshima Smelter and Refinery, Mitsubishi Metal Corp., Naoshima, Kagawa Japan

The distribution behavior of VA minor elements (As, Sb and Bi) in the smelting stage of the Mitsubishi process were evaluated thermodynamically based on thermodynamic data and operating data by using the method of calculation proposed by Itagaki and Yazawa. The fractional distributions between the gas, slag and matte phases considered as the degree of vapor saturation of 0.8 are as follows: 53.8, 27.2 and 19% for As, 23.6, 56.6 and 19.8% for Sb, and 91.3, 1.1 and 7.6% for Bi, respectively. Arsenic is mostly distributed to the gas and slag phases. It is effectively eliminated by volatilizing and slagging. Antimony is mainly in the slag phase which is suitably removed by slagging. Bismuth is easily vaporized to gas phase. The degree of vapor saturation has a large effect on the distribution behaviors of the three elements between the three phases. The amount of a minor element in the charge has an effect on the distribution of As but no effect on the distributions of Sb and Bi. Increasing matte grade and temperature results in the change of distributions for As and Sb, significantly. The change in the distribution ratio between slag and matte phases also considerably affects the distributions of As and Sb. The distribution behavior of Bi is not significantly changed by the change in matte grade, temperature and distribution ratio.

11:45 AM

The Influence of Reverb Slag Composition on Copper Losses: *Natasa Mitevska*[1]; *Zivan D. Zivkovic*[1]; [1]Copper Institute, Zeleni bulevar 35, Bor 19210 Yugoslavia

The statistical analysis of the slag composition influence on copper losses to discard slag in the reverberatory furnaces No. 1 and No. 2 in the RTB BOR, Copper Smelter and Refinery (Yugoslavia) is presented in this paper. The model for the slag basicity calculation is determined, and connected to the copper distribution coefficients between the matte and slag phase. The influence of all components on the slag structure is also illustrated.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Materials Processing Technologies: General I

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday AM	Room: Point Loma
March 5, 2003	Location: San Diego Marriott Hotel

Session Chairs: Toshiyuki Kozuka, Kumamoto University, Dept. of Matls. Sys., Kumamoto 860-8555 Japan; Y. B. Hahn, Chonbuk National University, Chonju 561-756 Korea

8:30 AM Invited

Fabrication of Blue Light-Emitting Diodes Using GaN-Based Multiple Quantum Wells Grown by Metal Organic Chemical Vapour Deposition: *Yoon-Bong Hahn*[1]; Rak-Jun Choi[1]; Hyung Jae Lee[1]; [1]Chonbuk National University, Sch. of Chemi. Eng. & Tech., Semiconductor Physics Rsrch. Ctr., 664-14 Duckjin-Dong 1 Ga, Chonju 561-756 Korea

Gan-based blue light emitting diodes (LEDs) were fabricated by utilizing nanoscale triangular- and rectangular-type multiple quantum wells and their characteristics were compared in terms of structural,

electrical and optical properties. Optimization of the LED fabrication process was also investigated by minimizing a plasma-induced damage. The InGaN/GaN multiple quantum wells (MQWs) were grown by a low-pressure metalorganic chemical vapour deposition method. The size of quantum dots formed in the active layer of InGaN was in a range of 2 to 50 nm, and the dots in the triangular QWs showed very uniform spatial distribution compared to those in the rectangular QWs. Both photoluminescence (PL) and electroluminescence (EL) showed higher emssion and smaller full-width at half maximum in QWs and LED structures with triangular QWs than those with rectangular QWs. Especially, EL spectrum as a function of injection current showed that the peak energy is nearly independent of the injection current in the triangular-QW -based LEDs. In the course of LED fabrication a physical degradation of sidewall along with rough surface morphology of n-GaN caused by increased ion scattering induced the deterioration of forward and reverse voltages. It was found that the turn-on voltage is sensitive to the surface roughness of the etched n-GaN and the breakdown voltage is strongly affected by the sidewall contamination. Annealing under nitrogen after the mesa etching improved the electrical properties of the InGaN/GaN MQW LEDs.

9:00 AM
Sintering of Pb{Zr,Ti,(Mg1/3Nb2/3)}O3 Ceramics by Spark Plasma Sintering and their Compositional Distribution: *Kazuyuki Kakegawa*[1]; Satoru Sawahara[2]; Naofumi Uekawa[3]; [1]Chiba University, Grad. Sch. of Sci. & Tech. & Dept. of Matl. Tech., Fac. of Eng. & Ctr. for Frontier Elect. & Photonics, 1-33 Yayoi-cho, Inageku, Chiba-shi, Chiba 263-8522 Japan; [2]Chiba University, Grad. Sch. of Sci. & Tech., Chiba 263-8522 Japan; [3]Chiba University, Dept. of Matl. Tech., Fac. of Eng. & Ctr. for Frontier Elect. & Photonics, Chiba 263-8522 Japan

Both sintering and homogenization of ceramic solid solutions are the result of diffusion during the heating at high temperatures. Thus homogenization generally accompanies with sintering. It is a normal fact that distribution of composition decreases as the sintered density of the sample increases. We found an interesting fact that dense sintered body could be obtained with almost no change in the compositional distribution, when spark plasma sintering (SPS) method was employed. This paper shows such results in Pb{Zr,Ti,(Mg1/3Nb2/3)}O3 solid solution. The change in the compositional distribution of the sintered body by SPS was compared with that by the normal sintering. By normal sintering, the compositional distribution decreased as the sintered density increased as normally expected. On the contrary, the compositional change by SPS was much smaller than that by the normal sintering. This sintering characteristic of SPS enabled a fabrication of sintered material having a desired compositional distribution.

9:25 AM
Mg Alloy Composite Materials with Solid-State Synthesized Mg2Si Dispersions: *Katsuyoshi Kondoh*[1]; Wenbo Du[1]; Ritsuko Tsuzuki[1]; [1]The University of Tokyo, Rsrch. Ctr. for Adv. Sci. & Tech. (RCAST), 4-6-1, Komaba, Meguro-ku, Tokyo 153-8904 Japan

The synthesis processing of Mg_2Si or Mg_2Si/MgO at lower temperature from the elemental magnesium alloy chips and Si or SiO_2 powder mixture was established via the cyclic plastic working based on Powder Metallurgy (P/M) process, which is effective on the mechanical breakage of MgO surface films preventing from the solid-state reaction between Mg and Si powder. The Mg_2Si grain of 200-500nm is extremely fine, compared to that via the conventional casting process, because of not coarsening during the solid-state synthesis of Mg_2Si. By using in-situ forming process of Mg_2Si via the cyclic plastic working, the magnesium composite material with Mg_2Si/MgO dispersions, which particle size was less than 1-3µm, was developed. For example, when employing AZ31 chips as raw materials of the matrix, it has low density of 1.85g/cm3 and extremely superior mechanical properties, such as tensile strength over 350MPa and Youngís modulus of 50GPa, to the conventional Mg-Al-Zn alloys. Concerning to the tribological property under the wet lubricant condition, it shows a low friction coefficient less than 0.04 and no seizure phenomenon with contacting to the S35C steel counter material. In particular, when including the formed MgO particles, the coefficient reduced to 0.02 due to its mild offensive property on the counter part. Its wire and pipe were produced via the mass hot extrusion production in manufacturing plant, and also showed excellent mechanical properties. The optimization of breezing and welding conditions on this developed Mg composite are going in this study.

9:50 AM
Reaction of Sn-Containing Solders with Nickel Based Under Bump Metallizations: *Guojun Qi*[1]; Min He[2]; Zhong Chen[2]; [1]Singapore Institute of Manufacturing Technology, Process Tech. Div., 71 Nanyang Dr. 638075 Singapore; [2]Nanyang Technological University, Sch. of Matls. Eng. 639789 Singapore

This work relates to wafer bumping technology development for flip chip packaging applications in the electronics industry. Nickel is an alternative under bump metallization (UBM) material because of its slower reaction rate with Sn-based solders as compared to Cu-based UBMs. Two types of Ni-based UBMs are widely used: sputtered nickel and electrolessly plated Ni-P alloys (EN). In this study we compared the interfacial morphology of Sn-containing solders (Sn-Ag and eutectic Sn-Pb) with these two types of nickel based under bump metallizations. Both chunky and needle type of intermetallics were observed between the EN UBM and the solders. Their morphology changed with different cooling rates below the melting temperatures of the solders. In the case of the sputtered nickel UBM, there was only a layer of scallop-type intermetallics formed. These differences are discussed in terms of soldering reaction at the interface. Kinetics of the intermetallic growth was also examined on the two UBM systems.

10:15 AM Break

10:35 AM Invited
Effect of Intense Magnetic Field on CdTe Electro-Deposition: *Toshiyuki Kozuka*[1]; Yoshinori Sugita[1]; Masayasu Kawahara[1]; [1]Kumamoto University, Dept. of Matls. Sys., 2-39-1, Kurokami, Kumamoto 860-8555 Japan

CdTe metal composite semi-conducting material has high potential for good performance of energy conversion efficiency, so that there is much room for improvement of energy conversion efficiency according to the processing of making thin CdTe film. In this paper, the imposition of intense magnetic field on CdTe electro-deposition is proposed, which is one of possibility for improving the energy conversion efficiency. In the experiment, cryogen-free superconducting magnet up to 5T was used and CdTe film was deposited electrically in -0.72V vs. SHE. The electrolyte was Cd and Te ammonia alkali solution with controlled temperature in the acrylic cell of 70mm diameter. Intense magnetic field up to 5T can not only make the deposition surface smooth, but also the size of CdTe crystal.

11:05 AM Invited
Ceramic-Metal Composites Obtained by Reactive Pressureless Counterflow Infiltration/Penetration (RPCI) of a Ceramic Substrate with Two Different Metallic Infiltrants: *V. M. Kevorkijan*[1]; [1]Independent Researching, Betnavska cesta 6, Maribor 2000 Slovenia

A new method for faster production of functionally graded metal-ceramic laminates is based on reactive pressureless counterflow infiltration/penetration of a ceramic substrate with two different metallic infiltrants. The porous or dense ceramic preform is pressurelessly infiltrated or penetrated from one side by the first metallic infiltrant to the desired cross section of the preform, producing the top layer made up of the solidified infiltrant and the underlayer consisting of reactive infiltrated preform. The partly infiltrated preform is then completely infiltrated starting from the other side by the second metallic infiltrant, in this way fabricating the internal layer, consisting of the ceramic preform infiltrated with the second infiltrant and the bottom layer formed by the solidified second infiltrant. Systems already investigated include carbide, nitride and boride ceramic performs infiltrated with aluminum and magnesium alloys. The combination of ferrous and non-ferrous infiltrants (aluminum alloys and cast gray iron) was also experimentally studied.

11:35 AM
Preparation of Vanadium-Doped SnO2 Nanocrystallites: *Huaming Yang*[1]; Weiqin Ao[1]; Chenghuan Wang[1]; Guanzhou Qiu[1]; [1]Central South University, Dept. of Minl. Eng., Changsha 410083 China

The vanadium-doped SnO2 nanoparticle was synthesized by the co-precipitation reaction and subsequent calcination from the vanadium(III) chloride and tin(IV) chloride. The crystal size, pore size distribution, micrograph and properties of the nanocrystalline powders were examined by differential thermal analysis (DTA), thermogravimetric analysis (TGA), X-ray diffraction (XRD), desorption isotherm (Barrett-Joyner-Halenda method) and transmission electron microscopy(TEM). Thermal treatment of the precipitate powder at 600°C led to the formation of V-SnO2 nanoparticle of ~10nm in crystal size. Most of the pores in the nanoparticle are about 5~15nm in diameter. Effect of doped vanadium on the crystal size of the nanoparticle was discussed.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Aqueous and Electrochemical Processing IV

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday AM Room: Pacific
March 5, 2003 Location: San Diego Marriott Hotel

Session Chairs: Tsutomu Yamamura, Tohoku University, Sendai, Miyagi Prefecture 980-8579 Japan; Dingfan Qiu, Beijing General Research Institute of Mining and Metallurgy, Beijing 100044 China

8:30 AM Invited
Extraction and Separation of Metals from Sulfide by Slurry Electrolysis Process (SEP): *Qiu Dingfan*[1]; Wang Jikun[1]; [1]Beijing General Research Institute of Mining and Metallurgy, Wenxing St., Xizhirncnwai, Beijing 100044 China

Slurry Electrolysis Process (SEP) is a new hydrometallurgical method and clean technology. Sulfide ores can be treated by SEP without S02 emission, and the sulfur from the sulfides will be recovered in element form which is easy to stockpile and can be transported at low cost. In a special slurry electrolysis cell, it can achieve leaching of elements from the feed and electrowinning of metals from the electrolyte at the same time. Because the anode reactions are utilized for leaching some elements, the consumption of the techological energy is decreased. Author has researched the behavior of different metals in the process and point out that separation of metals in some minerals is possible by SEP. Beijing General Research Institute of Mining and Metallurgy (BGRIMM) has researched SEP to treat bismuth concentrate contain S, Be and F from laboratory to pilot-plant and commercial plants. The commercial plant was set up in 1997; it has been in operation since that time. SEP also has advantage of separation some elements. It is especially suitable for treatment of complex concentrates, which could hardly be accepted by any smelter. This kind of mineral contains Pb; Cu, Au, Ag and S. According to the results of lab experiment and pilot-plant test, a new plant to treat complex gold minerals was set up in 1998 and was in operation in 1999.

9:05 AM
Gold Leaching by Using Ammonium Thiosulfate Solution and Gold Recovery by Solvent Extraction and Cementation: *Toyohisa Fujita*[1]; Liu Kejun[1]; Atsushi Shibayama[1]; Harunobu Arima[2]; Wan-Tai Yen[2]; [1]Akita University, Fac. of Eng. & Resource Sci., 1-1 Tegata Gakuencho, Akita 010-8502 Japan; [2]Queenís University, Dept. of Mining Eng., Kingston, Ontario K7L 3N6 Canada

The effect of variables on the gold extraction with ammonium thiosulfate was investigated on sponge gold and ore samples. The Effects of $CuSO_4$, $(NH_4)_2S_2O_3$, NH_4OH, $(NH_4)_2SO_4$, pH, stirring speed and retention on gold leaching rate and thiosulfate oxidation (consumption) have been studied. Almost 100% of gold was leached from sponge gold and over 96% from ore sample at optimum conditions. Trioctylmethyl ammonium chloride was used to recover gold from thiosulfate pregnant solution. It was found that more than 99% of gold was recovered by the extractant diluted with n-octane at O/A ratio of 1:1 without the re-adjustment of pH or others. Also, the gold cementation was conducted without de-aeration by using zinc, copper and aluminum powders. The result indicated that the gold was effectively recovered from a solution of low ammonia and copper concentrations and higher thiosulfate concentration. The gold recovery process by using ammonium thiosulfate is an environmental friendly method comparing to conventional cyanidation process.

9:30 AM
Corrosion Behaviors of the Pb-Ag-Ca Anodes for Zinc Electrowinning in Sulfuric-Acid Electrolyte: *Yasushi Takasaki*[1]; Hitoshi Watanabe[1]; Kazuo Koike[1]; [1]Akita University, Fac. of Eng. & Resource Sci., 1-1 Gakuen-cho, Tegata, Akita 010-8502 Japan

In the zinc hydrometallurgical extraction, minimizing of the electric power consumption of the electrowinning process is an important issue for energy saving. At the Zinc & Lead 2000 symposium, the authors suggested that using the Pb-0.5%Ag-0.6%Ca alloy decreased the electrolysis energy compared to the conventional Pb-0.9%Ag alloy. In this study, corrosion behaviors of the Pb-Ag-Ca anodes in sulfuric-acid electrolyte were investigated. Referring to the results for the Pb-0.5%Ag-0.6%Ca anode after 20-days of electrolysis, the bath voltage and the quantity of anode slime were decreased respectively as compared with the Pb-1%Ag anode. Subsequently, beta-PbO2 was detected by XRD and its peaks were sharper comparing to respective ones of the Pb-1%Ag anode. Moreover, dense beta-PbO2 layer on the Pb-0.5%Ag-0.6%Ca anode surface was observed. Therefore considering the results, it was suggested that the dense beta-PbO2 layer of the anode surface was a factor that leads to a lower bath voltage and decreasing quantity of anode slime.

9:55 AM
De-Oiling of Industrial Water Effluents Using Column Flotation: *F. J. Tavera*[1]; R. Escudero[1]; [1]Universidad Michoacana de San Nicol·s de Hidalgo, Inst. de Investigaciones Metal˙rgicas, Santiago Tapia 403, Morelia, Michoac·n 58000 MÈxico

Column flotation technology, at first developed for application in mineral dressing, has received special attention in order to process non-mineral dispersions as a result of its low costs, high efficiency, and relatively simple operation. This paper presents the results from the operation of a laboratory flotation column in order to separate vegetable oil and soap from aqueous industrial effluents. The flotation system was operated under continuous countercurrent conditions, without and with additions of a cationic collector. The experiments have shown that it is possible to separate about 80% of the organic phase in a rougher flotation stage; also, the results in this work suggest that there is an optimum bubble size from which either below or above the recovery of the organic decreases.

10:20 AM Break

10:30 AM Invited
Mechanism of the Electrolysis of Rare-Earth Chlorides in Molten Chloride Bath: *Tsutomu Yamamura*[1]; [1]Tohoku University, Grad. Sch. of Eng., 02 Aoba, Aramaki, Aoba-ku, Sendai, Miyagi Prefecture 980-8579 Japan

Recently, there has been a wide interest in the development of a process for producing rare earths and actinides, and their oxides by a pyro-process using molten salts as operational fluids in nuclear fuel reprocessing. The≈\@electrolytic reduction mechanism of rare-earth chlorides such as LaCl3, CeCl3, NdCl3, SmCl3 and DyCl3 in alkali chloride baths has been investigated in NaCl, KCl and eutectic LiCl-KCl by means of electrochemical transient methods. The thermodynamic behaviors of rare-earth containing species have been discussed in the frame of Electrode potential - pO2- diagram. Low current efficiency found in the case of Nd electro-winning has been attributed to the dissolution of Nd into the bath. The analyses of electrochemical measurement required the elucidation of the effects of the factors such as under-potential deposition, metal dissolution, moisture and oxygen.

11:05 AM Invited
The Interfacial Chemistry of Sulfur in the Pressure Leaching of Sulfide Minerals: *David Dreisinger*[1]; Zhimin Zheng[1]; Jianming Lu[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

The pressure leaching of sulfide minerals has become widely applied for the recovery of base and precious metals. The leaching of zinc sulfide concentrates at 150 ?aC in the Dynatec Zinc Pressure Leach process has been successfully applied commercially for over 20 years. Pressure oxidation of refractory gold ores containing pyrite and other sulfide minerals has been applied commercially since the 1980°¶s at T > 190 ?aC. It is widely anticipated that pressure leaching of copper sulfides and in particular chalcopyrite will be the next widespread application of pressure leaching of sulfide minerals. The behaviour of elemental sulfur and the role of surfactants in the pressure leaching of sulfide minerals have received relatively little fundamental study. Sulfur melts at ?a119 C and it is well known that liquid elemental sulfur will tend to wet sulfides and inhibit leaching reactions from proceeding to completion. Surfactants have been widely used in zinc pressure leaching, pressure oxidation of refractory gold ores and are being advocated for the pressure leaching of chalcopyrite. There are two aspects of the interfacial chemistry of sulfur that have been studied

experimentally in a custom built high pressure and high temperature cell. First, the interfacial tension of the liquid sulfur °V aqueous solution system has been studied by photographing droplets of liquid sulfur in solution. The shape of the droplets is then used to calculate the interfacial tension. Second, the contact angle between liquid sulfur and various sulfides has been measured in the presence of various aqueous solutions by photographing droplets of liquid sulfur resting on horizontal mineral specimens. All of these measurements have been performed in the absence and presence of surfactants. In this paper, measurements of interfacial tension and contact angles in the liquid sulfur °V aqueous solution °V mineral system are reported and discussed in relation to current and future applications in zinc, gold and copper pressure leaching.

11:40 AM

Ecologically Safe Technology of Non-Ferrous Metals Obtaining from Sulfide Raw Materials by Means of Bacterial Leaching: *O. V. Slavkina*[1]; N. V. Fomchenko[1]; V. V. Biryukov[1]; [1]Moscow State University of Environmental Engineering, 21/4 Staraya Basmannaya St., Moscow 107066 Russia

The principally new two-stage technological realization of bacterial leaching process by means of bacteria Thiobacillus ferrooxidans is proposed. Optimal conditions for main electrochemical reactions of sulfides oxidizing must be created for the first stage (the active oxidizing agent supply and reaction products withdrawal). The active concentrate subdivision in two fractions (easy and hard oxidizable) happens here. Then these fractions are to be separated and hard oxidizable fraction is returning to the first stage or can be removed from the process. Easy oxidizable fraction is transferring to the second stage of bio leaching. The first stage duration and the suspension separation procedure are to be calculated according to developed method for every processing concentrate. The second stage of bio-leaching shall be carried-out under high activity of microorganisms. Such conditions are contributing to most complete non-ferrous metals transferring into solution and sulfide sulfur transformation into elemental sulfur with its partial oxidation to sulphate-ion. In addition, the sediment, containing mainly ferric iron, sulphate-ions and hydroxide-ions is forming on the second stage. The quantity of sediment is depending on the processing concentrate and pH at the second stage of bio-leaching process. At the same time the ferric iron equilibrium concentration in solution is determined by these parameters. Ferric iron, being an active oxidizer in bio-solution at optimal pH is returning to the first stage of bio-leaching. Process realization according to proposed technological strategy makes possible to achieve higher rates of bacterial leaching in comparison with traditional one-stage process. At the same time two-stage bio-leaching process is in rather good accordance with standard methods of non-ferrous metals recovery from solutions. It is advisable to organize the stage of non-ferrous metals recovery between the first and the second stage of bacterial leaching. Such way permits to obtain selective sediments of non-ferrous metals while the iron (in the form of the ferrous sulphate) is stayed in the solution.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: Copper II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday AM	Room: Santa Rosa
March 5, 2003	Location: San Diego Marriott Hotel

Session Chairs: Florian Kongoli, FLOGEN Technologies Inc., Matls. Tech. Dept., Montreal, Quebec H3S 2C3 Canada; Yasuo Ojima, Sumitomo Metal Mining, Toyo Smelter & Refinery, Saijo, Ehime 793-0005 Japan

8:30 AM Keynote

Future of Copper Converting Process: *Yasuo Ojima*[1]; [1]Sumitomo Metal Mining, Toyo Smelter & Refinery, Otu 145-1 Funaya, Saijo, Ehime 793-0005 Japan

The mainstream of copper smelting process in the world is the combination of Outokumpu type Flash smelting and PS converting process. Presently it will be estimated that in the world copper production about 50% is produced by Flash smelting process and 90% is by PS converting process. PS converting process has been widely used for many smelters more than 100 years owing to its operational flexibility in spite of the batch operation. However, an environmental issue has been pointed out because of the difficulty of fugitive gas handling. Recently several copper smelters in the world have been newly constructed and executed the major modification and modernization. Continuous converting process has emerged some of them instead of PS converting process. This paper describes the comparison of PS and emerging continuous converting process, and the prospect of the future of copper converting process.

9:00 AM

Efficiency of Porous Plugs in Fire Refining of Crude Copper: *C. M. Acuna*[1]; M. Sherrington[2]; [1]Codelco, Chuquicamata Div., Chuquicamata Chile; [2]Instituto Nacional de CapacitaciÛn, INACAP, Calama Chile

In the fire refining of crude copper the use of porous plugs, by stirring with an inert gas, has been mainly considered as a surface renewing agent to increase product quality and/or shortening the refining cycle. However its application is rather limited, specially because of different industrial practices as well as insuficient data on its aside effects on the refractory lining. Furthermore, the combined stirring-reaction mechanism has to be clarified. The standard procedure in fire refining consist of sulfur removal via air injected through tuyeres, arsenic reduction via soda-lime flux and oxygen control by substoichiometrical air or steam fuel mixtures. In the present study the effect of bottom stirring by use of porous plugs and nitrogen gas injection in the treatment of a dirty crude copper, in the range 400-150 ppm sulfur, approximately 2,500 ppm arsenic and 14,000-9,000 ppm oxygen, was investigated. The combined effect stirring-reducing air/steam:fuel mixtures follows a kinetics of order one where the kinetics constant can be represented by a polynomial of the form: K = A * R2 + B * R + C with K as the kinetics constant, A, B and C as constants depending on the stirring nitrogen flow rate and R as the ratio air-fuel or steam-fuel used in the reducing step. If just the oxidation, complexing and reducing steps are considered the process time may be shortened by 30%-40%, depending on the optimization of the air-fuel or steam-fuel mixture used.

9:25 AM Invited

ISASMELTñAn Update on Latest Developments: Philip Arthur[1]; Britt Butler[1]; James Edwards[1]; *Chris Fountain*[1]; Simon Hunt[1]; Philip Partington[1]; Jorma Tuppurainen[1]; [1]Mount Isa Mines, Ltd., Brisbane, QLD 4000 Australia

The Isasmelt process is making a significant contribution to the global metals industry. Isasmelt furnaces are now operating successfully in eight different countries and treating approximately 3 million tonnes of concentrates and secondary materials per year. The process is currently used in both lead and copper smelting. The most recently commissioned plants were in Germany and China, one for treating scrap copper and the other treating low-quality copper concentrates. The Copper Isasmelt furnace at Mount Isa Mines continues to set new operational records, with throughput of more than 1 million tonnes of concentrate per year and very low operating costs. This paper summarises the latest operational data from the Isasmelt plants and introduces plans for the latest process enhancement, a replacement for Peirce Smith Converters.

9:50 AM

Development of Sumitomo Premixed Concentrate Burner for Copper Flash Smelting: *Yasumasa Hattori*[1]; Yoshiaki Mori[1]; Yasuhiro Kondo[1]; Yukihito Sasaki[1]; Toyokazu Okubo[1]; Kozo Baba[1]; [1]Sumitomo Metal Mining Company, Ltd, Niihama Rsrch. Labs., 17-5, Isoura-Cho, Niihama, Ehime 792-0002 Japan

An extensive study including fluid dynamic calculation, cold model tests, pilot plant tests as well as measurements in a concentrate burner of a commercial flash furnace was carried out in order to develop a new Sumitomo flash smelting concentrate burner. The pressure measurements in the commercial burner indicated that the premix of concentrate and reaction air in the burner was insufficient. Pilot plant tests exhibit that the enhancement of the premix of concentrate and reaction air is very effective to improve burner performance. Using the additional pressure drop of the reaction air as an indicator of the

premix, a new type Sumitomo premixing concentrate burner was developed.

10:15 AM Break

10:25 AM

Effect of the Oxygen Potential on the Viscosity of Copper Smelting Slags and its Relation to the Liquidus Surface: *F. Kongoli*[1]; I. McBow[1]; S. Llubani[1]; [1]FLOGEN Technologies, Inc., Matls. Tech. Dept., 5757 Decelles, Ste. 511, Montreal, Quebec H3S 2C3 Canada

Viscosity is an important parameter of copper smelting and converting processes. It affects the settling of matte or metal droplets in the slag, distribution of elements, etc. Despite this importance, considerable confusion exists on the viscosity of copper smelting slags as a result of many disagreements found among the measured viscosity data of several authors especially in the slags of high ferric iron content and close to magnetite precipitation. In this work the effect of oxygen potential on the viscosity of copper smelting slags has been quantified through a coupled quantification of viscosity and liquidus temperature. The confusion in the literature has been clarified and the importance of the relation between the viscosity and the liquidus temperature is discussed.

10:50 AM Invited

Two Copper Smelting Processes at Onsan: *In-Ho Song*[1]; Young-Chul Kang[1]; [1]LG-Nikko Copper, Inc., Daejung-Ri 70, Ulju-Gun, Onsan-Eup, Ulsan City 689-892 Korea

The Onsan flash smelter of LG-Nikko Copper Inc. was commissioned at the capacity of 80,000tpy from copper concentrate in 1979 and its capacity was increased up to 164,000tpy through the two timesí expansion in 1988 and 2001. As the Onsan smelter was started Mitsubishi continuous process in 1998, it has two processes within one plant. This paper outlines two processesí difference and operating dada, i.e., process flow, feed materials, output product quality, impurity distribution, man power, dust generation ratio, power consumption, productivity, steam production, etc.

11:15 AM

High-Intensive Operation of Flash Smelting Furnace at Saganoseki Smelter & Refinery: Masatoshi Ogasawara[1]; *Toshihiro Kamegai*[1]; Masatoshi Maeda[2]; [1]Nippon Mining & Metals Company, Ltd., Saganoseki Smelter & Refinery, Saganoseki-machi, Oita 879-2201 Japan; [2]Metal Economics Reseach Institute, Japan, Mori Bldg. 11, 2-6-4 Toranomon, Minato-ku, Tokyo Japan

Saganoseki smelter & refinery operated two flash smelting furnaces, producing a combined 330,000 metric tons per year (hereinafter mtpy) of copper. In 1996, however, it successfully shifted to single furnace operation while maintaining production at the same level. Since then, the production capacity has been further increased to the present 450,000 mtpy by technological improvements. Also, in order to reduce the converter load which represents a bottleneck in efforts to increase production, the matte grade was gradually increased and currently runs at 65 to 66% attaining the low slag loss of 0.7 to 0.8%. This paper introduces some improvements, recent operation results and thermodynamic analyses conducted.

11:40 AM

The Copper Loss in Slag of Flash Smelting Furnace in Tamano Smelter: *Tsuneo Maruyama*[1]; Nobuyuki Furui[1]; Makoto Hamamoto[1]; [1]Hibi Kyodo Smelting Company, Ltd., No 6-1-1 Hibi, Tamano City, Okayama 706-8511 Japan

In 1972, the Tamano Smelter of Hibi Kyodo Smelting Co., Ltd. went into operation. The Flash Smelting Furnace, used at the Tamano Smelter, was equipped with electrodes that were attached to the settler substitute of the slag cleaning furnace. After that we promoted the development of our own original coke combustion technology. A new Tamano Type Flash Smelting Furnace (T-FSF), without electrodes, was put into operation in 1988. We have continued to improve the technology of coke combustion and combustion the burner. As a result, we have been able to reduce the copper loss in slag as well as achieve stable operation. Furthermore, we have been able to accomplish this while increasing the concentration feed and matte grade. We will continue to strive for high matte grade operation and to raise production efficiency, while paying attention to the important subject of copper loss in slag. In this report, we will present the detailed data with regards to the copper loss in slag reduction, as well as of the influence of the thickness, settling time and coke. We would also like to report the recent operation conditions of the T-FSF.

Notes

15th International Symposium on Experimental Methods for Microgravity Materials Science - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee
Program Organizers: Robert Schiffman, R. S. Research Inc., Barton, VT 05822 USA; Carlo Patuelli, Universita di Bologna, Departimento di Fisica & Instituto Nazionale di Fisica della Materia, Bologna 40127 Italy

Wednesday PM Room: 10
March 5, 2003 Location: San Diego Convention Center

Session Chair: William H. Hofmeister, Vanderbilt University, Cheml. Eng., Nashville, TN 37235 USA

2:00 PM
Spherule Formation of Meteorites on Microgravity Condition by Collision: *Yasunori Miura*[1]; [1]Yamaguchi University, Fac. of Sci., Inst. of Earth Scis., Yoshida 1677-1, Yamaguchi 753-8512 Japan

New type of spherules with high pressure and high temperature is found at dynamic reaction with ultra-high velocity materials of meteoroidal parent body during atmospheric passing. Various types of spherules and melt fragments at meteorite falling site in rice-field after 104 years are found at Niho chondritic meteorite, Yamaguchi, Japan. Direct Simulation of Monte Carlo (DSMC) model can explain dynamic reaction of meteoritic projectile to produce various spherules and melt fragments during atmosphere of the Earth by dynamic fluid reaction under microgravity condition.

2:20 PM
Conduction-Limited Melting in Microgravity: *Afina Lupulescu*[1]; Martin E. Glicksman[1]; Matthew B. Koss[2]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng., 110 8th St. (CII-4223), Troy, NY 12180-3590 USA; [2]College of the Holy Cross, Physics Dept., PO Box 143A, Worcester, MA 01610-2395 USA

Conduction-limited melting is of importance in convection-free processes in low-Earth orbit, during mushy zone fusion, and in weldingñall examples where the length scales for thermal buoyancy are highly restricted. Steady-state crystal growth data reported earlier indicate that dendritic growth under microgravity conditions occurs by pure thermal conduction. We now report on melting process, observed for the first time using video images, where both freezing and melting sequences for pivalic acid (PVA) were observed. PVA dendrites generally melt in a stable manner following an accelerating square-root-of-time dependence. The theoretical kinetics against which these experiments are compared is based on quasi-static kinetic analysis for melting under shape-preserving conditions. Comparison between theory and experiment yield Stefan numbers (dimensionless superheating) in good agreement with thermal data telemetered from the space-borne thermostat. The experiments and their analysis raise several new questions concerning the roles of capillarity, kinetics, and convection during melting processes.

2:40 PM
Impact Macrostructures in the South West Egyptian Region: *E. Farabegoli*[1]; G. Onorevoli[1]; C. Patuelli[2]; R. Serra[2]; [1]Universit‡ di Bologna, Dipartimento di Scienze della Terra e Geologico-Ambientale, Piazza di Porta S. Donato 1, Bologna 40126 Italy; [2]Universit‡ di Bologna, Dipartimento di Fisica ed Istituto Nazionale di Fisica della Materia, Viale Berti Pichat 6/2, Bologna 40127 Italy

The area in the South West region of Egyptian Great Sand Sea was analysed by remote sensing using multi-spectra images in order to investigate the impact structures. The research was focused on old empty craters, old craters filled with alluvial-lake sediments and on elongated linear structures by abrasion on the rocky substrate. The results are discussed taking into account the recent impact or volcanic theories.

3:00 PM
The Measurement of Precise Solute Diffusion Coefficients in Molten Metals and Semiconductors: The Effect of Gravitational Fields: *Reginald William Smith*[1]; Weidong Huang[1]; B. J. Yang[1]; [1]Queenís University, Matls. Sci. of Microgravity Applic., Nicol Hall, Rm. 228, Kingston, Ontario K7L 3N6 Canada

This paper will describe the development and characterisation of a new shear cell for use with the Advanced Thermal Environment Facility(ATEN) being developed for the ISS by the Canadian Space Agency.

3:20 PM Break

3:40 PM
The Effects of Improved Heat Extraction on the Microstructure of Directionally Solidified Immiscible Alloys: *J. V. Puckett*[1]; J. B. Andrews[1]; [1]The University of Alabama-Birmingham, Microgravity Solidification Lab., 1150 10th Ave. S., BEC 361, Birmingham, AL 35294 USA

Immiscible alloy systems that possess a high-miscibility gap can generally be solidified under coupled growth conditions to produce an aligned, fibrous microstructure. However, under certain processing conditions, rows of aligned spheres are more likely to form in some alloys, instead of the more desirable fibrous microstructure. Speculative theories about the mechanisms leading to this aligned droplet structure include Rayleigh breakdown of the liquid ìfibersî into droplets near the solidification front and oscillatory variations in solidification rate that cause variations in fiber diameter that facilitate the breakdown of fibers into doubly orientated arrays of droplets. This investigation focuses on the influence of efficient heat extraction during solidification on the tendency of samples to form droplets as opposed to fibrous microstructures by virtue of reducing the time that is available for cylindrical instabilities or ripening to occur after primary solidification. Microstructures corresponding to different heat extraction rates as well as the influence of processing alloys slightly off monotectic composition will be presented.

4:00 PM
The Influence of Gravity on the Solid-Liquid Interfacial Free Energy: *Reginald William Smith*[1]; C. Patuelli[2]; R. Tognato[2]; [1]Queenís University, Matls. Sci. of Microgravity Applic., Nicol Hall, Rm. 228, Kingston, Ontario K7L 3N6 Canada; [2]Universita di Bologna, Dipto. di Fisica & Inst. Nazionale di Fisica della Materia, Alma Mater Studiorum, Bologna 40127 Italy

Theoretical analysis suggests that the free energy of a solid/liquid surface, and hence the local surface tension, may be influenced by the gravitational field. This paper reports recent experimental work which attempts to establish the physical significance of this analysis.

4:20 PM
Investigation of Libyan Desert Glass by X-Ray Micro-Diffraction Analysis: *C. Patuelli*[1]; R. Serra[1]; S. Coniglione[1]; M. Chiarini[2]; [1]Universitaí di Bologna, Dipartimento di Fisica ed Istituto Nazionale di Fisica della Materia, Viale Berti Pichat 6/2, Bologna 40127 Italy; [2]INFM Unit‡ di Bologna, Via Gobetti 101, Bologna 40129 Italy

Samples of Libyan Desert Glass (LDG) collected by Serra et al. (1) in the South West region of Egyptian Great Sand Sea were analyzed by X-ray micro-diffraction technique. A short order crystal structure was observed using a well-collimated X-ray Cu k□ beam of 30 m in diameter and interesting FCC metal alloys phases were identified by matching the whole LDG spectrum. The matched phases confirm that this natural tektite glass has an extraterrestrial origin. The important role played by micro-gravity environment during melting and solidification of this nearly pure silica (98% SiO2) is taken into account and it is advanced the hypothesis that SiO2 was still present in the comet core before the impact. BARAKAT A. A., DE MICHELE V., NEGRO G., PIACENZA B. and SERRA R. (1997). Some new data on the distribution of Libyan Desert Glass (Great Sand Sea, Egypt) in Proc. ´ Silica 96 ª Meeting on Libyan Desert Glass and related desert events. Bologna University, July 18 1996. Proceedings. Segrate (Milano), Pyramids, 29-36.

Actinide Materials: Processing, Characterization, and Behavior: Advanced Fuels and Materials II

Sponsored by: Light Metals Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Nuclear Materials Committee-(Jt. ASM-MSCTS), LMD-Reactive Metals Committee
Program Organizers: Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division Materials Development Section, Argonne, IL 60439-4837 USA; Michael F. Stevens, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Wednesday PM Room: 4
March 5, 2003 Location: San Diego Convention Center

Session Chair: Sean M. McDeavitt, Argonne National Laboratory, Cheml. Tech. Div. Matls. Dvlp. Sect., Argonne, IL 60439-4837 USA

2:00 PM

Production of Urania/Thoria Microspheres by Spray Drying: *Alvin Solomon*[1]; S. Anthonysamy[1]; S. Kuchibhotla[1]; [1]Purdue University, Sch. of Nucl. Eng., W. Lafayette, IN 47907 USA

Spray drying has been used to produce (U,Th)O2 fuel microspheres for dispersion fuels. This methodology avoids the problem of non-recyclable waste streams, but the recyclable dry ìfinesî must be collected and controlled. A facility has been built for performing the spray drying using a commercial labscale spray dryer with an upward-facing two fluid nozzle and counter flow heated air. Dryer modifications include a vessel extension to increase sphere size, and a remote cleaning system. Safety is assured because the system operates at negative pressures during operation or automatically shuts down. The fines are collected in a sump/filtration system. Optimized slurries were obtained by controlling the Zeta Potential, slurry viscosity and rheology. Spray drying runs were carried out using 25 to 30% solids loading with various percentages of triethanolamine as dispersing agent and binder. Spheres of ~90%TD and a modal size of 200 microns were obtained with uniform porosity.

2:25 PM

Precursor Selection for Chemical Vapor Deposition of Niobium on Zirconia Microspheres: *Christine T. Snyder*[1]; Jude M. Runge[1]; Thomas C. Carter[1]; Andrew S. Hebden[1]; Bryce W. Campbell[1]; Sean M. McDeavitt[1]; [1]Argonne National Laboratory, Chem. Tech. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA

As part of the effort to study non-fertile nuclear dispersion fuel containing (Pu, Am, Np)O2 for actinide transmutation systems, a chemical vapor deposition (CVD) method is under development for coating non-radioactive zirconia microspheres with niobium. In the CVD process, the solid film results from the decomposition and reaction of the chemical constituents of a precursor, or starting material. Precursor selection is one of the crucial steps in avoiding nucleation of solid particles that can result in films containing impurities, defects, particulates, as well as poor adhesion. In this investigation, precursor selection was evaluated using the following criteria: 1) chemistry of reaction (intermediate steps, by-products), 2) volatility and temperature of vaporization, 3) composition and stability at room temperature, and 4) hazards and toxicity.

2:50 PM

Compatibility of U-Mo Alloys and Al in RERTR Dispersion Fuels: *Dennis D. Keiser*[1]; Curtis C. Clark[1]; Mitchell K. Meyer[1]; [1]Argonne National Laboratory, Eng. Tech. Div., PO Box 2528, Idaho Falls, ID 83403-2528 USA

Argonne National Laboratory is developing fuels to convert reactors that employ fuels containing highly-enriched uranium to fuels that contain low-enriched uranium (less than 20% 235U). This work is being done as part of the Reduced Enrichment for Research and Test Reactors program (RERTR). The leading candidate fuel for this conversion is a plate-type fuel that has a fuel meat comprised of U-Mo alloy powders dispersed in an Al matrix. This fuel meat is clad in Al. When these dispersion fuels are irradiated in-reactor, reaction occurs between the Al matrix and the U-Mo alloy powders. As a result, out-of-reactor diffusion studies have been performed to investigate the interdiffusion behavior of U, Mo, and Al. This paper will discuss the results from diffusion tests that employed solid-solid diffusion couples that were annealed at temperatures from 400∞C to 640∞C for varying times. Couples were examined for diffusion structure development using a scanning electron microscope equipped with both energy-dispersive and wavelength-disepersive spectrometers (SEM/EDS/WDS). Point-by-point and linescan analysis was used to generate composition profiles and diffusion paths. The interdiffusion behavior of the various elements and development of interdiffusion zones will be discussed. Comparisons will be made to structures observed for fuels irradiated in a reactor.

3:15 PM

The Morphology and Structure of Annealed U-Nb-W Alloys: *E. Kahana*[1]; M. Talianker[1]; A. Landau[2]; A. Venkert[2]; [1]Ben-Gurion University of the Negev, Dept. of Matls. Eng., PO Box 653, Beer-Sheva 84105 Israel; [2]Nuclear Research Center-Negev, PO Box 9001, Beer-Sheva Israel

Dilute uranium alloys with small additions of the second transition metal solute such as molybdenum, niobium, titanium and zirconium, have been the subject of extensive investigations. In general, these alloying additions, extensively soluble in the high temperature uranium γ phase, are almost completely insoluble in the low temperature a phase. Therefore, on slow cooling rate the γ phase decomposes via diffusional phase transformation forming a two-phase structure of a phase and either an intermetallic phase or an alloy rich γ_o phase. This work presents the effect of addition of tungsten as a second solute element, on the structures formed in annealed ternary U-14at%Nb-W alloys. The solubility of tungsten in uranium is extremely limited, less than 1 at%, and there is no intermetallic compound in the uranium-tungsten system, however, there is full solubility of tungsten in niobium. The niobium content in the alloys was chosen near the eutectoid composition of U-Nb system. The range of the tungsten content was between 0.6at%W to 2.3at%W. In common with other U-Nb base alloys it was found that the microstructure of annealed U-Nb-W alloy consists of two main phases: orthorhombic α-uranium and γ_o tetragonal phases that form as a result of eutectoid reaction. However, in addition to these expected phases, the existence of dendrites and small particles containing U-Nb-W were found. Longer annealing periods resulted in the formation of only one type of small precipitates of a new ternary U-Nb-W phase. The composition of this phase as determined by EDS analysis in TEM was Nb=49.75at%, W=45.71at%, U=4.54at%, and the unit cell was identified as cubic BCC with the lattice parameter a=3.185≈.

3:40 PM Break

4:00 PM

Testing of Commercial Materials for Melt Containment of Uranium-Bearing Metal Waste Form: *Ken C. Marsden*[1]; [1]Argonne National Laboratory-West, PO 2528, Idaho Falls, ID 83403 USA

Electrometallurgical processing of metal fuel rods at ANL-W generates chopped stainless steel cladding coated with process salts and containing small amounts of residual uranium. This waste stream will be melted and alloyed with zirconium to form a dense, corrosion resistant waste form. The resulting alloy is approximately SS304-15Zr-7U and is formed by melting near 1600C. The waste form is allowed to solidify in the crucible. Development testing has proceeded in several furnaces to explore feasibility of commercially available materials. The materials problem is complicated by the presence of reactive chloride species in the process salt. Various commercially available materials have been explored, including coated graphite, yttria, boron nitride, and alumina. Tests have been conducted at charge masses between 3 and 69 kg. An inexpensive alumina refractory displays good resistance to the process when coated with a thin oxide coating.

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The Mechanical Response of Depleted Uranium as a Function of Strain Rate and Temperature: *Carl M. Cady*[1]; Shuh-Rong Chen[1]; George T. (Rusty) Gray[1]; David F. Teter[1]; Dan J. Thoma[1]; Gary K. Lewis[1]; Deniese R. Korzekwa[1]; Philip K. Tubesing[1]; Ann M. Kelly[1]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA

The compressive stress-strain response of several wrought depleted uranium (DU) ingots as a function of temperature and strain rate was investigated. The yield and flow stresses of DU was found to exhibit a pronounced sensitivity to strain rate, temperature and carbon content. The stress-strain response of uranium under quasi-static uniaxial tension will be compared to the compression data to show the sensitivity of stress-state. Constitutive modeling efforts are underway to predict the mechanical behavior of materials outside the regions where data for strain-rate and temperature already exists. The Mechanical Threshold Strength Model can accurately capture the constitutive response of DU. The Taylor impact test was used to validate the model and show how well the model fit works even outside the region where experimental data exists.

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Modern Day Alchemy: Development of Ceramic Fuels for the Transmutation of Nuclear Waste: *Ryan F. Hess*[1]; R. Margevicius[1]; K. McClellan[2]; M. Stan[2]; K. Sickafus[2]; S. Voit[1]; E. Henderson[2]; G. Egland[2]; [1]Los Alamos National Laboratory, NMT-11, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

Transmutation fuels work at Los Alamos National Laboratory is currently focused on mononitride ceramic fuel forms, and consists of closely coordinated actinide (hot) and inert and surrogate (cold) fuels work. This work involves three major components: 1) fuel material synthesis and fabrication, 2) fuel performance, and 3) fuel materials modeling. Results from all three components will be presented. The cold work focuses on the synthesis and ceramic processing of the proposed ZrN fuel matrix. The actinide (hot) fuels effort at Los Alamos emphasizes the synthesis and fabrication of actinide-bearing nitride fuel pellets. These pellets will be inserted into the Advanced Test Reactor early in 2003 and continue for the next couple of years. The nitride pellets are designed to contain varying amounts of Pu, Am, Cm, and Np. Results presented will outline fabrication techniques which are designed to reduce the volatility of americium.

Aluminum Reduction Technology: Advanced Processes

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

Wednesday PM Room: 6B
March 5, 2003 Location: San Diego Convention Center

Session Chair: Harald Oye, Norwegian University of Science and Technology, Dept. of Chem., Trondheim N-7491 Norway

2:00 PM
Aluminum Carbothermic Technology: Comparison to Hall-Heroult Process: *Marshall Bruno*[1]; [1]Alcoa Inc., Next Generation Al. Processes, Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069-0001 USA

Production of aluminum by carbothermic reduction has been investigated by every major aluminum company over the past forty seven years. As an alternative to the Hall-Heroult process, carbothermic offers potential energy, cost and environmental advantages if solutions to critical technical hurdles could be developed. These include efficient delivery of energy to attain 2000-2200C, while minimzing loss of volatile aluminum-containing species; capture and recycle of those volatiles that do evolve to recover mass and energy values; effective decarbonization of the Al-C metal phase; and recovery of energy values in the by-product CO to reduce net energy consumption. To address these hurdles, Alcoa and Elkem, with support from DOE, initiated development of a new Advanced Reactor Process. Previous efforts by the industry will be reviewed; estimates will be given for energy, cost and environmental benefits from the new process; and process challenges will be identified.

2:25 PM
Aluminum Carbothermic Technology: Alcoa-Elkem Advanced Reactor Process: *Kai Johansen*[1]; Jan A. Aune[2]; Marshall Bruno[3]; Anders Schei[4]; [1]Elkem ASA Research, PO Box 8040 Vaagsbygd, Kristiansand N-4602 Norway; [2]Elkem Technology a/s, PO Box 4376, Torshov , Oslo N-0402 Norway; [3]Alcoa Inc., Next Generation Al. Processes, Alcoa Techl. Ctr., 100 Techl. Dr., Alcoa Ctr., PA 15069-0001 USA; [4]Consultant, Formerly of Elkem ASA Research, Kristiansand N-4602 Norway

Alcoa and Elkem are jointly developing the Advanced Reactor Process for production of aluminum by carbothermic reduction of alumina. The new process is based on a continuous, staged, ultra high intensity electric slag resistance reactor utilizing Elkemís state-of-the-art high temperature smelting technology. The reactor design meets the specific process requirements for aluminum as defined by Alcoa, based on the thermodynamic properties of the Al-O-C system, an operating diagram of the chemistry involved, and fundamental studies of vapor recovery parameters including computor modeling, conducted with Carnegie Mellon University. The thermodynamics will be reviewed; the process will be described; progress and future program will be reported.

2:50 PM
A Finite Element Computational Fluid Dynamics Sensitivity Analysis for the Conceptual Design of a Carbothermic Aluminium Reactor: *Dimitrios I. Gerogiorgis*[1]; B. Erik Ydstie[1]; [1]Carnegie Mellon University, Dept. of Cheml. Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Carbothermic reduction is a nontraditional chemical process for aluminium production, based on the endothermic chemical reduction reaction occurring between aluminium oxide and carbon. This process has potential for drastic reduction of fixed and operation costs of the investment. Furthermore, it is environmentally benign and in principle significantly more energy-efficient, as the costly electrolytic ionization is effectively avoided by the direct chemical reduction pathway. This method is identified as a potential alternative to Hall-HÈroult electrochemical reduction by several studies, but its complexity still poses remarkable technical obstacles for implementation. A conceptual multistage electrothermic reactor recently proposed (Johansen, Aune et al., 2000) is a quite attractive idea for achieving reactor scaleup but entails significant design challenges. Actual carbothermic reduction of alumina occurs in the second stage of this multiphase reactor: a high-temperature molten slag formed at a temperature of ca. 1950∞C (1st stage) is fed into a submerged arc furnace (2nd stage), where it reacts at a temperature of ca. 2050∞C to CO and Al. Temperature and flow control are crucial in achieving high yield and minimal Al evaporation. The present study elaborates on a finite element CFD model developed for the conceptual reactor (Gerogiorgis et al., 2001), considering only a reactor sector to minimize computational expense. The rectangular computational domain comprises a cylindrical electrode and symmetry planes, and a pseudohomogeneous molten slag is assumed, so as to relax multiphase flow complications. Appreciable modeling complexity is caused by the interaction and nonlinear couplings among the Joule electrothermic phenomenon within the electric field between the inert DC electrodes, the endothermic reduction reaction (modeled by considering Arrhenius-type heat consumption) and the Boussinesq natural convection that is inducing a vertical recirculation of the molten slag. The complete triple-PDE problem (electric charge balance, heat balance and momentum balance) is solved using a commercial, modular PDE modeling suite (FEMLABÆ v. 2.2-COMSOL) in order to obtain the potential, temperature and velocity distributions, respectively, and thus probe the combination of Joule heating, endothermic reaction and Boussinesq natural recirculation. The main objective of the present CFD simulation study is to extract reactor design conclusions by explicitly addressing the effect of process parameters on the temperature and velocity fields. A sensitivity analysis is thus being conducted with respect to a number of design variables (reactor dimensions, electrode length, imposed voltage); profiles obtained reveal the nontrivial optimization problem arising when sufficient heating is to be maintained at adequate circulation. This delicate balance is obvious in preliminary experimental observations of a pre-pilot plant and is believed to govern Al production rates; thus, it is quintessential to feasibility and profitability.

3:15 PM Break

3:25 PM
An Improved Pyroconductivity Test of Spinel-Containing Cermet Inert Anodes in Aluminum Electrolysis: *Yanqing Lai*[1]; Qingwei Qin[1]; Zhongliang Tian[1]; Gang Zhang[1]; *Jie Li*[1]; Yexiang Liu[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

An improved pyroconductivity test device, consisting of a specially constructed closed furnace and a potentiostat, was constructed based on the conventional direct current four-point technique. Symmetrical current distribution in the specimen was obtained by keeping a fixed pressure and good contact between the specimen and clamps at any temperature. The potentiostat was used to supply direct current and continuously monitor the current intensity and voltage between two probes, which can be adjusted outside the heating furnace to well contact with the specimen. Test results of copper and graphite specimens show that the reliability and reproducibility were excellent. The electrical conductivity as a function of temperature for various spinel-containing cermet inert anodes was investigated spanning the Hall cell operating temperature. The factors influencing the electrical behaviour were studied, which included the particle size of row materials, manufacture process, phase composition and morphology et al.

3:50 PM
On Expansion of TiB2-Carbon Composites Used as Wettable Cathode in Aluminum Electrolysis Due to Sodium Penetration: *Qingyu Li*[1]; Yanqing Lai[1]; Jianhong Yang[1]; Jie Li[1]; Hengqin Zhao[2]; Yexiang Liu[1]; [1]Central South University, Sch.of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China; [2]Zhengzhou Institute of Multipurpose Utilization of Mineral Resources, CAGS, No. 328, Longhai W. Rd., Zhengzhou, Henan 450006 China

The current Hall-Heroult process could be renovated by introduction of new cell design based on the TiB2 wettable cathode, inert anode and other materials. In the present work, a convenient method for the preparation of the TiB2-Carbon Composites cathode materials composed of TiB2 thermosetting resins and carbon was studied by means of the cold press forming and sintering. The relations between the sintering temperature or the TiB2 content and sodium penetration into the composite were measured. The sodium penetration into the composite was much less than that into the current carbon materials. The increase of sintering temperature and the TiB2 content was beneficial to reduce sodium penetration and expansion of the composite resulted from sodium penetration.

4:15 PM
Modeling of the Solubilities of NiO/NiAl2O4 and FeO/FeAl2O4: Yunshu Zhang[1]; Xiaoxia Wu[1]; *Robert A. Rapp*[1]; [1]The Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Based on the literature data, combined with a number of solubility data measured by the present authors, the solubility of $NiAl_2O_4$ in

Al_2O_3-saturated cryolite melts at 1300K were modeled thermodynamically in terms of two Ni-containing acidic solutes Na_2NiF_4 and Na_4NiF_6 over a wide composition range of 2 ≤cryolite ratio r ≤12. Equilibrium constants and ΔG o/f values for the formation of these two solute species were thereby estimated. This two Ni-containing solute model provides an essentially perfect match with the experimental solubility values. The solubilities of $NiO/NiAl_2O_4$ in Al_2O_3-undersaturated cryolite melts in dependence upon dissolved Al_2O_3 concentration were calculated from the present model and plotted for a number of melt compositions. Similarly, the solubilities of $FeO/FeAl_2O_4$ in cryolite melts with dissolved Al_2O_3 at 1300K were modeled in terms of Fe-containing solute complexes.

4:40 PM
Result of Aluminum Electrolysis Test of Minitype Cermets Anode: *Qun Zhao*[1]; Yanli Xie[1]; Zhuxian Qiu[1]; [1]Northeastern University, Nonferrous Dept., 3 Ln., 11# Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China

A minitype NiFe2O4 based cermets was tested in low temperature electrolyte. Test cells operated under the following conditions. Anode current density was set at 1 A cm-1 and the current was kept at 22A accordingly. Alumina was added in bath every 1 hour, 5g for each time. Electrolysis was conducted at a temperature of 850° and last 11 hours. Gas ejection from the surface of anode was observed and a few amount of metal was obtained. The cracks were formed in the bottom of anode after test and problems were existed at electricity resource and anode joint. With EPM-810 type microscope, changes of microstructure and element distribution in cermets anode were studied and the corrosion process of anode during electrolysis was discuss.

Carbon Technology - IV

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Amir A. Mirchi, Alcan Inc., Arvida Research and Development Centre, Jonquiere, QC G7S 4K8 Canada; Don T. Walton, Alcoa Inc., Wenatchee Works, Malaga, WA 98828-9784 USA; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday PM Room: 6D
March 5, 2003 Location: San Diego Convention Center

Session Chair: Philippe Beghein, SGL Carbon Group, Business Line Cathodes Techl. Serv., Passy 74190 France

2:00 PM
Operational Changes in Carbon Cathode of Aluminum Cell: *S. M. El-Raghy*[1]; M. O. Ibrahiem[2]; F. M. Ahmed[2]; H. A. Ahmed[1]; [1]Cairo University, Fac. of Eng., Cairo Egypt; [2]Aluminium Company of Egypt, Naga Hammad Egypt

Since its start, 1975, Aluminium Company of Egypt, EgyptAlum, is using two types of anthracite cathode blocks. Graphite content of these cathodes were either 20% or 50%. This paper is reporting EgyptAlumís experience over these years with respect to structural changes in the carbon cathode during operation. Graphitization, oxidation of carbon cathode as well as sodium and ash content have been studied as a function of cell life over 10 years for the two types of the cathode blocks mentioned above. The exothermic peak of carbon block, by DTA, decreased with increasing cathode life. Oxidation temperature of new carbon sample is 680°C while it comes down to 620°C and 615°C for samples of 1 and 4 years life respectively. Electrical resistivity decreased from 41.5 to 25 µohm.m for cathode blocks after 10 years service. The initial graphite content proved to have some influence on structural changes during operation.

2:25 PM
Multiaxial Mechanical Behavior of the Carbon Cathode: Understanding, Modeling and Identification: *Guillaume DiAmours*[1]; Mario Fafard[1]; Augustin Gakwaya[1]; Amir A. Mirchi[2]; Olivier Doucet[1]; [1]Laval University, Sci. & Eng. Fac., Adrien-Pouliot Bldg., Sainte-Foy, QuÈbec G1K 7P4 Canada; [2]Alcan Inc., Arvida R&D Ctr., PO Box 1250, JonquiËre, Quebec G7S 4K8 Canada

As part of the on-going project START-Cuve, involving the thermo-electro-mechanical modelling of a cell coke bed electrical preheating, a new mechanical constitutive model has been developed for the simulation of the multiaxial behavior of carbon cathode during preheating and heat-up of aluminum electrolysis cells. This model will be able to calculate strains and stresses that are generated in the cathode during thermally induced expansion of the lining. This paper presents the mechanical development of the model who was guided by recent experimental observations on laboratory specimens which were loaded under direct tension, uniaxial compression, shear and multiaxial compression field at different levels of lateral confinement. All tests have been done at ambient temperature. A single surface plasticity model was formulated to account for the dependence of both strength and ductility of carbon under confinement. The constitutive parameters of the model have been calibrated from the experimental results.

2:50 PM
Performance of Graphitized Carbon Cathode Blocks: *Andreas Schnittker*[1]; Holger Nawrocki[1]; [1]Erftcarbon GmbH & CoKG, Aluminiumstrasse 4, Grevenbroich 41515 Germany

The use of fully graphitized cathode blocks, particularly in modern high-amperage technology cells, is growing. It is important to understand the specific requirements and demands of individual reduction technologies, and to characterize the performance trend of graphitized blocks in existing applications in order to optimize the blockís properties for future projects. The most important characteristics are the Electrical Resistivity, wear rate (erosion) and Thermal Properties. The operational performance is determined by a collection of factors e.g. raw material, granulometry, forming technology, baking, impregnation and graphitization. Recently, impregnation and the customized fine tuning of the graphitization process are used to enhance critical properties targeted at specific operational and performance improvements. The aim of this paper is to give an overview of the state of the art and an outlook for future developments and applications.

3:15 PM
Improvement of Abrasion Resistance of Graphitized Cathode Block for Aluminum Reduction Cells: *Shinjiro Toda*[1]; Tsutomu Wakasa[1]; [1]Nippon Electrode Company, Ltd., R&D Dept., 5600 Kambara, Kambara-cho, Ihara-gun, Shizuoka-ken 421-3203 Japan

Abrasion resistance of graphitized cathode is a key parameter to attain longer cell life. An apparatus for abrasion resistance measurement was developed to evaluate cathode blocks in aluminum electrolysis cells. Abrasion resistance of various kinds of cathode blocks, including anthracite based carbon blocks, was measured with this apparatus. The abrasion rate of graphitized blocks was found to be 3 times larger than that of anthracite blocks. This result agrees with the erosion rate of each grade of carbon blocks in the actual cells. Several cokes were investigated as raw material for graphitized blocks. A coke from high quinoline insoluble content pitch showed good hardness. This coke has large areas of optical isotropic texture and graphitized carbon blocks with this coke show good abrasion resistance relative to conventional grade.

3:40 PM Break

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Laboratory Test Method for Measuring Wear Rates of Carbon Cathode Materials: *Frank Hiltmann*[1]; Henrik Gudbrandsen[2]; Sverre Rolseth[2]; Lisbet St□en[2]; Jomar Thonstad[3]; [1]SGL CARBON GmbH, CFL, Griesheim Plant, Stroofstrasse 27, Frankfurt 65933 Germany; [2]SINTEF, Matl. Tech., Trondheim 7465 Norway; [3]Norwegian University of Science and Technology, Trondheim Norway

The wear of graphitized cathode blocks used in industrial aluminium electrolysis limits the lifetime of the cells. To study this problem a laboratory cell was developed to determine chemical wear rates of carbon cathode materials. It is based on the assumption that the carbon lining reacts with aluminium and is subsequently transferred to the cryolite bath in the form of dissolved carbide, which is eventually oxidised at the anode. The test cell that simulates this sequence consists of a sintered alumina crucible, housing a carbon test sample underneath an aluminium pad, and an oxygen-evolving inert anode immersed in the bath. As the only source of carbon is the cathode specimen, the rate of carbon transfer is monitored via the amount of CO2 and CO in the anode off-gas. Resulting wear rates are of the same magnitude as wear rates experienced in industrial cells, i.e. 2-5 cm per year.

4:15 PM
A New Technology for Cathode Rodding Used in Aluminium Electrolytic Cells: *Martin GagnÈ*[1]; Chantal Labrecque[1]; Dany Lavoie[2]; Annie LÈvesque[2]; Brian Murphy[2]; [1]Rio Tinto Iron & Titanium, 770 Sherbrooke St. W., Ste. 180, Montreal, Quebec H3A 1G1 Canada; [2]Aluminerie Alouette, Sept-Iles, Quebec Canada

The quest for the reduction of electrical resistance of the cathode blocks of aluminium smelting electrolytic cells has put every component of these cells under scrutiny, including the high phosphorus grey iron (HPGI) used as sealant between the steel cathodic bar and the graphite block. During this program, a detailed characterization of the as-cast and after-service HPGI roddings was performed and their microstructure and properties compared to those of ferritic Ductile Iron

(FDI), a potential substitution material. Once the feasibility of production of FDI rodding confirmed, FDI cathode roddings were cast. Up to now, 130 cathode blocks have been put in service and six full cells are operational, the oldest one since August 2000. In this paper, the promising performance of the cathodes in service, which is probably related to the higher electrical conductivity and thermal stability of FDI, is discussed in function of cell stability and cathodic distribution.

4:40 PM
Electrical Resistance of Al-Steel Transiton Joints vs. Time and Temperature: *John G. Banker*[1]; Antoine Nobili[2]; [1]DMC Clad Metal Division, 5405 Spine Rd., Boulder, CO 80301 USA; [2]DMC Nobelclad Division, 1 Allee Alfred Nobel, Rivesaltes 66600 France

Electric transition joints (ETJ) are used for welding aluminum to steel in reduction cells. ETJs can experience temperatures up to 600∞C which can cause strength loss and resistance increase. This results form formation of brittle, high resistance Fe-Al intermetallics at the bond. Over the time required for mechanical failure to occur, the resistance can increase dramatically. The increase can be altered by inserting a titanium or chromium interlayer. Compared to bi-clad, the temperature at which resistance begins to increase is raised by 200∫ with chrome, and 300∫C with titanium. As-manufactured, resistance of a standard bi-clad specimen is 38.8 m-ohm, chromium interlayer ETJ is 55.8, and titanium interlayer ETJ is 44.4. After 275 hours at 500∞C, the bi-clad had fallen apart, the chrome interlayer ETJ resistance had increased to 11,250 and the titanium interlayer ETJ exhibited no change.

Cast Shop Technology: Solidification and Foundry Technology I

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

Wednesday PM Room: 6C
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Peggy Jones, General Motors Corporation, Powertrain, Saginaw, MI 48602-2641 USA; Philippe Jarry, Pechiney Group, Ctr. de Recherches, Voreppe 38341 France

2:00 PM
Effects of Hydrogen/Solidification Rate/ and Ca on Porosity Formation in As-Cast Aluminum Alloy A356: *Prince N. Anyalebechi*[1]; [1]Grand Valley State University, Padnos Sch. of Eng., L. V. Eberhard Ctr., Ste. 718, 301 W. Fulton, Grand Rapids, MI 49504-6495 USA

The effects of solidification rate, relatively high levels of melt hydrogen content and Ca on the amount and characteristics of porosity formed during solidification of aluminum alloy A356 have been quantitatively determined. Unidirectionally cooled laboratory-size ingots with solidification rate range of 0.2-7.5 K/s, 0.27-0.63 cc/100 g melt hydrogen concentration, and 0-52 ppm of Ca were used for the study. Amount of porosity and average pore size increased with increase in hydrogen concentration, but decreased with increase in solidification rate. At the 0.27 cc/100 g hydrogen level, 59 ppm of Ca discernibly decreased the amount of porosity. This is provisionally attributed to the contention that, like alkali (Li and Na) and other alkaline earth (Mg) metals, Ca probably increases the solubility of hydrogen in aluminum and its alloys.

2:25 PM
Optimal Heat Treatment of A356.2 Alloy: *Daryoush Emadi*[1]; Laurence V. Whiting[1]; Mahi Sahoo[1]; Jerry H. Sokolowski[2]; Paul Burke[3]; Mitch Hart[3]; [1]CANMET, Matls. Tech. Lab., 568 Booth St., Ottawa, Ontario K1A 0G1 Canada; [2]University of Windsor, NSERC/Ford-Nemak Industl. Rsrch. Chair, Rm. 209, Essex Hall, 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [3]Grenville Castings, 2 Air Care Dr., PO Box 785, Smiths Falls, Ontario K7A 4W6 Canada

The factors necessary to obtain an optimal heat treatment of ASTM B108 test bars in A356.2 were investigated. Test bars were solutionized at various times and temperatures, and then quenched in different media and cooling rates. In addition, the length of natural aging prior to artificial aging was studied to examine the scatter in the properties. The mechanical properties, electrical conductivity and hardness were measured for various artificial aging times and temperatures. Dimensional changes during heat treatment were measured by dilatometry. For test bars, an optimal heat treatment consists of a 4 hour solutionizing at 540°C, followed by water quenching, and then 12-20 hours natural aging followed by 6 hours aging at 155°C. This treatment has the advantages of good properties, with good reproducibility (least scatter and standard deviation), lower energy per heat treatment cycle and higher furnace throughput.

2:50 PM
Precipitation of Dispersoids in DC-Cast AA3103 Alloy During Heat Treatment: *Yanjun Li*[1]; Lars Arnberg[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech., 7491, Trondheim Norway

The precipitation behavior of dispersoids in DC cast AA3103 alloy during heating and homogenization at 600∞C has been studied by means of TEM, electrical conductivity measurement and image analysis. During heating, a-Al(Mn,Fe)Si is the first phase to precipitate in the alloy. When heated to higher temperature, long rod like and plate like Al6(Mn,Fe) dispersoids precipitate in the alloy. During homogenization, the size of Al6(Mn,Fe) dispersoids grows with homogenization time while a-Al(Mn,Fe)Si dispersoids dissolve quickly. The evolution of dispersoids during heat treatment is controlled by nucleation, growth, coarsening and dissolution. The size, size distribution and number density of dispersoids have been measured. The volume fraction of dispersoids formed during heating, measured from TEM images, is in good agreement with the volume fraction calculated from the electrical conductivity of the alloy.

3:15 PM
Modelling of the Thermo-Physical and Physical Properties for Solidification of Al-Alloys: *Nigel Saunders*[1]; Xiuqing Li[2]; Alfred Peter Miodownik[1]; Jean-Philippe Schille[2]; [1]Thermotech, Ltd., Surrey Tech. Ctr., The Surrey Rsrch. Park, Guildford, Surrey GU2 7YG UK; [2]Sente Software, Ltd., Surrey Tech. Ctr., The Surrey Rsrch. Park, Guildford, Surrey UK

The thermo-physical and physical properties of the liquid and solid phases are critical components in casting simulations. Such properties include the fraction solid transformed, enthalpy release, thermal conductivity, volume and density all as a function of temperature. Due to the difficulty in experimentally determining such properties at solidification temperatures, little information exists for multi-component alloys. As part of the development of a new computer programme for modelling of materials properties (JMatPro) extensive work has been carried out on the development of sound, physically based models for these properties. Wide ranging results will be presented for Al-based alloys, which will include more detailed information concerning the density change of the liquid that intrinsically occurs during solidification due to its change in composition.

3:40 PM Break

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Effects of Solidification Rate/Fe/ and Ca on the Cast Microstructure of Aluminum Alloy A356: *Prince N. Anyalebechi*[1]; [1]Grand Valley State University, Padnos Sch. of Eng., L. V. Eberhard Ctr., Ste. 718, 301 W. Fulton, Grand Rapids, MI 49504-6495 USA

The effects of solidification rate, Fe, and Ca on the characteristics of the constituent phases formed during solidification of aluminum alloy A356 have been investigated. This involved the use of unidirectionally cooled laboratory-size ingots with a range of solidification rate of 0.2-7.5 K/s. Two levels Fe (0.05 wt.% and 0.20 wt.%) and Ca (0 ppm and 59 ppm) were investigated. As expected, average dendrite cell and constituent phase particle size decreased exponentially with increase in solidification rate. Effect of Ca depended on the Fe content in the alloy. For example, at 0.20 wt.%Fe and 0.05 wt.%Fe, Ca slightly increased and decreased, respectively, the average constituent phase particle size. At the low levels investigated, Fe content did not affect the average size and/or volume fraction of the constituent phases.

4:15 PM
Microstructure Formation in Directionally Solidified Al-Cu Alloys: *Shan Liu*[1]; Je H. Lee[1]; Heath Walker[1]; Rohit K. Trivedi[1]; [1]Iowa State University, Ames Lab., USDOE, Ames, IA 50011 USA

Microstructure lengthscales determines the properties (mechanical, corrosion, heat-treatment, electro-magnetic, etc.) and thus the service life of a casting and directional solidification has been used to disclose the relationship of different lengthscales with the control parameters, e.g. alloy composition, temperature gradient and growth velocity. We have systematically studied the microstructure development during upward solidification of Al-Cu alloys with compositions

ranging from 4.0~40.0 wt%Cu. It was found both numerically and experimentally that liquid convection dominates the microstructure evolution in a sample of size >1.0mm diameter, which causes serious problems when one tries to correlate the microstructure dimensions with the control parameters. A new experimental technique has been developed where diffusion controls the microstructure formation. A thorough comparative study on the microstructure evolution of Al-Cu alloys will be presented over the above-mentioned composition range where the interface morphology can be cellular/dendritic or eutectic.

4:40 PM
Squeeze Casting of a Conventionally Wrought Aluminium Alloy: *Chris D.J. Manson-Whitton*[1]; Brian Cantor[1]; Keyna OíReilly[1]; Peter Schumacher[1]; [1]Oxford University, Dept. of Matls., Begbroke Business & Sci. Park, Sandy Ln., Yarnton, Oxfordshire OX5 1PF UK

High structural integrity aluminium castings can be achieved through controlled, refined microstructures, low porosity and low levels of inclusions. Mechanical properties can be further improved through the use of high strength alloys, such as those used in conventionally wrought applications. Traditional casting techniques preclude the use of such alloys, however squeeze casting offers the opportunity for controlled microstructures in wrought alloys direct from the melt. 7xxx series components have been cast using an experimental direct squeeze caster. Filling and pressurisation parameters were controlled in order to understand key aspects of the solidification regime, particularly the formation of macrosegregation in long freezing range alloys. As-cast microstructures of gravity and pressurised castings were characterised in terms of structural scale and segregation, and discussed with reference to measured solidification profiles.

5:05 PM
The Mechanics of Casting Condition and Quality of As-Cast Products Interrelationships During High-Pressure Die-Casting of Al-Si Alloys: *Reza Ghomashchi*[1]; [1]University of Quebec at Chicoutimi, Ctr. for Univ. Rsrch. on Aluminium-CURAL, 555 University Blvd., Chicoutimi, Quebec G7H 2B1 Canada

The effects of casting conditions, including fluid flow rate, casting and ingate thickness, and melt and die temperatures, were studied on the microstructure of LM24 high pressure die casting alloy using a fully-controlled cold chamber high-pressure die-casting machine coupled with an experimental die set of a rectangular die cavity shape of 65x130x(2, 4, 8) mm. All castings showed a bimodal distribution of dendrites in which the morphology of dendrites was completely different to the classic tree shape characteristic. It was found that the casting parameters influence the quality of the coupons surface finish, porosity size and percentage, volume fraction and size of dendrites, while have no effect on the silicon morphology and percentage of Al-Si eutectic.

Computational Phase Transformations: Solidification and Crystallization

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

Wednesday PM Room: 11B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Ralph E. Napolitano, Iowa State University, Ames Lab. Dept. of Matls. Sci. & Eng., Ames, IA 50011 USA; Alain Karma, Northeastern University, Ctr. for Interdisciplinary Rsrch. on Complex Sys., Boston, MA 02115 USA

2:00 PM Invited
Phase-Field Modeling of Dendritic Solidification with Coupled Heat and Solute Diffusion: Juan C. Ramirez[1]; *Christoph Beckermann*[1]; [1]University of Iowa, Dept. Mechl. Eng., 2412 SC, Iowa City, IA 52242 USA

A phase-field model for dendritic solidification of dilute alloys has been developed that accounts for the simultaneous diffusion of heat and solute, including unequal diffusivities. The model incorporates the thin-interface asymptotics developed by Karma and coworkers for the purely thermal and solutal cases, allowing calculations to be performed for coupled heat and solute transport using an interface thickness that is large compared to the capillary length and in the limit of vanishing interface kinetics. In addition, an extra term is included in the solute diffusion equation that eliminates the artifical solute trapping effect due to smearing of the interface. Simulations are performed for free dendritic growth into an undercooled melt of a dilute binary alloy. The effect of solute additions on the dendrite tip growth velocity, tip radius, and operating point selection are investigated.

2:30 PM Invited
Phase-Field Modeling of Alloy Directional Solidification: *Alain Karma*[1]; [1]Northeastern University, Ctr. for Interdisciplinary Rsrch. on Complex Sys., Boston, MA 02115 USA

Despite several decades of theoretical study of directional solidification since the pioneering analysis of Mullins and Sekerka on morphological stability, several fundamental aspects of the formation of cellular and dendritic array structures remain poorly understood. We have carried out a numerical study of these array structures that is based on a new phase-field formulation of the solidification of dilute binary alloys [A. Karma, Phys. Rev. Lett, {\bf 87}, 115701 (2001)]. This formulation makes it possible to simulate quantitatively microstructural evolution in the low velocity regime where the solid-liquid interface is close to thermodynamic equilibrium (i.e. with negligible interface kinetics and solute trapping). We present new results that shed light on the relative importance of thermodynamic noise, i.e. concentration fluctuations of microscopic origin, and the purely deterministic growth dynamics in the formation of secondary branches (sidebranching). The main new finding is the existence of a stable limit cycle for a range of large spacing of the array that can drive sidebranching even in the absence of noise. We also present a quantitative comparison of our results with available experimental data.

3:00 PM
Competitive Growth During Directional Solidification: A 3D Cellular Automaton Model: *Wei Wang*[1]; Peter D. Lee[1]; Malcolm McLean[1]; [1]Imperial College, Matls., Prince Consort Rd., London SW7 2BP UK

Grain selection plays an important role when producing directional solidified and single crystal Nickel-based superalloy turbine blades. Misorientated grains are normally overgrown by grains with their preferred growth direction, <100>, better aligned normal to the isotherms, producing a sharp texture in the final component. A 3D model that combines a Cellular Automaton description of grain growth with a Finite Difference solution of solute diffusion has been developed to simulate competitive growth. This model, which reproduces the full dendritic structure, including primary and secondary arms, was applied to simulate the competitive growth between both converging and diverging grains. The results show an excellent correlation to experiment, allowing an investigation of the overgrowth and branching mechanisms during both quasi-steady state and perturbed growth, leading to conditional overgrowth of misorientated grains.

3:20 PM
Effects of Multicomponent Diffusion and Thermodynamics on the Dendritic Structure in Ternary Fe-C-Mn: Hermann Josef Diepers[1]; Dexin Ma[1]; *Ingo Steinbach*[1]; [1]Access e.V., Intzestrasse 5, Aachen D-52072 Germany

An experimental and numerical study is performed to investigate the effect of multicomponent diffusion and thermodynamics on the dendritic structure of an Fe-Mn-C alloy. The directionally solidified dendritic structures are uncovered using the new technique of introducing artificial pores into the sample. This allows for the direct observation of the feritic structure even after peritectic transformation. The numerical approach uses a multicomponent phase-field model. The simulations are compared to the experimental results. Focus of the research is the importance of kinetics, thermodynamics and their interaction. In order to separate both effects ternary simulations with realistic diffusion and phase diagram data are compared to simulations with idealized quasibinary data. It is shown, that in general there is no quasibinary model available to describe both effects of diffusion and

thermodynamics in multicomponent systems. The differences in tip selection, microsegregation and primary spacing are quantified.

3:40 PM Break

3:55 PM Invited
The Three Dimensional Evolution of Topologically Complex Structures During Coarsening: I. Savin[1]; *P. W. Voorhees*[1]; [1]Northwestern University, Matls. Sci. & Eng., Cook Hall, 2225 N. Campus Dr., Evanston, IL 60208 USA

The coarsening process in systems consisting of spherical particles in a matrix has been studied extensively. In contrast, coarsening in systems that possess both positive and negative curvatures, such as those present following dendritic solidification, have received less study. A challenge in understanding the evolution of such microstructures is that the evolution must be studied in three dimensions. We have employed three dimensional phase field calculations to follow the evolution of a topologically complex dendritic microstructure during coarsening. We compare the results of experiments on the coarsening of dendritic microstructures with the calculations by using the experimentally measured microstructures as initial conditions in our calculations. The calculations show the important role of topological singularities in the evolution of dendritic structures during coarsening, particularly the pinching of liquid channels via a Rayleigh instability.

4:25 PM Invited
Phase Field Theory of Multi-Domain Solidification in Alloys: *L·szlÛ Gr·n·sy*[1]; Tam·s Pusztai[1]; Tam·s Bˆrzsˆnyi[1]; [1]Research Institute for Solid State Physics & Optics, PO Box 49, Budapest H-1525 Hungary

We present a phase field theory for describing the nucleation and growth of one and two-phase crystals solidifying with different crystallographic orientations in binary liquid alloys. The regular solution model is used to describe the thermodynamic properties. We investigate the kinetics of crystallization as a function of model parameters (anisotropy, symmetry, initial liquid composition) for primary, eutectic, and peritectic solidification under equiaxial conditions.

4:55 PM Invited
Prediction of Solidification Microstructures in Multi-Component Alloys Using Fixed Grid Methods and Thermodynamic Calculations: *Alain Jacot*[1]; Michel Rappaz[1]; [1]EPFL, Matls. Inst., MX-G, Lausanne CH-1015 Switzerland

The pseudo-front tracking method and the phase-field model are fixed grid numerical techniques which permit to describe diffusive phase transformations without having to explicitly track the interface. These methods have proved to provide accurate solutions to moving boundary problems. However, important issues are still to be addressed in order to apply such models to practical problems: the high computation time and the need to couple with thermodynamic databases to describe multi-component systems. The first part of this contribution will focus on the pseudo-front tracking method and its application to solidification in multi-component systems. Examples of calculations for various thermal and grain size conditions will be shown. Comparisons with phase field calculations will be presented with emphasis on the advantages and limitations of the two methods. In a second part, different strategies to couple microstructure models with thermodynamic databases will be presented. The issue of computation time will be discussed using simple estimators to show the influence of the solidification conditions and numerical parameters.

5:25 PM
Phase Field Modeling of Solidification with Liquid Convection and Solid Motion: *Adam C. Powell*[1]; Jorge Alberto Vieyra Salas[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139-4301 USA

A novel mixed-stress model permits coupling of phase field modeling of solidification with Navier-Stokes fluid dynamics in the liquid and linear elastic deformation in the solid. Stress is transitioned between Newtonian viscosity in the liquid and elasticity in the solid across the diffuse interface using an interpolation function of the phase variable. Solid stress is related to local solid strain, which is an additional tensor field in the model. The strain tensor and crystalline orientation are both rotated according to the local vorticity. Results are given for an incompressible fluid and solid with Poisson ratio of 1/2, using both Cahn-Hilliard and anisotropic Allen-Cahn phase field formulations in two dimensions, and extension to three dimensions is discussed.

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

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

Wednesday PM Room: 17A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Craig Hartley, Air Force Office of Scientific Research, Arlington, VA 22203-1977 USA; Vijay Vasudevan, University of Cincinnati, Dept. of Cheml. & Matls. Eng., Cincinnati, OH 45221 USA

2:00 PM Invited
High Temperature Mechanical Behavior of Cryomilled Al-Al_3Ti: *J. R. Weertman*[1]; B. Dehiya[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

Cryomilled Al-Al_3Ti has good high temperature strength and microstructural stability. Stress-strain curves over a range of temperatures show little or no strain-hardening. At high temperatures and fast strain rates a decided yield point is observed. The yield point disappears as the strain rate drops and serrated curves become evident. Holding a sample at elevated temperature before testing, with or without load, enhances the yield point. The yield point and serrations are simply modeled on the basis of initial low dislocation density, rapid generation and concurrent annihilation.

2:25 PM Invited
Effects of Dislocation-Crack Interactions on Fatigue Crack Growth: *K. Sadananda*[1]; Gregory Glinka[2]; Jerry Hsieh[3]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 20375 USA; [2]University of Waterloo, Dept. of Mechl. Eng., Waterloo, Ontario N2L 3G1 Canada; [3]Thomas Jefferson High School for Science & Technology, Fairfax Co. Board of Edu., 6560 Braddock Rd., Springfield, VA USA

Fatigue crack growth occurs due to irreversibility of plastic flow. Plastic flow occurs by the nucleation and movement of dislocations. Hence dislocation-crack interactions play a significant role in the mechanics of fatigue crack growth. It is well known that crack-tip plasticity contributes to fatigue crack growth via plastic-blunting process. However, there are several questions regarding the role of plasticity in the wake of a growing crack. Many theories and models have been developed based on the notion that plasticity behind the crack tip contributes to premature crack closure thereby retarding crack growth. This concept of plasticity induced crack closure is reexamined using dislocation-crack interactions. The results and their implications will be discussed in relation to fatigue crack growth kinetics.

2:50 PM
On the Mechanism of Mechanical Deformation Twin Thickening in Titanium: *Steven Celotto*[1]; Gareth G.E. Seward[2]; Robert C. Pond[1]; [1]University of Liverpool, Dept. of Eng., Matls. Sci. & Eng., Brownlow Hill, Liverpool L69 3BX UK; [2]University of Liverpool, Dept. of Earth Scis., Liverpool L69 3GP UK

In this study we have investigated the mechanism of deformation twinning in commercially pure titanium by experimental and theoretical means. Scanning electron microscopy has been used to determine the morphology of twins in lightly deformed specimens. Lateral boundaries of the twins often exhibit irregular shapes rather than lenticular form. The crystallography of individual twins has been established using electron back-scattered diffraction. In addition, the magnitude and direction of the shear have been found by measuring the distortions induced on a fiducial grid scribed on the initial specimen surface using a focussed laser beam. It was observed that in the vicinity of irregular twin boundaries, reorientation of the fiducial lines only occurred where they intersected the interface. These observations confirm that deformation has only occurred within the twins, and no emissary slip process as described by Sleeswyk has taken place near

irregular lateral boundaries. Using atomic scale modelling, such irreg ular deformation can be explained in terms of localised twinning dislocation dipoles that are activated by the effect of applied stresses on stress concentrations near sessile interfacial defects.

3:10 PM
DHC Velocity and KIH of Zr-2.5Nb Tubes with Hydrogen Concentration: *Young Suk Kim*[1]; [1]Korea Atomic Energy Research Institute, Zirconium Grp., 150, Dukjin-dong, Yusong, Daejon 305-353 Korea

This study focuses on the elucidation of delayed hydride cracking (DHC) of zirconium alloys, which has not been clearly understood up to date. DHC tests were conducted on the compact tension (CT) specimens of a CANDU Zr-2.5Nb pressure tube with hydrogen concentration varying from 12 to 100 ppm. Hydrogen was charged electrolytically into the CT specimens followed by a homogenization treatment. The DHC velocity of the Zr-2.5Nb pressure tube had a temperature dependence with an activation energy of 49 KJ/mol in a temperature range from 100 to 300°C, and increased linearly to a constant with increasing hydrogen concentration. In contrast, the threshold stress intensity factor, KIH in the axial direction of Zr-2.5Nb tube had a drastic decrease of KIH to a constant with increasing hydrogen concentration. Thus, DHCV and KIH were nicely described as a function of the supersaturated hydrogen concentration over the terminal solid solubility for dissolution (TSSD) independent of temperatures. Based on these results , we propose that KIH is a critical stress intensity factor to initiate the nucleation of hydrides at the crack tip region, driving the crack tip with the supersaturated hydrogen in solution to reach the TSSD at any temperatures. Therefore, we conclude that the gradient of the equilibrium hydrogen concentration or TSSD at the crack tip region and the supersaturated one at the matrix region is a governing factor to initiate DHC. Normalization of the striation spacing by hydrogen diffusivity and hydrogen solubility demonstrates that the striation spacing decreases with increasing temperature. Therefore, the striation spacing, inversely proportional to DHCV, is a good parameter representing the DHC velocity of the Zr-2.5Nb tubes.

3:30 PM Break

3:50 PM Invited
Creep Anisotropy and Radiation Effects in Zr-Alloy Tubing: Application to In-Reactor Dimensional Prediction: *K. L. Murty*[1]; [1]North Carolina State University, 2500 Stinson Dr., PO Box 7909, Raleigh, NC 27695-7909 USA

Zirconium alloyed mainly with Sn and Fe known as Zircaloys commonly used in reactors are highly textured and exhibit anisotropic physical and mechanical properties. We summarize here the crystallographic textures commonly noted in Zircaloy cladding that lead to mechanical anisotropy and the effects of stress-relief and recrystallization anneals following tube reduction processes. Anisotropic creep behaviors were characterized through biaxial creep tests and creep loci. Quantitative description of the crystal texture through CODF combined with prism slip dominance predicted the creep locus of the recrystallized tubing. Deviations exhibited by the stress-relieved cladding were seen to stem from grain shape anisotropy. Neutron radiation exposure of the stress-relieved tubing resulted in radiation creep and stress-free radiation growth. The texture-creep model combined with radiation effects enabled us to predict the axial position dependence of the diametral decrease. The paper concludes with challenges posed by the new advanced cladding alloys with composition gradients as well as Nb-additions and surface modifications.

4:15 PM
Oxide Formation on the Creep Cavities of Type 316L Stainless Steel: *Yongbok Lee*[1]; Jinsung Jang[2]; Woo-Seog Ryu[2]; Dokyol Lee[1]; [1]Korea University, Dept. of Matl. Sci. Eng., Seoul 136-353 Korea; [2]KAERI, Nucl. Matls. Tech. Rsrch. Team, Daejeon 305-600 Korea

Creep failure of austenitic stainless steels is usually occurred through the nucleation and growth of cavities. Solution treated 316 L stainless steel was creep tested at 620 C and 220, 240 and 260 MPa in vacuum as well as in atmosphere. Creep rupture life, creep elongation and steady state creep rate in vacuum were better than those in atmosphere. Creep constants from the normalized creep curves were estimated as 2.55 and 2.89 in air and in vacuum, respectively. Oxide film, $(Fe_{0.8},Cr_{0.2})$ O, was found on the inner surface of creep cavities of type 316 L stainless steel and the change of surface energy of the cavity wall is expected to affect the nucleation or growth process. Crystal structure and the lattice parameter of the oxide film were investigated using TEM (JEM 2000FX) with EDS. Oxide film was FCC structured polycrystal and the lattice parameter of the oxide was about 4.056 angstrom.

4:35 PM Invited
Improvement of Creep-Fatigue Resistance Modifying the Carbide Interfacial Energy in Austenitic Stainless Steels: *Soo Woo Nam*[1]; Kyung Seon Min[1]; Ki Jae Kim[1]; Hyun Uk Hong[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701 Korea

Creep-fatigue life is reduced by cavitation at grain boundary carbides at high temperature in austenitic stainless steels. Because the interfacial energy between carbides and neighboring matrix can affect creep-fatigue life, it is suggested that interfacial energy can be decreased by modification of carbide and grain boundary characteristics. In AISI 304 and 316 stainless steels, it is found that the interfacial energy can be remarkably reduced through grain boundary serration to extend the creep-fatigue life. Therefore, the mechanism of the grain boundary serration is investigated. It is expected that planar carbides on serrated grain boundaries have a lower interfacial energy than that of triangular carbides on straight grain boundaries. The different influence of TiC and $Cr_{23}C_6$ on creep-fatigue life in AISI 321 stainless steel with the same carbide density at the grain boundary is based on the stronger cavitation resistance of TiC. It is verified that formation and growth of grain boundary cavities in TiC aged alloy are more difficult than those in $Cr_{23}C_6$ aged alloy.

5:00 PM
Defect Structures in the Massive γ_m Phase in a Quenched Ti-46.5 At.% Al Alloy: Ping Wang[2]; Mukul Kumar[3]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA; [3]Lawrence Livermore National Laboratory, 7000 East Ave., L-356, Livermore, CA 94550 USA

The defect structures in the massively formed gamma (γ_M) grains in a Ti-46.5 at.% Al alloy, rapidly quenched from the high-temperature α-phase field, have been studied by conventional and high-resolution TEM. Defect structures composed of dislocations, stacking faults (SFs) and antiphase boundaries (APBs) intimately associated with dislocations or stacking faults were observed. Analysis indicates that both 1/2<110] and 1/2<101] unit dislocations were present in the gm phase, with the latter linked by highly curved APBs. Comparison of experimental and computer simulated TEM images established that wide SFs, which are created by the dissociations of1/2<101> unit dislocations, lie on {111} planes and are bound by b = 1/6<121> Shockley partial dislocations of all possible types. In addition, APBs are found to commence or terminate on the SFs at the partial dislocations with b = 1/6<121], but not those with b = 1/6<112]. Confirmation for both the latter, as well as the intrinsic nature of the stacking faults was obtained using atomic models and analysis of the HREM images. Based on the observations and subsequent analyses, a model for the formation of these defects and defect configurations is proposed. The authors are grateful for support of this research by the National Science Foundation under grants DMR-9224473 and 9731349, Dr. Bruce MacDonald, Program Monitor.

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

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

Wednesday PM Room: 16B
March 5, 2003 Location: San Diego Convention Center

Session Chair: Hans Conrad, North Carolina State University, Matls. Sci. & Eng., Raleigh, NC 27695-7907 USA

2:00 PM
Plastic Deformation Behavior of A356/357 Aluminum Cast Alloys: *Qigui Wang*[1]; [1]General Motors Corporation, CDVC-Powertrain, 1629 N. Washington Ave., Saginaw, MI 48605 USA

Strain hardening rate of A356/357 aluminum cast alloys during plastic deformation depends on eutectic particle size and morphology, secondary aluminum dendrite arm spacing (SDAS), and Mg contents. At low plastic strain, the eutectic particle aspect ratio and matrix strength dominate the work hardening, while at larger plastic strains the hardening rate depends on the secondary dendrite arm spacing (SDAS). For all materials, the average internal stresses increase very rapidly at small plastic strains gradually saturating at large plastic strains. Elongated eutectic particles, small SDAS or higher Mg content result in a higher saturation value. The difference in the internal stresses, due to different microstructural features, determines the rate of eutectic particle cracking and in turn the tensile instability of the materials. The fracture strain of alloys A356/357 corresponds to the critical fraction of cracked eutectic particles locally or globally, irrespective of the fineness of the microstructure.

2:20 PM
NEMD Simulations of Metallic Friction: *James E. Hammerberg*[1]; Brad L. Holian[2]; Timothy C. Germann[1]; Ramon J. Ravelo[1]; [1]Los Alamos National Laboratory, Appl. Physics Div., MS D413, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Theoretl. Div., MS B268, Los Alamos, NM 87545 USA

With the advent of large-scale parallel computers in the past decade it has become possible to study the microscopic physics of complex nonlinear many-body systems at scales approaching the mesoscale. The increase in computational power has made possible the study of very large systems, of order 10^7-10^8 atoms. This has, in turn, made possible the study of dislocations, nano-scale grain dynamics and nucleation, and non-equilibrium plastic flow in unprecedented detail. One of the areas where non-equilibrium deformation arises is that of dry sliding friction between ductile metals. We discuss the results of atomistic simulation studies of sliding friction in the velocity range 10-10^3 m/sec under a variety of loading conditions from 10^{-1}-10^3 GPa for a variety of tribo-pairs,including Cu/Cu, Cu/Ag, and Ta/Al,as well as model systems such as Lennard-Jones. The effective tangential force as a function of sliding velocity exhibits generic behavior at high velocities determined by nano-scale structural transformation, and in certain cases material mixing, at high strain and strain rates. The Non-Equilibrium Molecular Dynamics (NEMD) techniques used in these large-scale simulations will be discussed and their connections to the multi length- and time-scale theory of sliding friction will be presented.

2:40 PM
Three-Dimensional Dislocation Dynamics Simulations of the Interaction Between Dislocations and Low-Angle Grain Boundaries: *Tariq A. Khraishi*[1]; Yu-Lin Shen[1]; [1]University of New Mexico, Dept. of Mechl. Eng., Albuquerque, NM 87131 USA

Parametric simulation studies of dislocations interacting with low-angle grain boundaries were carried out using a discrete 3D Dislocation Dynamics code. The tilt boundaries, consisting of dislocation arrays and assumed to be fixed in space, are mainly characterized by the tilt angle which in turn controls the separation distance between dislocations in the wall (i.e., grain boundary). Dislocation sources were placed in between two walls representing the boundaries of a single grain. The simulations revealed an interesting sequence of events which depended primarily on the length scales of the problem: the separation distance between the dislocations in the wall, and the separation distance between the walls themselves, representing the grain size. In addition, the simulations illustrated the arrest of propagating dislocations into the strain field of the stationary wall dislocations. Implications on the strengthening effect and relevant micromechanical issues are also discussed.

3:00 PM
Dynamic Recrystallization: Plasticity Enhancing Structural Development Induced by Strain: *H. J. McQueen*[1]; [1]Concordia University, Mech. Industl. Eng., Montreal H3G 1M8 Canada

Dislocation mobility upsurge usually causes significant improvement in the plasticity of crystalline solids, partly through its ability to deter crack formation. Dynamic recrystallization DRX that proceeds during deformation reduces the flow stress and markedly raises the ductility, which characterize the hot working range. While it facilitates straining, its nucleation rate and equilibrium grain size are defined by the temperature and strain rate. Its operation is thermally activated like other deformation assisting mechanisms such as source unpinning, cross slip and climb; these last two reduce the strain hardening in combination with annihilation and polygonization of substructure that constitute dynamic recovery DRV. The flow stress in steady state is not directly defined by the grain size but rather by the average dislocation density which is tied to both DRV and DRX in so far as the migration of GB reduce the dislocation density below that attained by DRV alone. In hexagonal metals, stress concentrations at grain boundaries lead to a mantle of fine recrystallized grains in which the strain becomes concentrated while the old grain cores contribute little. Dynamic recrystallization does not in its operation produce any strain. It is a dynamic restoration process which does facilitate straining with respect to ease and extent. In distinction from its static conterpart its progress and its effect are intimately associated with the plastic deformations.

3:20 PM
Mechanisms of Deformation in Nanostructured Metals: *Robert John Asaro*[1]; [1]University of California-San Diego, Structl. Eng., R0085, La Jolla, CA 92093 USA

This talk will be concerned with establishing the mechanisms of deformation and strengthening in metals and alloys with nano-sized grains. Specifically, the role of dislocation motion both within and normal to slip planes will be developed within the context of a full constitutive theory and used, via numerical simulation, to describe the formation of grain boundary, and interface, ìboundary layersî that impart high strength and strain hardening. The role of grain boundary sliding and grain rotations will also be explored. Experimental evidence will also be provided to verify the concepts developed.

3:40 PM Break

3:50 PM
Quantitative Extraction of High Explosive Drive and Material Constitutive Parameters from Velocity Histories: *Wayne E. King*[1]; I. Harold Zimmerman[2]; Desmond Pilkington[1]; Oliver T. Strand[1]; Michael W. Danforth[3]; [1]University of California, Lawrence Livermore Natl. Lab., Chem. & Matls. Sci. Direct., L-356, Livermore, CA 94551 USA; [2]University of California, Lawrence Livermore Natl. Lab., Defense & Nucl. Tech. Direct., L-095, Livermore, CA 94551 USA; [3]University of California, Lawrence Livermore Natl. Lab., Defense Technologies Eng. Div., Livermore, CA 94551 USA

Fabry-Perot velocimetry is widely used, often in combination with hydrocode simulations, to study the physical processes underlying shock effects in materials. However, matching experiment with simulation is to some extent a qualitative undertaking. In this work, we employ non-linear least squares methods to develop a quantitative match between the velocity histories of two high-explosive-driven flat-plate experiments and predictions of 1 and 2-dimensional hydrocode simulations. Results extracted from the optimization include the Jones-Wilkins-Lee (JWL) equation of state parameters for the high-explosive drive and constitutive parameters describing the behavior of the flat-plates. This work performed under the auspices of US Department of Energy by University of California, Lawrence Livermore National Laboratory under contract no. W-7405-Eng.-48.

4:10 PM
Comparative Grain Size Influences in Dynamic Deformation and Creep: *Frank R.N. Nabarro*[1]; [1]University of the Witswatersrand, Condensed Matter Physics Rsrch. Unit, Private Bag 3, Johannesburg WITS 2050 S. Africa

Further consideration is given, as far as the polycrystal grain size may influence the material deformation rate, to whether the applied stress: (i) is first greater than the material Peierls stress; (ii) then to whether the the grain size is larger than the Taylor dislocation lattice spacing corresponding to the Peierls stress; (iii)followed by whether the product of stress and grain size exceeds the value for operation of modeled dislocation sources; and, lastly, (iv) to whether the product of stress and square root of grain size exceeds the Hall-Petch value for propagation of slip across grain boundaries {see Soviet Physics - Solid State Physics, Vol. 42, No. 8, pp. 1417-1419 (2000)}.†

4:30 PM
Effect of Low-Temperature Shock Compression on the Microstructure and Strength of Copper: *David H. Lassila*[1]; Tien Shen[1]; Marc A. Meyers[2]; [1]Lawrence Livermore National Laboratory, Livermore, CA 94551 USA; [2]University of California-San Diego, La Jolla, CA 92093 USA

Copper with two purities (99.8 and 99.995%) was subjected to shock compression from an initial temperature of 90 K. Shock compression was carried out by an explosively accelerated flyer plates at velocities generating pressures between 27 and 77 GPa. The residual microstructure evolved from dislocation cells to mechanical twins and, at the 57 and 77 GPa pressures, to complete recrystallization with

a grain size larger than the initial one. The shock-compressed copper was mechanically tested in compression at 10(superscript: -3) s(superscript: -1) at 300 K; the conditions subjected to lower pressures (27 and 30 GPa) exhibited work softening, in contrast to the conventional work hardening response. This work softening is due to the breakdown of the unstable shock deformation microstructure consisting high density of uniformly distributed dislocations and the formation of larger cells, with a size of approximately 1m. The 99.995% copper subjected to the higher shock compression pressures (57 and 77 GPa) exhibited a stress-strain response almost identical to the unshocked condition. This indicates that the residual temperature rise was sufficient to completely recrystallize the structure and eliminate the hardening due to shock compression. Thermodynamic calculations using the Hugoniot-Rankine conservation equations predict residual temperatures of 357 and 560 K for the 57 and 77 GPa peak pressures, respectively. The complete recrystallization is evidence for a higher residual temperature and suggests that a plastic deformation term should be added to the release isentrope.

4:50 PM

TiC-NiTi Cermets: Structure and Ballistic Performance: *Eugene A. Olevsky*[1]; Elizabeth Strutt[2]; Marc A. Meyers[1]; Werner Goldsmith[3]; David J. Benson[2]; [1]San Diego State University, 5500 Campanile Dr., San Diego, CA 92182-1323 USA; [2]University of California-San Diego, Dept. of MAE, La Jolla, CA 92093-0411 USA; [3]University of California-Berkeley, Dept. of Mechl. Eng., Berkeley, CA 94705 USA

TiC-NiTi cermets were produced by a new process consisting of two stages: combustion synthesis followed by quasi-isostatic pressing (QIP) using a granular pressure transmitting medium. The structure of the cermet consists of spheroidal TiC particles surrounded by a NiTi matrix, that has the thermoelastic properties. The material was charcterized by transmission electron microscopy. Ballistic testing was carried out in the DOP(depth of pentration) configuration. Steel specimens were impacted at the same velocities as cermet specimens and the ballistic efficiency was estimted; it was found to vary between 6 and 8, in comparison to hot rolled AISI 1018 steel. The experimental ballistic results are successfully compared with computed predictions. Research funded by the US Army Research Office MURI Program.

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Chaotic and Power Law States in the Portevin-Le Chatelier Effect: Madurai Srinivasan Bharathi[1]; Garani Ananthakrishna[1]; [1]Indian Institute of Science, Matls. Rsrch. Ctr., Bangalore, Karnataka 560012 India

Recent studies on the PLC effect report an intriguing crossover phenomenon from a low dimensional chaotic to an infinite dimensional power law regime in experiments on Cu-Al single crystals and Al-Mg polycrystals, as a function of strain rate. We devise a fully dynamical model which reproduces these results. At low and medium strain rates, the model is chaotic with the structure of the attractor resembling the reconstructed experimental attractor. At high strain rates, power law statistics for the magnitudes and durations of the stress drops emerge as in experiments. The spectrum of Lyapunov exponents changes from a set of positive, few zero and negative exponents in the chaotic regime to a power law distribution of null exponents in the power law state. These results can be made more transparent by investigating the spatial configuration of the dislocations on the slow manifold. This shows that while a large proportion of dislocations are pinned in the chaotic regime, most of them are pushed to the threshold of unpinning in the scaling regime, thus providing insight into the mechanism of crossover.

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Nonuniform Motion of Supersonic Dislocations: *Xanthippi Markenscoff*[1]; Luqun Ni[1]; [1]University of California, Mechl. & Aeros. Eng., 9500 Gilman Dr., La Jolla, CA 92093 USA

A nonuniform supersonic motion of a dislocation, i.e. with variable speed, is analyzed. In this case,the Mach wave fronts are not straight lines as in the uniform case, but curved. Comparisons will be made with the numerical simulation results of Gumbsch and Gao(1999). The dislocation core will be assumed to be either a step function discontinuity (Volterra dislocation), or ramp-like. The energetics will be also discussed.

Friction Stir Welding and Processing II: Friction Stir Joining: Mechanical Properties

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee

Program Organizers: Kumar V. Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Murray W. Mahoney, Rockwell Science Center, Thousand Oaks, CA 91360 USA; Thomas J. Lienert, University of South Carolina, Mechanical Engineering Department, Columbia, SC 29208 USA; Rajiv S. Mishra, University of Missouri-Rolla, Metallurgical Engineering, Rolla, MO 65409-0340 USA

Wednesday PM — Room: 7B
March 5, 2003 — Location: San Diego Convention Center

Session Chair: Maria Posada, Naval Surface Warfare Center, Carderock Div., W. Bethesda, MD 20817 USA

2:00 PM

Process Parameters-Mechanical Properties Relationship in Self-Reacting Friction Stir Welds of Aluminum Alloys: *Z. X. Li*[1]; G. P. Adams[1]; [1]Lockheed Martin Space Systems Company, Michoud Ops., PO Box 29304, New Orleans, LA 70189 USA

Self-reacting friction stir welding process (SR-FSW) is a relative new technology that is derived from conventional friction stir welding (FSW) process. A number of advantages including elimination of backing anvil and significant lower process loads have made the self-reacting FSW process a competitive technology as compared with the conventional FSW process. In present work, butt joint configurations for joining plates in thickness ranges from 0.320" to 1.000" were friction stir welded using a self-reacting pin tool system. Various pin tool designs and process parameters were applied to understand the effect on the mechanical properties, microstructure, and defect formation of the self-reacting welds. Mechanical properties and microstructure of the resulting welds as a function of pin tool designs and process parameters will be highlighted.

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Welding Tool and Process Parameter Effects in Friction Stir Welding of Aluminum: *Kevin J. Colligan*[1]; James J. Fisher[2]; Joseph R. Pickens[3]; [1]Concurrent Technologies Corporation, 27980 Kim Dr., Harvest, AL 35749 USA; [2]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA; [3]Concurrent Technologies Corporation, 13815 Kennard Dr., Glenelg, MD 21737 USA

A unique style of welding tool, based on concepts originated by The Welding Institute (TWI, Cambridge, U.K.), was used in this study to make welds in 25.4-mm 5083-H131 aluminum. A variation of the TWI tool was used, employing a number of flats cut into the pin surface and much finer threads. It was hypothesized that the material flow produced by this pin design was based upon the capture of material by the flats, followed by deposition behind the pin. If this were the case, an important parameter governing the behavior of the pin would be the travel per flat per revolution, a measure of the amount of material captured by each flat on each revolution. Prelminary welding trials were performed to study the characteristics of this pin design in terms of power required, specific energy, torque, weld forces and macrostructure produced.

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Texture Development in Friction Stir Welded 2519 Aluminum: *John F. Bingert*[1]; Richard W. Fonda[2]; [1]Los Alamos National Laboratory, on temporary assignment to Naval Research Laboratory, Matls. Sci. & Tech., Code 6352, 4555 Overlook Ave. SW, Washington, DC 20375 USA; [2]Naval Research Laboratory, Matls. Sci. & Tech., Code 6324, 4555 Overlook Ave. SW, Washington, DC 20375 USA

Friction stir welding produces severe thermomechanical transients that generate crystallographic texture evolution throughout the weld-affected microstructure. In this study, a friction stir weld in a coarse-grained 2519 aluminum plate was investigated in order to resolve the influence of these thermal and deformation effects on texture development. Automated electron backscatter diffraction (EBSD) was applied to spatially resolve orientations in the base metal, weld nugget, and thermomechanical and heat-affected zones. Results show a gradient demarcated by an alteration in boundary character, texture, and precipitate distribution. EBSD scans and microstructural characterizations reveal substructure evolution from the base plate to the nugget indicative of dynamic recovery and recrystallization processes. Comparison of experimental results to those from polycrystal plasticity

modeling suggests that texture evolution did not follow that predicted from simple-shear processing assuming restricted glide. Consequences of rigid-body spin and elevated-temperature shear deformation will be explored in relation to their influence on the observed texture.

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Property Characterization of 2024Al/7075Al Bi-Alloy Friction Stir Welded Joints: *John A. Baumann*[1]; Richard J. Lederich[1]; David R. Bolser[1]; Rajesh Talwar[1]; [1]The Boeing Company, Adv. Mfg. R&D, PO Box 516, MC S245-1003, St. Louis, MO 63166-0516 USA

Direct joining of damage tolerant 2XXX alloys with high strength 7XXX alloys offers the promise of fabricating tailor-made structural components. Butt joints in 1" thick plates of 7075-T7351 and 2024-T351 were produced by friction stir welding. Joints were sound and free of voids and root surface disbonds. The joints have a joint efficiency (Fty) that is 80% of the parent metal 2024-T351. Properties are consistent as functions of depth and position along the length of the weld. Tensile failures are always ductile; elongations are typically 5%, which is a consequence of the deformation being localized to the lower strength heat affected zones. The fatigue response (Kt=1.5 and R=0.05) of these weldments was determined in the weld nugget, both heat affected zones, and parent material. The lifetimes were shortest in the heat affected zones, but were at least 1/3 of the parent metal lifetimes.

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Local Properties and Strain Distribution in Conventional and Self-Reacting Friction Stir Welds: *Anthony Peter Reynolds*[1]; Wei Tang[1]; [1]University of South Carolina, Dept. of Mechl. Eng., 300 Main St., Rm. A224, Columbia, SC 29208 USA

In previous work, the authors and others have demonstrated the ability to obtain the local constitutive properties from various regions of friction stir welded joints (e.g the nugget, TMAZ, and HAZ) by combination of a full-field strain measurement technique, a single transverse tensile test, and an assumption of iso-stress loading through the weld. The authors have applied this technique primarily to conventional FSW joints. In this paper, the local strain and property distributions in aluminum alloy friction stir welds made by conventional FSW and by use of a self-reacting or bobbin tool will be compared. The effects of the differing joint geometries will be examined for welds made by the two techniques, but with otherwise equivalent (in so far as is possible) welding conditions. Also, the local properties and strain distributions in transversely loaded joints made using welding conditions enabled by the self-reacting technology will be examined.

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Effect of FSW Parameters on the Structure and Superplastic Formability of AA 2095 SPF Sheet: *Hanadi G. Salem*[1]; Anthony P. Reynolds[2]; Jed S. Lyons[2]; [1]American University in Cairo, Dept. of Mechl. Eng., 113 kasr El Aini St., PO Box 2511, Cairo 11511 Egypt; [2]University of South Carolina, Dept. of Mechl. Eng., Columbia, SC 29208 USA

Joining of aluminum alloys with initial fine grain structures using conventional welding processes results in the deterioration of the properties due to grain growth. Friction stir welding (FSW) has proven its capability of producing sound welds almost defect free; in addition to refinement of the grain structure within the weld zone. FSW was employed to join dynamically recrystallized sheets of Al-Cu-Li base alloy with initial grain size of 2 mm. Microstructural, grain boundary misorientations and texture evolution of the welded sheets were investigated using optical, transmission and orientation image microcopy. The superplastic behavior of the weld nugget in the longitudinal direction was characterized. FSW of the dynamically recrystallized structure was successfully conducted with almost no grain growth throughout the weld. Hardness profiles indicated that hardness variation primarily depend on the particle size and distribution within the weld nugget. Uniform superplastic deformation greater than 400% was retained after FSW.

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Microstructural and Mechanical Evaluation of HSLA-65 and DH-36 FSW: *Maria Posada*[1]; John J. DeLoach[1]; Anthony P. Reynolds[2]; [1]Naval Surface Warfare Center, Carderock Div., Welding & NDE Branch, 9500 MacArthur Blvd., W. Bethesda, MD 20817 USA; [2]University of South Carolina, Mechl. Eng. Dept., 300 Main St., Columbia, SC 29208 USA

Double-pass friction stir welds were produced from Ω-inch thick HSLA-65 and DH-36. The microstructure of the weld nugget was comprised of four regions that include the stir zone, the swirl region within the stir zone, the thermal-mechanically affected zone (TMAZ), and the heat-affected zone (HAZ). Typical hardness plots of DH-36 showed an increase in hardness from the base metal through the HAZ /TMAZ into the stir-zone. The peak hardness corresponded to the swirl region located on the advancing side of the nugget. The hardness profile for HSLA-65 differed dramatically from typical steels evaluated to date. In fact, the hardness profile of HSLA-65 softened in the HAZ/TMAZ region and the peak hardness within the stir zone region was only slightly higher than its base hardness. The peak hardness was 3 times greater in DH-36 than in HSLA-65. HSLA-65 demonstrated greater impact toughness than DH-36. Longitudinal tensile tests were performed on both materials.

5:05 PM
Microstructural Characterization of FSW 7050 Al Alloys: S. R. Sharma[1]; *R. S. Mishra*[1]; J. A. Baumann[2]; R. J. Lederich[2]; R. Talwar[2]; [1]University of Missouri, Dept. of Metallurgl. Eng., 218 McNutt Hall, Rolla, MO 65409 USA; [2]The Boeing Company, Adv. Mfg. R&D, PO Box 516, MC S245-1003, St. Louis, MO 63166-0516 USA

Microstructure of a thick plate FSW 7050Al was characterized to address the issues of strength and ductility variations. The variation of microstructure at various locations in as-FSW and after heat treatment conditions will be presented. The post-FSW heat treatment leads to recovery of strength, but results in ductility problems. The variation of strength and ductility is linked with microstructural changes during heat treatment. The FSW nugget region shows abnormal grain growth. The origin of abnormal grain growth will be discussed.

General Abstracts: Fatigue, Fracture, and Plastic Deformation

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Wednesday PM Room: 19
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Ellen Cerreta, Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA; Brian Cockeram, Bechtel Bettis Laboratory, ZAP 08D/MT, W. Mifflin, PA 15122-0079 USA

2:00 PM
Correlating Microporosity with Mechanical Properties in Die-Cast AM50 and AM60 Magnesium Alloys: *Gurjeev Chadha*[1]; John E. Allison[2]; J. Wayne Jones[1]; [1]University of Michigan, Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; [2]Ford Motor Company, Matls. Sci. Dept., Dearborn, MI 48124 USA

The role of porosity on the mechanical behavior of die-cast AM50 and AM60 magnesium alloy components has been investigated with special emphasis on ductility. Metallographic and fractographic studies have been conducted on samples with varying volume fractions of porosity to determine the nature of damage initiation during tensile deformation. Surface strain mapping has been performed to determine strain concentrations and subsequent failure in these alloys as a function of porosity size, volume fraction and pore spatial distribution. The results of these studies will be described in this presentation.

2:20 PM
Room Temperature Fatigue and 1000∞C-1400∞C Compression Response of a Mo-2Si-1B Alloy: *Amruthavalli P. Alur*[1]; Sharvan Kumar[1]; [1]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA

The ambient temperature fatigue response of a Mo-2wt%Si-1wt.%B alloy is compared with that of a powder-processed TZM alloy (MT-104). Both, S-N curves and da/dN-DK behavior were assessed; the results demonstrate that the Mo-Si-B alloy is superior in fatigue to TZM. Fracture in the TZM alloy primarily initiates at ZrO2 particles that often result from powder processing. In both materials, fracture is a mixture of transgranular cleavage and intergranular failure. Compression tests were conducted at elevated temperatures (1000∞C-1400∞C) over a range of strain rates (10-4 to 10-7 s-1) and the variation of flow stress with strain rate was determined. Isothermal oxidation studies were conducted in the temperature range 300∞C to 1200∞C and weight gain/loss was measured. Microstructural evolution following high temperature deformation was studied in a transmission electron microscope. Mechanical property results and microstructural observations will be presented and implications will be discussed.

2:40 PM
The Fracture Toughness, Flexural Strength and Shear Strength of Monolithic Silicon Carbide (SiC) Produced by Chemical Vapor Deposition and SiC/SiC Joints Fabricated by a Molybdenum Diffusion Bonding Technique: *Brian V. Cockeram*[1]; [1]Bechtel Bettis Laboratory, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122 USA

The production of robust SiC to SiC joints is required to enable the fabrication of larger and more complicated SiC-based structures. One new technique involves the use of molybdenum foils to develop a diffusion bond with SiC during a simple thermal treatment. In this work fracture strength data for SiC joined using a molybdenum foil diffusion bond is compared with results for monolithic Chemical Vapor Deposited (CVD) SiC. Flexure results are obtained using a standard 4-point flexural test method (ASTM C1161) from room-temperature to 1500C. Shear strength results obtained using a double-notched specimen are also compared for the joined and monolithic specimens. As has been observed for other ceramic materials, the shear strength values for the monolithic and joined specimens were lower than the flexural strength data. Fracture toughness values for monolithic CVD SiC are found to be independent of the CVD growth direction and test temperature from ambient to 1100C. Differences in elastic properties and coefficient of thermal expansion between SiC and phases that are produced in the molybdenum foil bond region result in the formation of slightly larger flaws in the SiC near the joint region. These flaws are shown to produce the fracture initiation sites in both the flexure and shear testing of molybdenum joined SiC. Given the fracture toughness of monolithic CVD SiC is inherently low (about 3.4 MPa m^1/2), the slight increase in flaw size can explain the slightly lower flexural strength values observed for molybdenum bonded SiC. However, the shear strength values for the molybdenum-joined SiC are found to be within the data scatter of the monolithic material.

3:00 PM
Structure and Mechanical Properties of FeAl-WC Prepared by Combustion Synthesis: *Akihiro Matsumoto*[1]; Keizo Kobayashi[1]; Toshiyuki Nishio[1]; Kimihiro Ozaki[1]; [1]National Institute of Advanced Industrial Science and Technology, Inst. for Structl. & Eng. Matls., 2266-98 Anagahora, Shimoshidami, Moriyama-ku, Nagoya, Aichi 463-8560 Japan

FeAl-WC composites have been synthesized by combustion synthesis. Effects of particle size on the structure and mechanical properties of the products were investigated. Pure iron, aluminum and tungsten carbide powders were blended using an automatic agitator and consolidated using pulse current sintering process. As the particle size of iron becomes finer, the structure of products becomes homogeneous. The obtained products consisted of FeAl intermetallic compound and WC. FeAl-50vol%WC has a hardness 800Hv and a transverse rupture strength 2.0GPa. Moreover, wear resistance of FeAl-WC were investigated.

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Microbands and Microtwins Associated with Impact Craters in Copper and Brass Targets: Role of Stacking-Fault Energy (SFE): *Benjamin Gonzalez*[1]; Lawrence Eugene Murr[1]; Olga L. Valerio[1]; Hugo Lopez[1]; Erika Esquivel[1]; [1]University of Texas-El Paso, Dept of Metallurgl. & Matls. Eng., 500 W. University Ave., El Paso, TX 79968 USA

Deformation microbands coincident with {111} slip planes are observed to be associated with impact craters in large-grain (0.8mm) copper (sfe~80 mJ/m2) while microtwins coincident with{111} slip planes are observed in large-grain (~1.1 mm) brass (70 Cu-30 Zn; SFE~10 mJ/ m2). Taken together with other impact crater studies the evidence for a strong influence of SFE in the microband-microtwin transition seems compelling.

3:50 PM
Evaluation of Interfacial Adhesion of Hygrothermally Conditioned Carbon Fibre/Epoxy Composites by Tensile Tests: *B. C. Ray*[1]; [1]National Institute of Technology, Dept. of Metallurgl. & Matls. Eng., Rourkela-769008 India

The mechanical behaviour of interface is an important consideration in the assessment of polymeric composites. The magnitude of the stress transfer from the matrix to the fibre depends on the state of this interface and, in particular, the level of the fibre/resin adhesion. The standard deviations of matrix-dominated properties of such materials are relatively large. The important reason for the sensitive behaviour is due to the hygrothermal ageing of FRP composites. The present study aims to investigate the effects of hygrothermal exposure on transverse and ± 45' tensile properties of carbon/epoxy composites. The test results indicate that it could be the sensitive technique for the assessment of the interfacial adhesion strength in the composite. The degree of degradation of transverse tensile strength with the amount of absorbed moisture is incomparably higher in compare to the variation of ± 45' tensile strength. The quantitative correlation among the various techniques of interfacial adhesion in FRP composites, especially under moist and thermal environments, has yet to be concluded. Many studies demonstrated the importance of interfacial bond in controlling the transverse strength and stiffness of a composite.

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On Another Mechanism for Fatigue Strength Improvement of Metal Parts by Shot Peening: *Mei Yao*[1]; [1]Yanshan University, Col. of Matls. Sci. & Eng., 438 W. Hebei Ave., Qinhuangdao, Hebei 066004 China

Un-peened and shot-peened specimens made of quenched and tempered 300M steel were fatigue tested. The fatigue sources in specimens being tested at stress a little higher than their fatigue limits are located at the surface for un-peened specimens while, for shot-peened ones, in the interior. Obvious change of X-ray diffraction effect of both kinds of specimen being fatigue tested at stress equal to their fatigue limit has taken place in the surface layer for un-peened specimen, while, for shot-peened specimen, in the sub-surface layer. The calculated actual critical stress at the fatigue source position (the ìinternal fatigue limitî) for shot-peened specimen is about 138% of the (surface) fatigue limit of un-peened specimen. Then, the improvement of apparent fatigue limit of shot peened specimen with internal fatigue source is due to the fact that the internal fatigue limit of metal is higher than its surface fatigue limit.

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The Crack Propagation in Generalised Space and the Solid State Deformation Description: *Miklashevich A. Ihar*[1]; [1]Belarusian National Technical University, Theoretl. Mech., Fr. Skaryny Ave. 65, Minsk 220027 Belarus

The crack trajectory is described as a geodesic line of energy of fracture production in the Finsler space associated with the solid state deformation. The geometrical structure (metric properties) of material is determined by the distribution and kinds of defects through the structure of the connection coefficients definition. Three Cartanís tensors of curvature can be introduced independently. This help to describe all sorts of defects correctly. The symmetry group of deformation could be obtained from the fibering of manifolds into horizontal and vertical bundle. The gauge fields could be introduced basing on fibering. Method of determination of the fracture surface fractal dimension is proposed. Fractal dimension of the crack is determined as a ratio of Riemann crack trajectory length to Finsler crack trajectory. It is shown that fractal dimension is independent on the dislocation distribution in materials. Method of the fractal dimension control is proposed.

High Temperature Alloys: Processing for Properties: Cast Alloys and Machining

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee
Program Organizers: Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA; Jacqui B. Wahl, Cannon-Muskegon Corporation, Muskegon, MI 49443-0506 USA

Wednesday PM	Room: 13
March 5, 2003	Location: San Diego Convention Center

Session Chair: Gerhard E. Fuchs, University of Florida, Dept. of Matls. Sci. & Eng., Gainesville, FL 32611-6400 USA

2:00 PM
Effect of Carbon Additions to Single Crystal Ni-Base Superalloys: *Gerhard E. Fuchs*[1]; *Khalid Al-Jarba*[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

The effect of carbon additions to a model third generation single crystal superalloy was examined. Alloys were processed with 5 different carbon levels, including a baseline without an intentional additions. The addition of carbon to the model alloy resulted in the formation of MC-type carbides and a significant reduction in the number of solidfication defects observed. The effect of these carbon additions on

the microstructure, properties, microstructural stability and castability was examined. The microstructure and tensile and creep properties of the samples were examined after conventional solution and aging heat treatments and after long-term thermal exposures, and correlated back to the carbon additions.

2:20 PM
Effect of Solidification Processing Parameters on Microstructures and Properties of CMSX-10: *Gerhard E. Fuchs*[1]; *Elyssa Cutler*[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

A single master heat of CMSX-10, a third generation, single crystal Ni-base superalloy was processed with withdrawal rates ranging from 5cm/hr to 35cm/hr. The as-cast microstructure and solidification partitioning was characterized as a function of withdrawal rate and used to determine the gradient. After heat treatment, the microstructure and tensile and creep properties were determined. The microstructure and properties were correlated back to the withdrawal rate.

2:40 PM
Comparison of Directionally Solidified Castings Using Radiation and Liquid Metal Cooling (LMC): *Andrew J. Elliott*[1]; Tresa M. Pollock[1]; Michael F.X. Gigliotti[2]; Warren T. King[3]; [1]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]GE Corporate Research & Development, Niskayuna, NY 12309 USA; [3]GE Power Systems, Greenville, SC 29602 USA

A columnar grain variant of single crystal RenÈ N4 has been directionally solidified using a simplified geometry with cross-sections similar to an industrial gas turbine blade. Castings were solidified at rate of 2.5mm/min using conventional radiation cooling and at rates between 2.5 and 8.5mm/min using liquid metal cooling (LMC) with tin as a cooling medium. Thermal gradients were directly measured by thermocouples in the casting. The LMC process exhibited higher thermal gradients at all withdrawal rates. Combining high thermal gradients with the capability for faster withdrawal rates produces significantly increased cooling rates. The higher cooling rates result in refined structure measurable by the finer dendrite arm spacing. Additionally the conventionally cast material exhibited several freckles while none where observed in the LMC castings.

3:00 PM
Solution Heat Treatment Reponse of CMSX-4: *Gerhard E. Fuchs*[1]; *Brandon Wilson*[1]; Jennifer Hickman[1]; Ryan Scott[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 11640, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

The standard solution heat treatment of the second generation single crystal Ni-base superalloy, CMSX-4, requires several holds at high temperature. Cost reductions may be possible if the duration and/or temperatures of the holds in solution heat treatment could be reduced. Therefore, this study examines the effects of each step of the solution heat treatment on the microstructure, segregation and phase transformation temperatures of CMSX-4 single crsytal samples. The results of this study were then evaluated with respect to the potential to reduce the solution heat treatment temperatures and hold times.

3:20 PM Break

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The Effect of Solution Heat Treatment on the Microstructure and Properties of CMSX-4: *Gerhard E. Fuchs*[1]; *Brandon Wilson*[1]; Jennifer Hickman[1]; Ryan Scott[1]; [1]University of Florida, Matls. Sci. & Eng., PO Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA

The mechanical properties of CMSX-4 are critical for its application to gas turbine engines. In order to achieve the optimal balance of properties, the materials must given the appropriate solution heat treatment. Without a solution heat treatment, superalloys would not be able to withstand the stresses or the environment to which they are subjected. Therefore, if the effect solution heat treatments have on mechanical properties could be determined, then the heat treatment step could possibly be shortened. Because of these concerns, research was conducted to observe the effect of solution heat treatments on the mechanical properties. Three sample groups were used for the testing. One group has no solution heat treatment, another has the full solution heat treatment, and the third group has the PWA 1480 solution heat treatment. The PWA 1480 heat treatment consists of lower temperatures and a shorter duration than the standard heat treatment. These results help determine the feasibility of shortening the solution heat treatment step; therefore, reducing production costs.

4:00 PM
Hydrogen Annealing Superalloys for Improved Oxidation Resistance: *James L. Smialek*[1]; [1]NASA Glenn Research Center, Matls. Div., 106-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

Hydrogen annealing was used to desulfurize superalloys from nominally 10 ppmw to less than 0.1 ppmw to eliminate sulfur segregation and to improve scale adhesion and cyclic oxidation resistance. A range of sulfur contents was produced for PWA1480 samples by annealing at various temperatures (1000-1300°C), times (8-100 hr), and sample thickness (10-200 mils), as determined by the diffusion parameter, Dt/L2. Performance in 1100°C, 1000 hr cyclic oxidation tests show a distinct dependence of scale adhesion on low sulfur content. Similarly, hydrogen annealing ReneíN5 alloys produced 1150°C cyclic oxidation behavior superior to that of Y-doped alloys, due to desulfurization and decarburization. Finally, H2-annealing PWA1484 samples improved no-bondcoat PS-8YSZ-TBC 1100°C cyclic furnace lives to 1000-2000 hr, compared to 200 hr without annealing. A rule of thumb for maximum scale adherence is obtained whereby the total amount of sulfur available for segregation should be less than about 1 monolayer.

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Thermally Assisted Machining Processes: *Aparna Shilpa Durbhakula*[1]; *Sharada Gollapudi*[1]; Manasa Manga[1]; [1]Vasavi College of Engineering, Dept. of Mechl. Eng., Ibrahimbagh, Hyderabad, AP 500031 India

Advanced materials like ceramics and high-temperature alloys are widely used due to their superior mechanical properties like such as high hardness, thermal stability and wear resistance. They can be fabricated into products by a broad range of manufacturing processes but forming process can produce various shapes with a high production rate, though one has to compromise on dimensional accuracy and surface finish. A finishing operation is hence required. However, many advanced materials are known to be very difficult to machine. Hence, there exists a need for a more efficient machining techniques, particularly for such brittle materials. A promising technique is a thermally assisted machining process, an innovative and economic technique for machining ceramic materials by first softening them with heat from a laser.

Hot Deformation of Aluminum Alloys: Processing, Structure and Property - II

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

Wednesday PM Room: 6E
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Mike E. Kassner, Oregon State University, Mechl. Eng. Matls. Prog., Corvallis, OR 97331-6001 USA; Zhe Jin, Alcoa Technical Center, Thermomechl. Procg. & Alloy Dvlp., Alcoa Ctr., PA 15069 USA

2:00 PM Invited
Optimization of Sound Castings Aluminum Alloys for Automotive Structural Applications by L.H.I.P.: Marcello Cabibbo[1]; *Enrico Evangelista*[1]; Sergio Gallo[2]; Valentina Latini[1]; [1]University of Ancona, Mech., Via Brecce Bianche, Ancona I-60131 Italy; [2]Teksid SpA, Borgaretto 10040 Italy

The need for weight reduction in the automotive applications is bound in the increasing fuel consuming request due to a reduction need for CO2 pollution. Sand cast aluminum alloys often suffer from voids due to gas entrapped (H2, O2, N2) by the liquid alloy during turbulent filling of the die and from shrinkage during solidification. Both shrinkage voids and gas inclusion pores impair strength and fatigue resistance, as they can easily coalesce to premature fracture. Liquid Hot Isostatic Pressing (L.H.I.P.) process, by the combination of pressure and temperature, is able to close the voids due to shrinkage and drastically reduce the pores due to gases entrapment (H2, N2) through the dissolution of gaseous inclusions within the lattice. This process repre-

sents the evolution of H.I.P. (Hot Isostatic Pressing) in which a gas, argon, is used as the pressuring medium. L.H.I.P., presently, constitutes the best valuable solution for obtaining shrinkage-free and poresminimum castings involving low-cost automotive co nstituents. These production advantages of L.H.I.P. have lead to strong interest in aluminum castings for applications where stringent mechanical requirements must be met. Recent tests on aluminum alloys, processed by L.H.I.P., showed good mechanical response: tensile and yield strength increased of up to 30 pct after L.H.I.P., while ductility tripled. Distribution pores have been investigated and characterized by means of light (LM) and electron microscopy (SEM) techniques. Statistical evaluations on L.H.I.P. processed aluminum alloys for automotive applications (such A356, A357, A319) revealed a pores mean size reduction of up 1/3 through L.H.I.P., the volume fraction being lowered down to one order of magnitude; yet, shrinkage voids were minimized in number and volume.

2:25 PM

The Optimized Tensile and Fatigue Properties of Experimental Semi-Solid Aluminum Alloys: S. C. Bergsma[2]; *M. E. Kassner*[1]; M. Z. Wang[1]; [1]Oregon State University, Mechl. Eng., Rogers Hall, Corvallis, OR 97331-6001 USA; [2]Northwest Aluminum Company, The Dalles, OR 97058 USA

The mechanical properties of various semi-solid formed (SSM) aluminum alloys (experimental designations DF-44, 51 and 95) are reported. These were Al-Si alloys that had various additions of principally Cu and Mg. The SSM parts were formed from ingot that was not stirred during solidification but had undergone a semi-solid thermal transformation (SSTT) during heating to produce a spherical microstructure suitable for semi-solid forming. The T5 and T6 tensile properties were also optimized for these alloys using various solutionizing and aging studies of formed parts. The fatigue properties of some of these alloys were also investigated.

2:45 PM Invited

Changes of Textures, Microstructures and Particles of a Continuous Cast Al-Mg Alloy During Hot Rolling: *Xiyu Wen*[1]; Tongguang Zhai[2]; Zhendong Long[1]; J. G. Morris[2]; [1]University of Kentucky, Ctr. for Al. Tech., 1505 Bull Lea Rd., Lexington, KY 40511 USA; [2]University of Kentucky, Cheml. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA

This paper reports an investigation of textures, microstructures and particles of a continuous cast Al-Mg alloy during hot rolling using X-ray diffraction, transmission electron microscopy, scanning electron microscopy and optical microscopy. It was found that the changes in texture during hot rolling reduction were followed by changes in the orientation distribution function. When the rolling reduction was below 38%, the development of the Brass and S texture components were inhibited, while above 38% in rolling reduction they increased sharply. In comparison, the copper component was continuously increased with increase in rolling reduction. Microstructure changes were studied with optical microscopy. The particle size, particle distribution and particle chemical composition, including both intermetallic and dispersion particles, were determined by TEM and SEM. The reasons for the changes in the character of intermetallic particles and dispersoids during hot rolling are discussed and are related to the particular determined texture.

3:10 PM

VIR[FAB], A Joint European Approach Towards Through Process Modelling of Hot Deformation of Aluminium Alloys: *Menno van der Winden*[1]; [1]Corus Research, Development & Technology, IJmuiden Techl. Ctr., PO Box 10.000, IJmuiden 1970 CA The Netherlands

In March 2002, the VIR[FAB] project has started. In this project, all major European Aluminium Producers collaborate with leading universities and research institutes. The objective of this project is to create a so-called Through Process Model (TPM) of the rolling and annealing operations that are part of the fabrication of aluminium sheet. This model predicts the influence of the (upstream) processing parameters on the (final) microstructure. Inevitably, a TPM can only function if the sub-models which constitute it, are linked to each other. The way in which VIR[FAB] has handled this is discussed. Furthermore, the complexity of working on one TPM with 15 different partners brings its own challenges, which will also be discussed. Another aspect that needs attention is the relative balance of efforts between the different sub-models. Typically, the focus has been determined by the specific interests of individuals. Linking the models creates the opportunity to do a comparative sensitivity analysis of the individual submodels. Additionally, the involvement of so many industrial partners in the project created the opportunity to validate the TPM against full-scale trials on different alloys and different plants. The results of this exercise will be discussed.

3:30 PM

Spray Rolling Aluminum Strip: Process Modeling and Parametric Studies: *J.-P. Delplanque*[1]; S. B. Johnson[1]; E. J. Lavernia[2]; Y. Zhou[2]; Y. Lin[2]; K. M. McHugh[3]; [1]Colorado School of Mines, Eng. Div., Golden, CO 80401-1887 USA; [2]University of California, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616-5294 USA; [3]Idaho National Engineering and Environmental Laboratory, Industl. & Matl. Tech. Dept., Idaho Falls, ID 83415-2050 USA

A joint project, between the Idaho National Engineering and Environmental Laboratory, the University of California-Davis, and the Colorado School of Mines, has been undertaken to investigate the feasibility of the spray rolling process and to evaluate the material properties of spray-rolled aluminum alloys. This investigation is being accomplished in part through modeling based characterization of the spray-rolling process. Models describing the behavior of individual droplets may be integrated and a statistical approach taken to relate process parameters to the state of the spray prior to impact on the rolls. Knowing the condition of the deposited material, the rolling process may then be modeled. Simulations are conducted to explore the process parameter space.

3:50 PM

Spray Rolling Aluminum StripñProcess Development and Strip Properties: *Kevin M. McHugh*[1]; E. J. Lavernia[2]; Y. Zhou[2]; Y. Lin[2]; J.-P. Delplanque[3]; S. B. Johnson[3]; [1]Idaho National Engineering and Environmental Laboratory, Industl. & Matl. Tech. Dept., PO Box 1625, MS 2050, Idaho Falls, ID 83415-2050 USA; [2]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., CA 95616-5294 USA; [3]Colorado School of Mines, Eng. Div., CO USA

Spray rolling is a new manufacturing technology that is under development for producing aluminum strip and other flat products. It consists of atomizing molten metal with a high velocity inert gas, quenching the resultant droplets in-flight, and directing the spray between mill rolls. Hot deformation of the semi-solid material results in the formation of a fully consolidated, rapidly solidified product. Results of a collaborative project between the Idaho National Engineering and Environmental Laboratory, The University of California-Davis, and the Colorado School of Mines to develop spray rolling technology are presented. This paper describes process development issues, and compares the microstructure and material properties of spray-rolled 3003 and 5083 aluminum alloys with commercial material made by conventional ingot/metallurgical processing.

4:10 PM

Rolling Process Optimizations by Sensor Fusion Control Architectures: *George A. Fodor*[1]; [1]ABB Automation Technology Products, Force Measurement, Vasteras SE-72159 Sweden

Process optimization using real-time gray and white physical models is a favorite approach to assess material microstructure and surface properties in aluminum thermo-mechanical processes. Employing a set of models achieve the balance between the process optimization performance goals vs. estimating a large number of physical parameters. Additionally, a large set of sensors ensures that a vector space of appropriate size is available for the estimation of the model parameters. Industrial-grade flexible model-based systems that allow rich sensory equipment for non-laboratory conditions have been recently introduced in industry. The key technological advance that allows this introduction is the use of sensor fusion control architectures. The paper presents a modeling and control approach in hot / cold rolling of aluminum using ABB integrated modeling and geometry sensor equipment. Results show that parameter estimation has high accuracy, however a better model integration could increase substantially the estimated data available from measurements.

4:30 PM

The Effect of Homogenization Practice on the Microstructure of AA6063 Alloys: *Y¸cel B˝rol*[1]; Metin Usta[2]; Gˆkhan Kara[1]; Selda Ucuncuoglu[1]; Osman Cakir[1]; Ayhan N. Bal[3]; Timur Ulucak[3]; [1]Materials and Chemical Technologies Research Institute, Marmara Rsrch. Ctr., TUBITAK, Gebze, Kocaeli 41470 Turkey; [2]Gebze Institute of Advanced Technology, Gebze, Kocaeli Turkey; [3]SARAY Aluminum, G¸neʃli, Istanbul Turkey

Homogenization is an essential step in the preparation of aluminum billets for extrusion and is performed to produce a homogeneous solid solution and to transform the beta-AlFeSi particles to the finer and more equiaxed and thus more acceptable cubic alpha variety. Homogenized billets require lower extrusion pressures and give extrusions with better surface finish and higher strength than as-cast billets. The extrudability of the billet is maximized once the dissolutionizing of the

Mg2Si particles and the bÆa transformation are both complete. The latter takes higher temperatures and longer times and often requires help through revision of the alloy chemistry, i.e. addition of some Mn. The cooling practice in a homogenization treatment is just as significant as soaking and a variety of microstructures ranging from a fully solutionized homogeneous matrix to a heterogeneous one with a coarse dispersion of Mg2Si particles can be produced by simply adjusting the cooling rate. The optimum rate is that which gives a Mg2Si precipitation readily redissolvable during subsequent processing and is dictated by the preheating practice. The present work was undertaken to identify the optimum homogenization soaking and cooling practice for a semicontinous DC-cast AA6063 billet which, due to its composition (very low Mn), relies solely on the soaking practice for the bÆa transformation.

International Symposium on Gamma Titanium Aluminides: High Temperature Properties

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

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

Wednesday PM Room: 6F
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Thomas R. Bieler, Michigan State University, Metall. & Matls. Eng., E. Lansing, MI 48824-1226 USA; Patrick L. Martin, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433-7817 USA

2:00 PM Invited

Elementary Deformation Mechanisms Occuring During Creep of TiAl Alloys at 750∞C: *Couret Alain*[1]; Malaplate Joel[1]; [1]CEMES/CNRS, 29 Rue J. Marvig, BP 4347, Toulouse 31055 France

This presentation is aimed to study the elementary deformation mechanisms occurring during primary and secondary creep stages of TiAl alloys at 750∞C. Two Ti48Al48Cr2Nb2 alloys have been processed by powder metallurgy and casting, leading to the formation of duplex and lamellar microstructures, respectively. They have been subsequently crept at 750∞C under different applied stress. Stress jumps have been performed at different deformation levels in order to measure the activation volumes and the stress exponents. Results indicate that these two activation parameters are constant all over the curve. It is then concluded that primary and secondary stages are controlled by the same dislocation mechanism. The microstructures of crept samples have been investigated by transmission electron microscopy. Up to 3% of deformation, ordinary dislocations dominate the microstructure; they have been found to move by glide and climb. Respective contributions of these glide and climb processes are then discussed.

2:30 PM

Investigation of α_2/γ Phase Transformation Controlling the Creep Deformation Rate in Lamellar TiAl Intermetallics: *Seung Jin Yang*[1]; Hye Jin Jung[1]; Soo Woo Nam[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701 Korea

A lamellar TiAl alloy has been regarded as one of the strongest candidate for structural materials in high temperature use. In recent years, it has been known that the lamellar interface of TiAl alloy has an important role to control the creep deformation rate. In this study, the creep deformation mechanism and the phase transformation process occurred at the alpha2/gamma interfaces are discussed for the investigation of creep mechanism. At the early stage of primary creep α_2-ledges at the alpha2/gamma interface are transformed to gamma phase with the absorption of dislocations and the irregular shape of the interface is changed to be flat. After the early stage, continual α_2 to gamma phase transformation is occurred at the flat interface with the emission of dislocations. This phase transformation involving the emission and absorption of dislocations may control the creep deformation rate and atomistic models of this phase transformation are suggested.

2:50 PM

Influence of Different Texture Aspects on Creep Properties of a Fully Lamellar Ti-47Al-2Cr-2Nb Alloy: *Thomas Marc*[1]; Zghal Slim[2]; Sanchez Claire[1]; Couret Alain[2]; [1]ONERA, DMMP, 29 Ave. de la Division Leclerc, Chatillon, Cedex 92322 France; [2]CEMES-CNRS, 29 Rue J. Marvig, BP 4347, Toulouse, Cedex 4 31055 France

The texture-stress-creep relationships have been evaluated in this two-phase alloy of composition Ti-47Al-2Cr-2Nb (at%) with a fully lamellar microstructure. We have performed a number of mechanical tests on appropriately oriented specimens that have confirmed the texture behavior already assessed in PST-single crystals. This mechanical data base has been used successfully for the interpretation of deformation modes in a fully lamellar microstructure. Additionally, varying the cooling rate was found to significantly alter the decomposition modes of the high-temperature alpha phase, yielding different gamma/gamma interface type distributions. We will review TEM observations highlighting three different mechanisms occurring at different temperature ranges. The related microstructural development made relevant statistical studies of the distribution of individual lamellae to be carried out for the interpretation of lamellar phase transformations. This work aimed at determining if the different sequences of lamellar interfaces would play a significant role in measuring the creep properties in lamellar TiAl-based alloys.

3:10 PM

The Effect of Mo and Al Content on the Creep Deformation of Ti-48Al-2Cr-2Nb-xMo Titanium Aluminides: *Eric A. Ott*[1]; Tresa M. Pollock[2]; Patrick L. Martin[3]; [1]GE Aircraft Engines, Matl. & Process Eng., 1 Neumann Way, MD M89, Cincinnati, OH 45215 USA; [2]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., H. H. Dow 2042, Ann Arbor, MI 48109 USA; [3]Wright Patterson AFB, Matls. & Mfg. Direct., Air Force Rsrch. Lab., Dayton, OH 45433 USA

The creep testing of Ti-48Al-2Cr-2Nb-xMo alloys for x between 0 and 1 atom % has shown that the minimum creep rate is significantly affected by Al and Mo content. Increased Al levels in alloys on the Al-lean side of stoichiometry resulted in decreased creep rates for equiaxed type microstructures. Mo present in solid solution in the gamma phase also resulted in decreased creep rates for these structures. For alloys containing equal to or greater than 0.5 atom % Mo, small amounts of the B2 phase were present in the microstructure, however, the presence and amount of B2 between 0 and 5 volume % did not result in significant creep strengthening. Creep testing of alloys having microstructures which contain other alpha2 morphologies including combinations of lamellar and Widmanstatten alpha2 plates confirmed that the presence of Mo in solid solution also results in decreases in the minimum creep rate.

3:30 PM

Strength Properties of Niobium Containing TiAl Alloys: *Jonathan D.H. Paul*[1]; Uwe Lorenz[1]; Stefan Eggert[1]; Michael Oehring[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Str., Geesthacht D-21502 Germany

Titanium aluminides are nearing use in aerospace applications and are already being used in automobile applications. Alloys of industrial relevance must not only show high strength and ductility combined with oxidation and creep resistance but must also exhibit predictable mechanical behaviour. A novel class of alloys that seems to meet the above demands contain large amounts of niobium. However, according to the Griffith criteria, the high strengths obtainable in these alloys require that the minimum defect size be reduced. Therefore good microstructural and chemical homogeneity are required, combined with relatively small colony sizes in fully lamellar materials. Thus, the aim of this paper is to investigate the room temperature mechanical properties of a high niobium-containing alloy in terms of Weibull statistics and thus obtain information regarding material reliability.

3:50 PM Invited

Creep of Directionally Solidified TiAl-Base Alloys and their Tensile Properties: Yuji Omiya[1]; Haruyuki Inui[1]; *Masaharu Yamaguchi*[1]; [1]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan

TiAl-base alloys with a lamellar microstructure aligned parallel to the growth direction possess a good combination of mechanical properties. The lamellar orientation of a wide variety of TiAl-base alloys can be controlled through seeding and directional solidification. Mechanical properties of directionally solidified ingots of binary TiAl and TiAl-Si-X alloys have been investigated and, indeed, found that they exhibit an excellent combination of room temperature tensile properties and high temperature strength, in particular, creep strength. In this paper, the results of investigations on the creep and tensile

properties of directionally solidified ingots of the binary and multi-component alloys using relatively large-sized specimens are presented.

4:20 PM
Multi-Axial Creep Strength in Gamma-Ti Base Alloys: *Kamran Nikbin*[1]; [1]Imperial College, MED, Exhibition Rd., London SW7 2BX UK

Gamma-Titanium Aluminide were received in four batches, three of which consisted of rectangular blocks and one batch consisted of a section of a cast blade. The batches were of the same nominal chemical composition bu had a wide range of grain sizes, in particular the batch from blade section. Notched bar specimens, with sharp and medium notch acuities were machined and tested at 750°C under static load. In addition compact tension specimens were also tested in order to ascertain the multi-axial failure behaviour of this material. The results are compared with uniaxial results on the same batches. The mode of failure in this material shows that notch strengthening exists for short term tests. However for longer term tests the multiaxial failure shows insensitivity to variation in batch and grain size whereas the uniaxial failure shows a marked reduction in failure lives between their different blocks and blade material. A metallographic examination is undertaken and an effective stress criteria is used to identify the mode of failure in the notched bar specimens.

4:40 PM
Characterization of Gamma TiAl Castings for Engine Blade Component: *Sadao Nishikiori*[1]; [1]Ishikawajima-Harima, IHI, Matls. Dept., Rsrch. Lab., 1 Shin-nakaharachou, Isogo-Ku, Yokohama 235-8501 Japan

In this study, castabilities, weld repairabilities and mechanical properties have been investigated for four engineering TiAl alloys which include 47XD alloy, K5C alloy, and two IHI alloys (TiAl-Mo-V-Si and TiAl-Mo-V-Si-C). Through casting experience of the engine blade component on sub-production scale, castability of each alloy has been evaluated. TiAl-Mo-V-Si alloy shows good component filling. For other alloys, counter gravity casting process is discussed to improve component filling. In order to reduce the cost of castings, development of weld repair technology is investigated. Proper welding of casting cracks should yield increased productivity. The weldability of these alloys is compared along with pertinent welding processes. The mechanical properties of these cast alloys were evaluated in fully lamellar microstructure forms, with particular emphasis of carbon effect on creep resistance. The effect of the carbon addition on the lamellar colony size is also discussed.

5:00 PM
High Temperature Fatigue Evaluation of a Cast TiAl Intermetallic Compound: *Massimo Marchionni*[1]; Giovanni Onofrio[1]; [1]CNR-IENI, Tempe, Via Cozzi, 53, Milano 20125 Italy

Gamma Titanium aluminide alloys are of great interest in high temperature application, due to their low density, high stiffness and satisfactory elevated temperature mechanical properties. The material performance in low cycle fatigue and fatigue crack propagation regimes is very important from a design point of view, together with the evaluation of the thermomechanical fatigue (TMF) behaviour that can describe the material service condition with great accuracy. The paper describes the low cycle fatigue (LCF) and fatigue crack propagation (FCP) behaviour of a duplex gamma TiAl aluminide alloy in the temperature range of 600∞C-800∞C and the aspect of fatigue damage and material degradation. Some tests have been performed in the thermomechanical (TMF) fatigue regimes, assuming testing conditions similar to those of the component in service (diamond cycle, temperature range 300-800∞C, etc.) The fatigue results have been analysed according to Coffin-Manson and Paris relationships and the analysis of fracture surfaces has also been performed in order to evaluate the fatigue damage mechanisms.

5:20 PM
Investigations of the Creep Behavior and the Onset of Microstructural Instability During Short Term Creep in a Gamma TiAl Based Alloy with Fully Lamellar Microstructure: *Anita Chatterjee*[1]; Helmut Clemens[2]; Gerhard Dehm[3]; Christina Scheu[3]; Heinrich Mecking[4]; Eduard Arzt[3]; [1]GfE Metalle und Materialien, Nuremberg 90431 Germany; [2]GKSS Research Center, Geesthacht 21502 Germany; [3]Max-Planck Institut f¸r Metallforschung, Stuttgart 70569 Germany; [4]TU Hamburg-Harburg, Hamburg 21073 Germany

It is well established that the interface spacing in fully lamellar microstructures has a major influence on creep behavior of gamma-TiAl base alloys. In this paper, the dependence of the creep properties on interface spacing in fully lamellar Ti-46.5at.%Al-4at.%(Cr,Nb,Ta,B) sheet material as well as the onset of microstructural instability during short term creep at 800∞C was studied. Creep tests were conducted over a temperature range of 700 to 900∞C and at stresses between 100 and 260 MPa. The results indicate that the primary creep strain as well as the minimum creep rate decrease with decreasing interface spacing. A model based on the work hardening theory was applied to explain the role of the interface spacing on both the primary creep strain and secondary creep rate. The observed microstructural instability during creep is correlated with an Ti_3Al content exceeding the thermodynamical equilibrium due to the formation process of the fully lamellar microstructure.

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

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)
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Wednesday PM Room: 8
March 5, 2003 Location: San Diego Convention Center

Session Chairs: David G. Morris, CENIM CSIC, Dept. of Physl. Metall., Madrid E-28040 Spain; Guoliang Chen, University of Science & Technology-Beijing, State Key Lab. for Adv. Metals & Matls., Beijing 100083 China

2:00 PM Invited
Laves Phases: Structure, Structural Stability and Polytypic Transformations: *Sharvan Kumar*[1]; [1]Brown University, Div. of Eng., Box D, 182 Hope St., Providence, RI 02912 USA

Laves phase structures have been examined for their potential as high temperature structural alloys as well as for hydrogen storage. These compounds occur with cubic (3C) or hexagonal (2H, 4H) structures, and their compositional range of existence is apparently dictated by the e/a ratio. According to published phase diagrams of the Cr-X binary systems (X = Ti, Zr, Hf, Nb or Ta), the Laves phase is present in its hexagonal form at high temperatures and in the cubic form at low temperatures. Experimental observations of two-phase alloys in several of these systems demonstrate that the hexagonal structure is often metastably retained at room temperature and subsequent annealing at elevated temperatures transforms the hexagonal structure through a series of polytypes to the stable cubic structure. In two-phase alloys, this transformation is extremely sluggish and is accompanied by a compositional change. Current understanding and issues related to this transformation will be discussed.

2:20 PM Invited
Progress of Advanced Material Science and Technology in China: *Baiyun Huang*[1]; [1]Central South University, Changsha 410083 China

In this paper, the status quo of material science and technology in China has been discussed firstly. In some high-tech research field, for example, nano-materials, artificial crystals, magnetic materials, China has made significant progress. But, in some research field, for example, high performance steel, processing techniques for high quality Al, much work should be done. The affiliation to WTO has provided many opportunities and privileges to Chinaís material industry, but brought many challenges at the same time. In recent years, China has

paid much effort to accelerate its progress in material science and technology. It has launched an important programs, National High-Tech Research and Development Programme (863), focusing on high technique research. Material science and technology has been paid much attention in this programme, and is a subdivision of this programme, called National Advanced Materials Program. The organization, priority research field and the progress of some important projects in the National Advanced Materials Program have been described in this paper. As information industry grows very fast, its demands for high performance materials are more and more imminent. An important part of this program is allotted to photoelectronic materials and electronic materials. In order to enhance the competitiveness of Chinaís material industries and material-related industries, much efforts have been paid to R&D of high performance structural materials and functional materials. Considering the technical reserve for future development, nanometer materials and superconductive materials are also under investigation in this program.

2:40 PM Invited

Phase Equilibria and Structure Change in Quasibinary Ni_3Nb-Ni_3M Systems at Elevated Temperatures: *Masao Takeyama*[1]; Mitsuhisa Yamanaka[1]; Akane Suzuki[1]; Yukinori Yamamoto[1]; Takashi Matsuo[1]; [1]Tokyo Institute of Technology, Dept. of Metall. & Ceram. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Ni base A_3B compounds with different crystal structures can be classified into two types in terms of B atom arrangement on the close-packed planes; one is R-type that B atoms set in rectangular array, and the other T-type in triangular array. Ni_3Nb-δ ($D0_a$) belongs to R-type with its superlattice stacking sequence of abab, and others are Ni_3Ti-η ($D0_{24}$:T-type, abac), Ni_3Al-γí ($L1_2$:T-type, abc), Ni_3V-γî ($D0_{22}$:R-type, abcaíbící). Among them, $D0_a$ has the lowest crystal symmetry whereas $L1_2$ phase is the highest. In quasibinary systems of these compounds, some of them has been well established, i.e. Ni_3Al(T)-Ni_3Ti(T). However, as far as R-T combinations are concerned, a very limited number of studies has been reported. In this study, we have mainly studied the phase equilibria between Ni_3Nb(R)-Ni_3M(T or R) systems. The structure change and stability of these compounds including new intermediate phases will be discussed in terms of tetragonality.

3:00 PM Invited

A Study on the Directionally Solidified Nb/Nb5Si3 Composites: *Yafang Han*[1]; Shiyu Qu[1]; Rongming Wang[2]; [1]Beijing Institute of Aeronautical Materials, PO Box 81-4, Beijing 100095 China; [2]Peking University, Physics Dept., Beijing 100871 China

The directionally solidified Nb/Nb5Si3 composites have been fabricated using optical floating zone (OFZ) technology and heat-treated at 1550∞C for 100 hours in Ar atmosphere. The microstructure of the composites has been investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM) equipped with X-ray energy dispersive spectrum (EDS) The results show that the microstructure of the composites consists of consists of dispersed Nb particles and two kinds of niobium silicides matrix, i.e., Nb3Si and Nb5Si3 phases. After heat-treated at 1550∞C for 100 hours, the equilibrium Nb+Nb5Si3 dual-phase microstructure is acquired via a eutectoid reaction, Nb3Si->Nb+Nb5Si3. The room temperature compression property of the directionally solidified Nb-18.7Si (at.%) alloy have been investigated, and compared to those of the Nb-10Si (at.%) and Nb-18.7Si (at.%) alloys fabricated by arc melting. The results indicated that the fracture stress of the directionally solidified Nb-18.7Si (at.%) alloy is as high as 1.9 GPa, which is about 20% higher than those of composites fabricated by arc-melting and no yield has been found. The fracture surface of composites consists of a great amount of like-cleavage facets.

3:20 PM

Interstitial Chemistry and Oxidation Resistance of Ti_5Si_3 and $Ti_5Si_3Z_x$ (Z = C, N, O): *Andrew J. Thom*[1]; Jason J. Williams[2]; Mufit Akinc[3]; [1]Iowa State University, Ames Lab., 142 Spedding Hall, Ames, IA 50011 USA; [2]Arizona State University, Tempe, AZ 85287 USA; [3]Iowa State University, 3053 Gilman Hall, Ames, IA 50011 USA

Changes in the structure of Ti_5Si_3 were measured by x-ray and neutron diffraction as carbon, nitrogen or oxygen atoms were systematically incorporated into the lattice. The measured trends in lattice parameters of Ti_5Si_3 show that most of the previous studies on supposedly pure Ti_5Si_3 were actually contaminated by these pervasive light elements. The oxidation behavior of Ti_5Si_3 was then carefully studied to reconcile inconsistencies observed in the recent literature. Ti_5Si_3 has poor oxidation resistance in air due to formation of a rutile-rich scale and sub-scale phases. In contrast, $Ti_5Si_{3.2}$ has excellent oxidation resistance because of the formation a silica scale. Samples with interstitial oxygen or nitrogen show only slight improvements in the early stages of oxidation compared to Ti_5Si_3, which is in stark contrast to previous research. However, samples with interstitial carbon displayed excellent oxidation resistance, consistent with previous research.

3:35 PM Break

3:50 PM Invited

Anomalous Grain Size Effects During Creep of Some Intermetallics: *K. Sadananda*[1]; C. (Jerry) R. Feng[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 20375 USA

Grain size normally affects creep rates in metals and alloys in the Newtonian Viscous regime. The grain size exponent, p, is normally around 3 for Coble creep involving grain boundary diffusion. Its value is around 2 for Naborro-Herring creep. Intermetallics such as MoSi2 and stoichiometric TiAl show grain size exponents greater than 4. Furthermore, the grain size effects persists even into power-law creep regime. In addition, the governing mechanism depends on the loading history. These anomalies will be discussed in light of the existing theories of creep.

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ìFe Effectî on Environmental Embrittlement of NiTi Alloys: *Jiahong Zhu*[1]; Chain T. Liu[2]; [1]Tennessee Technological University, Dept. of Mechl. Eng., 115 W. 10th St., Box 5014, Cookeville, TN 38505 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831 USA

Environmental effect on mechanical behavior of intermetallic alloys has been an ongoing research topic for over a decade. Even though significant progress has been made in this area, there are still many challenges to be answered. Recently, a unique ìFe effectî on environmental embrittlement in NiTi alloys was discovered in our laboratory. It was shown that there exists a critical Fe content in NiTi, i.e., about 9 at.%, below which no environmental embrittlement was observed and above which hydrogen from test environments severely embrittled NiTi. The mechanism responsible for the observed ìFe effectî was addressed in this presentation. Hydrogen diffusivity measurements from hydrogen-charging experiment show that Fe addition reduces the hydrogen diffusivity in NiTi, implying that the observed ìFe effectî is not due to reduction of hydrogen diffusion in the material. Also, laser desorption mass spectrometric results indicate the kinetics of surface reaction to generate atomic hydrogen does not change very much with Fe additions. Based on the experimental evidences, it is proposed that the existence of a critical Fe content is closely related to the effect of Fe on the critical hydrogen concentration to induce hydrogen embrittlement in NiTi. This research was sponsored by the Division of Materials Sciences, US Department of Energy, under contract DE-AC05-96OR22464 with UT-Battelle, LLC.

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Effects of Microstructure on the Oxidation Behavior of Mo-Rich Mo-Si-B Intermetallics: *Voramon Supatarawanich*[1]; David R. Johnson[1]; Chain T. Liu[2]; [1]Purdue University, Matls. Sci. & Eng., 1289 MSEE Bldg., W. Lafayette, IN 47907-1289 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS6115, Oak Ridge, TN 37831-6115 USA

The effects of volume fractions and morphologies of the Mo, Mo3Si, and Mo5SiB2 (T2) phases on the oxidation behavior of the multiphase alloys at 600-1300C were examined. The alloys were cast and heat treated at 1600C before the cyclic oxidation tests. The multiphase alloys showed poor oxidation resistance at 800C while a protective glass scale formed at 1300C. At the oxide/matrix interface, a Mo layer was found adjacent to the T2 phase while a Mo5Si3 interfacial layer was found adjacent to the Mo3Si phase. The alloy with 50 vol.% bcc-Mo solid solution formed a protective scale at 1300C. However, the specimens with the highest B/Si ratio (the lowest volume fraction of Mo3Si) did not form a protective glass scale at 1300C. The results indicate that, the Mo3Si phase is an important source of silicon for controlling the composition of the protective glass scale.

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Effects of Environment and Temperature on the Mechanical Behavior of the Ni-19Si-3Nb Intermetallic Alloy Doped with Boron and Carbon: *Shian-Ching Jang*[1]; [1]I-Shou University, Dept. of Matls. Sci. & Eng., 1, Sec. 1, Hsueh-Cheng Rd., Ta-Hsu Hsiang, Kaohsiung Co. 84008 Taiwan

The effect of the small additions of boron and carbon on the environment embrittlement at different temperature of the Ni-19Si-3Nb based alloy was investigated by atmosphere-controlled tensile test in various environments. The results revealed that the Ni-19Si-3Nb base alloy performs very ductile mechanical behavior (UTS ~1250 MPa, e ~14%) at room temperature in vacuum (2 x 10®C4 torr) as well as in pure oxygen atmosphere. This indicates that the environmental

embrittlement of water vapor is the major factor to deteriorate the ductility of Ni-19Si-3Nb base alloy. In parallel, the effect of 300 ppm boron addition improves the ultimate tensile strength and the ductility (UTS ®R 1200 MPa, e ®R 12%) in the Ni-19Si-3Nb base alloy significantly at room temperature in various atmosphere, such as vacuum, pure oxygen, air (contains 14000 ppm water vapor), and pure water vapor (contains 850 ppm water vapor). In addition, the yield strength of the Ni®C19Si-3Nb-0.15B-0.1C alloy was revealed to exhibit an increasing trend of temperature dependence, and the maximum yield strength occurs at 600°Ê. However, the ductility of the Ni®C19Si-3Nb-0.15B-0.1C alloy drops obviously when the temperature increases to 700°Ê. In addition, the observation of fracture surface for the specimen tested at different temperature revealed a transition temperature of fracture mode between 600 °Ê to 700 °Ê. The fracture mode changes from ductile dimple fracture mode into the brittle cleavage fracture mode.

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Synthesis of Titanium Nitride Nano-Particles by Reduction of TiCl4 in Liquid Ammonia: *Hongmin Zhu*[1]; Boyan Yuan[1]; Gensheng Sun[1]; Zhangmin Cao[1]; [1]University of Science & Technology Beijing, Dept. of Physl. Chem., 30 Xueyuan Rd., Beijing 100083 China

In the media of liquid ammonia, titanium tetrachloride was reduced by sodium dissolved in liquid ammonia. The product of the reaction was nano-sized fine powder and sodium chloride dissolved in the solvent. The nono-powder was heated under different pressure, and crystalline titanium nitride were obtained. The particle size was in the range of some ten non-meters varying with the heat temperature. The product powder was analyzed by different method, and the component ratio of titanium to nitrogen in the powder was around 1.

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Mechanical and Structural Properties of Aluminum Doped Mo5Si3: *Pedro D. Peralta*[1]; W. Hernandez[1]; Thomas Groy[2]; Raul Coltters[3]; [1]Arizona State University, Dept. of Mech. Eng., Main Campus, MC 6106, Tempe, AZ 85287 USA; [2]Arizona State University, Dept. of Chem. & Biochem., Main Campus, MC 1604, Tempe, AZ 85287 USA; [3]Universidad Simon Bolivar, Dept. de Ciencia de Materiales, Caracas, DF Venezuela

Mo5(Si,Al)3 alloys with 3, 5, 7 and 10 at% Al (nominal) were prepared using arc-melting. Microstructural characterization was carried out using Scanning Electron Microscopy, and the lattice structure was studied using X-ray power diffraction. The results indicate that a second phase appears after a nominal composition of 7 at% Al and that the lattice parameters in the solid solutions increase with Al additions. Microhardness was used to characterize the mechanical properties as a function of composition. The hardness was found to decrease with increasing Al additions; however, the presence of extensive micro-cracking is still observed for all compositions. This suggests that Al does not contribute substantially to reduce the thermal expansion anisotropy in Mo5Si3, which is the main cause of post-processing cracks in this material. An alloy with 3 at% Nb and 5 at% Al presented drastically reduced micro-cracking, confirming previous literature reports on the beneficial effects of Nb additions.

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Glass Transition and Crystallization of $Mg_{75}Ni_{10}Nd_{15}$ Metallic Glass Studied by Temperature-Modulated DSC: *Z. P. Lu*[1]; C. H. Kam[2]; Y. Li[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6115 USA; [2]National University of Singapore, Dept. of Matls. Sci. 119260 Singapore

Glass transition and crystallization behavior of $Mg_{75}Ni_{10}Nd_{15}$ metallic glass has been systematically studied by a temperature-modulated differential scanning calorimeter (TMDSC). The truly vitreous state of as-spun $Mg_{75}Ni_{10}Nd_{15}$ alloy was confirmed by the direct observation of glass transition behavior for as-spun and isothermally annealed ribbons through TMDSC measurements. It was concluded that the glass transition on normal differential scanning calorimeter (DSC) heating curves was hidden due to the relatively strong heat release of the primary nanocrystallization of intermetallic Mg_3Nd phase. This was further verified by the fact that the comparable glass transition behavior was observed by both TMDSC and DSC for final residual amorphous matrix in which the primary nanocrystallization has finished. The $Mg_{75}Ni_{10}Nd_{15}$ metallic glass was confirmed to crystallize in a primary crystallization m ode, e.g. Amó>Am[i] + Mg_3Ndó>an unknown phase + Mg_3Nd. Clear observation of structural glass transition and full understanding of crystallization micromechanism in such alloys can offer us new alloying and processing strategies for the preparation of bulk glassy alloys (e.g. consolidation route) and development of new nanocrystalline/amorphous composite materials.

International Symposium on Structures and Properties of Nanocrystalline Materials: Phase Transformation

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

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

Wednesday PM Room: 14B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Michael Atzmon, University of Michigan, Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; Vijay K. Vasudevan, University of Cincinnati, Matls. Sci. Dept., Cincinnati, OH 45221-0012 USA

2:00 PM

Transformation Behavior in Nanoscale Powders of Binary Aluminum-Copper Alloys: *Jixiong Han*[1]; Jai A. Sekhar[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Matls. Sci. Dept., PO Box 210012, Cincinnati, OH 45221-0012 USA

A study was made of the synthesis, microstructure and transformation behavior of nanoscale Al-Cu alloy particles. Nanoparticles were synthesized by plasma ablation from Al-4.4 wt.% Cu ingots. These particles were exposed to air and formed a 3-5 nm thick oxide scale. The particles were found to be in the supersaturated state displaying a variation in the individual particle composition. Several of the particles were faceted and bound by (111) planes. The nanoparticles were heat treated to examine the precipitation sequence. At the beginning of transformation, precipitates that were considerably enriched in copper were observed; these reduced in copper content as the heat treatment progressed. These precipitates were observed to form along the aluminum oxide-particle interface. Details of the precipitation sequence, nature and structure of second phase precipitates and interphase interfaces and formation mechanisms will be reported. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

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Size-Dependent Characterization of Gas Atomized Amorphous AlñGd-Ni-Fe Powders: *Baolong Zheng*[1]; Yizhang Zhou[1]; Enrique J. Lavernia[1]; [1]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616 USA

Great interest in the development of Al alloy systems is generated after the finding of aluminum based amorphous alloys with an Al content above 80 at. %, which exhibited tensile strengths of over 800 MPa as well as good ductility. This research is aimed at characterizing the amorphous formation ability of the atomized Al-7at.%Gd-2at.%Ni-1at.%Fe alloy powder. Based on experiment, an Al-7at.%Gd-2at.%Ni-1at.%Fe alloy was gas atomized with different gas atomization pressure and different atomization gas mixture, and its microstructure, thermal stability, transformation sequence and Micro-hardness are investigated with SEM, DSC and XRD as a function of powder particle size. With decreasing powder particle size, the volume fraction of amorphous materials increase, and micro hardness also increase. Powder with <25 m particle size can be considered fully amorphous, and 25-38 consisted of FCC-Al nano-crystals embedded in an amorphous matrix. The high thermal stability of the amorphous + fcc-Al nanostructured state and the high hardness of this fine powder make it a good candidate for consolidate bulk material for technological applications.

2:40 PM

Production and Characterization of Metal-C Composites (Where Metal=Al or Fe and C=Graphite or Fullerene) Obtained from Mechanically Alloyed Powders: *F. C. Robles-Hern·ndez*[1]; H. A. Calderon[2]; V. Garibay-Febles[3]; M. Umemoto[4]; [1]University of Windsor, Formerly at the Dept. de Ing. Met. ESIQIE-IPN, MÈxico, Windsor, Ontario Canada; [2]ESFM-IPN, Dept. de Ciencia de Materiales, MÈxico, D.F. MÈxico; [3]Istituto Mexicano del Petroleo, Formerly of the Dept. de Ing. Met. ESIQIE-IPN, Mexico, MÈxico, D.F. MÈxico; [4]Toyohashi University of Technology, Dept. Production Sys., Toyohashi 441-8580 Japan

This paper summarizes the results obtained with regards to the production and characterization of composites (Al or Fe) 15%at.C (where C is either graphite or fullerene (C_{60}+C_{70})). These composites were produced by using mechanical alloying techniques (horizontal and SPEX) under inert atmosphere. As-milled powders were compacted by using spark plasma sintering (SPS) at 773K, for 10min. and under 50MPa. SPS allowed a moderate grain growth from an average size of ~50nm to ~100nm. After sintering all systems reacted to produce carbides except for Fe-$C_{fullerene}$ that was rolled at 1273K. After rolling, fullerene showed a novel crystalline structure (tetragonal-I4/mmm). Several techniques like X-ray diffraction, scanning and transmission electron microscopy, microhardness and porosity-density measurements were performed in order to characterize microstructure and physical properties of the composites. Microhardness results from the Al-$C_{fullerene}$, and Fe-$C_{fullerene}$ before and after sintering increased from 50 to 300 HV and from 100 to 722 HV, respectively.

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Thermal Plasma Processing of SiC Powders: *Leo V.M. Antony*[1]; Jonkion M. Font[1]; Ramana G. Reddy[1]; [1]University of Alabama, Metallurgl. Eng., Box No 870202, Tuscaloosa, AL 35487 USA

Thermal plasma processing of fine powders has been the subject increasing interest over the past years. Its potential over the production of advanced materials is highly recognized. In this study, synthesis of ultra fine powder of SiC using non-transferred arc plasma has been studied. The raw silicon oxide powder and methane gas were used as the starting materials. The experiments were carried out at different power levels of the plasma reactor. Based upon the Gibbs energy minimization method the molar ratio of SiO2/CH4 was fixed for the system. The effect of the particle size on the vaporization time of the particle was studied. Scanning electron microscope (SEM), energy-dispersive X-ray spectrometer (EDS) and X-ray diffraction analysis were used for products characterization. The results showed that ultra pure fine spherical shaped SiC particles are produced.

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Experimental and Modeling Study of Cooling Rates for Atomizing Amorphous Al-Gd-Ni-Fe Powder: *Baolong Zheng*[1]; Yaojun Lin[1]; Yizhang Zhou[1]; Enrique J. Lavernia[1]; [1]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., Davis, CA 95616 USA

In order to obtain amorphous alloy, the melt is rapidly cooled into a solid state without crystallizing. Cooling rate is one of the most important parameters for making metallic glass. Based on experiment, analyses of atomized 2024 Al powders are presented. Efforts were focused on determination of secondary dendrite arm spacing (SDAS), cooling rate, and their correlation with powder size. Characterization of powders includes surface and interior morphology, particle size distribution, dendrite arm spacing and cooling rate. The work is aimed at simulation of cooling rate in atomizing amorphous Al-7at.%Gd-2at.%Ni-1at.%Fe alloy powder. The calculation for cooling rate modeling is based on Newtonian cooling with forced convection. The energy balance, gas dynamics, droplet dynamics, properties of the model alloy and atomization gas, and heat transfer between gas and droplet are considered in modeling calculation. The modeling results are helpful for insight understanding the process of atomizing amorphous Al-7at.%Gd-2at.%Ni-1at.%Fe alloy powder.

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Transformation Behavior in Nanoscale Powders of Binary Aluminum-Zinc Alloys: *Martin J. Pluth*[1]; Jai A. Sekhar[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Matls. Sci. Dept., PO Box 210012, Cincinnati, OH 45221-0012 USA

A study was made of the synthesis, microstructure and transformation behavior of nanoscale Al-Zn alloy particles. Nanoparticles were synthesized by plasma ablation from Al-15 wt.% Zn ingots. The particles were found to be in the supersaturated state, but displayed variation in composition compared with the bulk alloys. These particles were exposed to air and formed a 3-5 nm thick oxide scale. Several of the particles were faceted and bound by (111) planes. The alloy nanoparticles were heat treated to examine spinodal decomposition and/or precipitation. A spinodal structure was observed, as were precipitates of pure zinc with an f.c.c. structure. The precipitates were seen to form along the aluminum oxide-particle interface. Details of the precipitation sequence, nature and structure of second phase precipitates and interphase interfaces and formation mechanisms will be reported. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

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Processing Bulk Structures from Nanoscale Powders of Aluminum: *Martin J. Pluth*[1]; Jixiong Han[1]; Jai A. Sekhar[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Matls. Sci. Dept., PO Box 210012, Cincinnati, OH 45221-0012 USA

A study was made of the compaction, sintering behavior and workability of nanoscale aluminum powders into bulk structures. The nanoparticles of pure Al ranged in diameter from ~30 to 150 nm and contained a 2-5 nm outer oxide layer. The powders were cold-compacted in air, then sintered between 400 and 600°C. Both hot and cold rolling of the pressed pellets was utilized to assess workability and hardness after processing. Density was measured, and thin foils were prepared for TEM. In some cases, the oxide films surrounding the nanopowders were breached, resulting in grain growth and texturing after rolling. However, many of the nanoscale oxides did not break during processing and a very fine grain structure resulted. Similar studies on nanoscale powders of binary Al-Cu and Al-Zn were also performed. Results of the sintering and consolidation behavior of pure Al and alloy nanopowders will be presented. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

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Threading Dislocation Deduction by Nanometer Scale Lateral Epitaxy Overgrowth of GaN Over SiNx Nanomask Employing Block Copolymer Lithography: *Wei Zhou*[1]; [1]University of Southern California, Dept. of Matls. Sci., Compound Semiconductor Lab., Los Angeles, CA 90089 USA

Hexagonally ordered arrays of nanoscale holes were defined on thin SiNx film covered GaN buffer surface employing block copolymer lithography. Selective nucleation of GaN inside the predefined nanoscale holes was carried out followed by nanoscale lateral epitaxy overgrowth. Coalescence of surface was achieved within a thickness of 200nm, resulting in a smooth GaN surface. We demonstrated here that lateral epitaxy overgrowth of GaN in nanometer scale distinguishes itself from that in much larger scale in the sense that the migration has to be subdued instead of enhanced at the initial stage of lateral overgrowth. TEM observation shows that this approach may provide a promising path for GaN threading dislocation deduction uniformly across the sample surface.

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Electrodeposited Magnetic Nanostructures: *Vladimir Grigorívich Shadrow*[1]; Lyudmila Vasilívna Nemtsevich[1]; Anatolyi Vasilívich Boltushkin[1]; [1]Academy of Sciences, P. Brovki, 17, Minsk 220072 Belarus

Growth processes,structure peculiarities and magnetic properties of electrodeposited magnetic nanostructures have been investigated by means of EM, XRD, AFM, VSM and AGFM. Regularities of content modulated FeCu and CoCu nanophase particles formation in the pores of aluminium anodic oxide and a mechanism of nanocrystalline structure formation in continuous Co based films are discussed as well as crystallized film structure formation process peculiarities. Intergranular magnetic interaction and magnetization reversal processes in the above structures are reported through remanence and delta M curves analysis and rotational hysteresis loss and angular variations of hysteresis parameters measurements.

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Nanostructured Titanium Doped Iron Oxides Prepared by Laser Pyrolysis: F. Dumitrache[1]; *Rodica Alexandrescu*[1]; I. Morjan[1]; I. Sandu[1]; I. Soare[1]; I. Voicu[1]; E. Vasile[2]; R. Piticescu[3]; [1]National Institute for Lasers, Plasma and Radiation Physics, PO Box MG 36, R-76 900, Bucharest Romania; [2]S.C. METAV S.A, Str. Zapada Mieilor 16-18, Bucharest Romania; [3]S.C. IMNR S.A., Bd. Biruintei 102, Bucharest Romania

The chemical, magnetical and electrical properties of nanostructured oxides are extremely sensitive both to the huge surface/volume ratio of nanoparticles and to the synthesis path employed. Titania doped iron oxide nanostructures are expected to promote increased sensitivity when acting as sensing or catalyst devices. This work aims to investigate the production and characterization of titania doped iron oxides nanopowders using the IR laser pyrolysis from a gas phase mixture containing several basic components: vapors of Fe(CO)5/TiCl4, an oxidizing agent (usually air) and a sensitizer (either Sf6 or C2H4). The pyrolysis is based on the resonance between the emission line of a CO2 laser and the infrared absorption band of the sensitizer component (acting like an energy transfer agent. Several analytical methods (TEM, XRD, IR and Raman spectroscopy, chemical determinations, thermal analysis) were used for the characterization of particle composition and morphology. Titanium iron oxide doping was

achieved in the range 0.5-1.5 wt%. It was found that the initially mainly amorphous g-Fe2O3 transforms at about 245C in crystalline g-Fe2O3 and further, at about 595C, in crystalline a-Fe2O3.

Lead-Free Solders and Processing Issues Relevant to Microelectronics Packaging: Creep and History-Influenced Deformation Processes

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee

Program Organizers: J. P. Lucas, Michigan State University, Chemical Engineering and Materials Science, East Lansing, MI 48824 USA; Srini Chada, Motorola, Department APTC, Plantation, FL 33322 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Jud Ready, MicroCoating Technologies, Atlanta, GA 30341 USA; Jin Yu, KAIST, Center for Electronic Packaging Materials 305-701 Korea

Wednesday PM Room: 15B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Jin Yu, KAIST, Ctr. for Elec. Pkgg. Matls., Daejon 305-701 Korea; Jud Ready, MicroCoating Technology, Atlanta, GA 30341 USA

2:00 PM Invited
The Anomalous Creep Properties of High-Sn Solders: *John William Morris*[1]; Ho Geon Song[1]; [1]University of California, Dept. Matls. Sci., 555 Evans Hall, Berkeley, CA 94720 USA

Creep tests on solder joints of Sn-rich, Pb-free solders show anomalies in creep behavior at temperatures near room temperature. The anomalies include a strong temperature dependence of both the stress exponent and the apparent activation energy. The anomalies appear to have their source in the behavior of the Sn consitutent itself. The microstructures of these solders are, primarily, Sn, and test joint of pure Sn show the same anomalies. The present paper discusses this anomalous behavior in terms of the creep behavior of pure Sn and the microstructures of the solder joints.

2:25 PM
The Constitutive Creep Equation of Eutectic Sn-Ag Alloy Using the Modified Theta Projection Concept: *Yoshiharu Kariya*[1]; Masahisa Otsuka[2]; William J. Plumbridge[3]; [1]National Institute for Materials Science, Eco-Matls. Ctr., Namiki 1-1, Tsukuba, Ibaraki 3050044 Japan; [2]Shibaura Institute of Technology, Matls. Eng. & Sci. Dept., Shibaura 3-9-14, Minato-Ku, Tokyo 1088548 Japan; [3]The Open University, Matls. Eng. Dept., Walton Hall, Milton Keynes MK7 6AA UK

Creep of solder alloys is generally analyzed by relating the steady state strain rate to the stress through a power law relation such as the Norton equation. However, the power law relations may not be suitable to describe the strain-time relationship of solders, as the creep of most of solder alloys consists of only primary and accelerating stages. In this study, creep data of eutectic tin-silver alloy at temperatures between 298K and 398K has been analyzed using the modified theta projection concept instead of the steady state creep constitutive equation. The constitutive equation is composed of one rate constant representing the reciprocal of relaxation time and two different strain factors representing work hardening and weakening. The equation well describes the creep curves of the eutectic tin-silver alloy up to tertiary stage. The advantages of the method, and the stress and temperature dependency of the materials constants will be presented.

2:45 PM
Creep Behavior of Sn-Ag/Cu Solder Joints at Small Length Scale: *Matthew Kerr*[1]; Jason J. Williams[1]; Nikilesh Chawla[1]; [1]Arizona State University, Cheml. & Matls. Eng., Mechl. Behaviors of Matls. Fac., Tempe, AZ 85287-6006 USA

In order to adequately characterize the behavior of solder balls in electronic devices, the mechanical behavior of solder joints needs to be studied at small-length scales. This study examined the creep behavior of Sn-Ag/Cu solder joints at room temperature and elevated temperatures. Lap shear testing of single solder ball joints was conducted using a microforce testing system. Microstructure characterization and fractography were conducted to elucidate the governing creep mechanisms. A change in the creep exponent with increasing stress was observed and will also be discussed. Research supported by the National Science Foundation (Program Manager: Dr. K. L. Murty, contract# DMR-0092530).

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Effects of Surface Finishes on the Creep and Low Cycle Fatigue Characteristics of Sn Based Solder Joints: S. W. Shin[1]; *K. O. Lee*[1]; Jin Yu[1]; [1]Korea Advanced Institute of Science and Technology, Ctr. of Elect. Pkgg. Matls., 373-1 Gusong-dong, Yusong-gu, Daejon 305-701 S. Korea

According to Kang et.al.[1], different surface finish layers on PCB laminates gave different dissolution rate of the surface finish layers and varying thickness of intermetallic compounds at the soldering interface, which ultimately affect the microhardness in the BGA solder joints and possibly their reliability. In this study, effects of surface finish layers on the creep and low cycle fatigue resistance of Sn-based solder joints have been investigated. Lead-free Sn-3.5Ag-XCu(X=0 and 0.75), Sn-3.5Ag-2.5Bi and Sn-0.7Cu alloys were prepared as solder balls, and two types of surface finishes including combinations of Cu and Au/Ni/Cu on opposite sides of the BGA balls were investigated. Firstly, single lap shear creep tests were conducted at 373K under 5-11Mpa, and then lap shear fatigue tests were done under controlled total displacement strokes, 10, 12, 15 and 20 μm at room temperature. Microstructures of reflowed solder balls showed dense dendritic structures similar to those of solder balls with the quenching rate of 145°C/sec. Shear strain rates of lap shear specimens were typically 2-3 orders lower than those of bulky specimens and affected by the surface finish conditions of the PCB laminates. Usually, Sn-3.5Ag and Sn-3.5Ag-0.75Cu alloys showed best creep resistance, while Sn-3.5Ag-0.75Cu, Sn-3.5Ag and Sn-0.7Cu alloys showed longest fatigue life. Bi containing alloys showed the worst fatigue resistance due to the fatigue propagation along the intermetallic/solder interfaces. Another points addressed in the paper include the effects of solder ball size on the microstructure of solder balls and mechanical reliability. [1]S.K. Kang, W.K. Choi, D.Y. Shih, P. Lauro, D.W. Henderson, T. Gosslin and D.N. Leonard, 52nd ECTC Proc. P.146(2002).

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Microstructural Effect on the Creep Behaviors of a Lead Free Sn-3.5%Ag Alloy and its Joint to Cu Wire: *Kazuya Miyahara*[1]; [1]Nagoya University, Dept. of Molecular Design & Eng., Furoh-cho, Chikusaku-Ku, Nagoya, Aichi-Plef. 464-8603 Japan

Three kinds of microstructure of a lead free Sn-3.5%Ag alloy are obtained by altering the cooling rate of its ingot cast into iron mold and the effect of these microstructure on the creep behaviors of the alloy have been investigated. Rapidly cooled material (cooling rate: 8.0 C/sec) showed the finest microstructure, which is composed of the particles of Ag3Sn and Sn matrix, and also had the largest creep strength. Microstructure of the material cooled at the lowest rate (0.016 C/sec) is composed of the plate like phase of Ag3Sn and Sn matrix and this indicated the smallest creep strength which is smaller by about two orders of magnitude than that of the rapidly cooled material. The material cooled at the medium rate (2.0 C/sec) indicated a medium creep strength among these three materials. The effect of microstructure on the creep behaviors of the solder joint part of Sn-3.5%Ag alloy and Cu wire were also investigated.

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Resonant Vibration Behavior of Lead-Free Solders: *Jenn Ming Song*[1]; Truan Sheng Lui[1]; Li Hui Chen[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Eng., Tainan 701 Taiwan

Failure of solder joints may occur due to vibration, particularly when local or general resonance happens. Therefore, the vibration fracture resistance should be taken into consideration in the development of lead-free solders. This study investigated the resonant-vibration characteristics of some potential lead-free solders, including Sn-Zn, Sn-Ag and Sn-Bi alloys. The effects of the third element were also examined. Results show that Sn-Ag eutectic alloy exhibits a higher vibration life than Sn-Zn and Sn-Bi under a fixed vibration force. This is closely related to the damping capacity and crack propagation resistance of the materials. Also, the striated deformation in Sn-rich phase plays an important role in absorbing vibration energy. The morphology and distribution of the compounds also affect the vibration behavior significantly. Moreover, microstructural modification can be achieved through doping with the third element and thus the damping capacity and vibration fracture resistance can be improved.

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Effects of Copper Content on Vibration Fracture Behavior of Sn-Ag-Cu Solders: Fang I. Li[1]; *Jenn Ming Song*[1]; Truan Sheng Lui[1]; Li Hui Chen[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Eng., Tainan 701 Taiwan

Considering applications under vibrating conditions, the vibration fracture characteristics of solder materials are worthy of notice to improve reliability. This study investigated the resonant vibration fatigue of Sn-3.5Ag-Cu alloys, which have been developed as a potential lead-free solder, especially in the copper content effect. The copper content of the samples ranges from 0wt% to 1.5wt%. Results show that the vibration life of the test samples decreases in turn from the 0Cu, 0.5Cu to 1.0Cu sample. This can be attributed to refined Sn-rich dendrites which lead to a lower damping capacity and higher deflection amplitude thus produced. Itís significant to note that the 1.5Cu sample possesses the highest damping capacity and vibration fracture resistance. The presence of numerous massive Cu6Sn5 dispersoids may account for this phenomenon.

4:40 PM
Effects of Cooling Rate on Tensile and Creep Behavior of a Sn-3.5Ag Lead-Free Solder: *Felipe A. Ochoa*[1]; Jason J. Williams[1]; Nikhilesh Chawla[1]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA

The mechanical behavior and microstructure of bulk, pure Sn-3.5 Ag solders as a function of cooling rate was studied. Creep tests were performed at constant stress at ambient temperature, 60°, 90°C and 120°C. Cooling rate was found to significantly affect the secondary dendrite arm spacing of the Sn-rich phase and the aspect ratio of Ag_3Sn. The changes in the microstructure induced by the cooling rate significantly affected the mechanical behavior of the solder. Scanning and transmission electron microscopy were used to characterize the effects of cooling rate on solder microstructure, as well as to understand creep deformation mechanisms in the solder. Creep mechanisms will be presented and discussed in terms of the creep stress exponent and the solder microstructure. Research supported by the National Science Foundation (Program Manager: Dr. K.L. Murty, contact# DMR-0092530).

5:00 PM
Reliability Investigation on Lead-Free Chip Scale Packages: *Shyh-Rong Tzan*[1]; Chien-Yu Hsu[1]; [1]Materials Research Laboratories, ITRI, Metallic Devices for Elect. Lab., 195-5 Chung-Hsing Rd., Sec. 4, Chutung, Hsinchu 310 Taiwan

Environmental friendly production has become worldwide trend recently. The importance of the lead-free soldering technologies has gathered the attention of the organizations, industries, and regulators. The alternative materials and the possible risk of the reliability problems for the lead-free products have been a major concern for the industries. In this paper, one of the major considerations, the reliability issue has been investigated by evaluating the material properties and the soldering performance. A set of chip scale packages will be focus and discussed. The thermal cycle and mechanical cycling tests have been used to examine the reliability performance of a few types of lead-free solder paste as well as the tin-lead solder paste. The thermal aging and the intermetallic effects are studied to characterize the properties of the lead-free solder pastes and packages. The weibull distribution model is also used to evaluate the lead-free soldering technologies.

5:20 PM
Electromigration Studies on SnCu Flip Chip Solder Joint: *C. Y. Liu*[1]; H. T. Chiew[1]; C. J. Lu[1]; [1]National Central University, Cheml. & Matls. Eng., No. 300, Jung-da Rd., Jung-li City, Taoyuan 320 Taiwan

Electromigration is one of most urgent issues for flip chip technologies. The phenomenon of electromigration in SnPb and SnAg Pb-free solder has been reported previously by K. N. Tu et al.. Besides SnAg3.5, SnCu0.7 is another potential Pb-free candidate for C4 flip chip solder bump. Alloying effect of Cu in Al interconnect line is a well-known phenomenon. Small Cu content greatly enhanced the lifetime of Al conductive line. To understand the Cu alloying effect on Sn-based solders, we investigated electromigration behaviors on four SnCu solder alloys, which are pure Sn, SnCu0.2, SnCu0.7, and SnCu1. The test structure is in the form of C4 flip chip bump. The stressing current density is 10-5 A/cm2. In this paper, void and hillock formation will be presented and possible mechanism of Cu alloying effect will be proposed during this talk.

Magnesium Technology 2003: Magnesium Alloy Development-Mechanical Properties - II and Magnesium Wrought Alloys

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

Wednesday PM Room: 2
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Jiang Feng Nie, Monash University, Sch. of Physics & Matls. Eng., Victoria 3800 Australia; Darryl L. Albright, Hydro Magnesium, Mg. Market Dvpt., Livonia, MI 48152 USA

2:00 PM
AMC-SC1: An Elevated Temperature Magnesium Alloy Suitable for Precision Sand Casting of Powertrain Components: Colleen Joyce Bettles[1]; Christopher Forwood[1]; David StJohn[2]; Malcolm Frost[3]; Darryl Jones[1]; Ma Qian[2]; Guangling Song[2]; John Griffiths[1]; Jian Feng Nie[4]; [1]CAST (CSIRO Manufacturing and Infrastructure Technology), PB 33, Clayton S. MDC, Victoria 3169 Australia; [2]University of Queensland, CRC Cast Metals Mfg., UDP No. 55, St. Lucia, Queensland 4072 Australia; [3]Australian Magnesium Corporation, PO Box 1364BC, Milton, Queensland 4064 Australia; [4]Monash University, CAST (Sch. of Physics & Matls. Eng.), Clayton, Victoria 3169 Australia

AMC and the Australian Cooperative Research Centre CAST (Centre of Alloy solidification Technology), with the support of VAW and AVL, have developed a new sand cast alloy suitable for engine block applications. The creep and bolt load retention properties of this alloy are comparable to those of Al 319. The alloy requires similar casting temperatures to Al 319 and a T6 heat treatment, and therefore no significant new process technology is required to cast this alloy. Parallel development work on production costs has been successful indicating that AMC-SC1 is an economically viable magnesium alloy. The mechanical properties of the new alloy are presented, and discussed in relation to the properties required for a specific engine block design.

2:20 PM
Magnesium for Crashworthy Components: *Trevor B. Abbott*[1]; M. Easton[1]; R. Schmidt[1]; [1]Monash University, CRC for Cast Metals Mfg., Sch. of Physics & Matls. Eng., Wellington Rd., Clayton, Victoria 3168 Australia

With the emergence of new materials in automotive applications there is a tendency among design engineers to be cautious until the properties of the new materials are fully characterised. Often the properties which receive the greatest attention are those where the current materials have some deficiencies. These deficiencies do not necessarily carry over to the new material. A case in point is strain rate sensitivity for crash worthiness of steel and magnesium. Low carbon steels exhibit a distinct yield point during deformation, usually followed by a drop in load followed by work hardening. As the strain rate is increased, the yield point tends to increase such that it exceeds the post yielding ultimate tensile strength. In this situation there is no effective work hardening, resulting in the localisation of deformation such that only a small part of the component participates in energy absorption. From their past experiences in steel, design engineers are keen to understand the high strain rate properties when deciding whether to design a component in magnesium. The work described in this paper shows that magnesium does indeed show a strain rate dependence, and that the degree of sensitivity is highly dependant on the aluminium content of the alloy. However, the nature of the strain rate sensitivity is quite different to steel and appears to be beneficial to energy absorption. This paper discusses how these beneficial properties can be applied to the design of efficient energy absorbing components.

2:40 PM
Magnesium Wrought Alloys for Automotive Applications: *R. Gradinger*[1]; P. Stolfig[2]; [1]ARC Leichtmetallkompetenzzentrum, Ranshofen GmbH Austria; [2]Stolfig GmbH, Geisenfeld Germany

For about ten years magnesium alloys have gathered strong interest by the automotive industry resulting from new legal and market requirements. While pressure die casting soon found its applications, wrought alloys suffered from high costs and limited supply. However, parallel to activities to improve the production of sheets and extru-

sions, Stolfig GmbH started feasibility studies on new applications: material tests in the range of room temperature to 250∞C, extrusion of thin walled hollow profiles and deep drawing of sheets as well as Nd:YAG laser welding were performed in collaboration with the Light Metal Competence Center Ranshofen (LKR). The aim of the authors was to develop and test components for automotive usage. This efforts finally resulted in the delivery of prototype parts to Volkswagen. Their record-breaking 1-litre-car was equipped with space frame parts and interior paneling made of wrought magnesium alloys by Stolfig GmbH.

3:00 PM

The Effect of Temperature and Strain Rate on the Tensile Properties of Textured Magnesuim Alloy AZ31B Sheet: Ozgur Duygulu[1]; *Sean R. Agnew*[1]; [1]University of Virginia, Matls. Sci. & Eng., 116 Engineerís Way, Charlottesville, VA 22904-4745 USA

One problem facing the application of wrought magnesium alloys is their poor cold-forming properties. Historically, this has been linked to an inadequate number of independent slip systems, however, our recent research has suggested that 5 independent slip systems do participate in plastic deformation albeit with very different critical resolved shear stresses and hardening behavior. This understanding has been obtained by measuring stress-strain behavior and deformation texture evolution under various straining paths, as well as modeling of the same using a polycrystal plasticity simulation code known as the viscoplastic self-consistent (VPSC) model. The objective of the current research is to develop a fundamental understanding of the formability problem with a view towards improvement. It is well known that magnesium alloys can exhibit excellent forming characteristics at elevated temperatures (>180ÎC). Thus, the temperature dependence of strain hardening, strain rate sensitivity, and plastic anisotropy of commercial AZ31B sheet will be presented.

3:20 PM Break

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Plasma Arc Lamp Processing of Magnesium Alloy Sheet: *Joe A. Horton*[1]; Craig A. Blue[2]; Sean R. Agnew[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, MS6115, PO Box 2008, Oak Ridge, TN 37831-6115 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg 4508-MS6083, PO Box 2008, Oak Ridge, TN 37831-6083 USA; [3]University of Virginia, Dept. of Matls. Sci., 116 Engineerís Way, PO Box 400745, Charlottesville, VA 22904-4745 USA

Plasma arc lamp processing of magnesium alloy sheet has the potential of reducing the number of processing steps involved in sheet production especially when coupled with a direct casting method. The high density infrared plasma arc lamp has continuously variable power densities up to 3.5 kW/cm2. Preliminary experiments on wrought commercial sheet, AZ31b, resulted in complete recrystallization with a uniform grain size through the thickness of the sheet. Incorporation of a pinch roller attached to and following the lamp will add to the versatility of this process by allowing for further cooling rate control and improved surface quality. Mechanical properties and microstructures will be presented as a function of the process variables on both wrought sheet and on arc lamp treated as-cast sheets.

3:55 PM

Indirect Extrusion of AZ31 and AZ61: *Klaus Mueller*[1]; [1]Technical University of Berlin, Extrusion R&D Ctr., Gustav-Meyer-Allee 25, Berlin 13355 Germany

The paper describes the results of indirect extrusion of the magnesium alloys AZ31 and AZ61. The extrusion trials were carried out on the 8 MN horizontal extrusion press of the Extrusion R&D Center (ERDC) of the Technical University Berlin, Germany. Different shape of product cross sections with nearly the same extrusion ratio were extruded at a billet temperature of about 300°C with different extrusion speeds and cooling rates. Since the extrusion press is equipped with load cells comparisons were made in necessary extrusion forces. The extrudates were tested for their mechanical properties such as compression/tensile strength, yield strength and fracture elongation. Texture analysis, hardness measurements and optical microscopy for grain size measurement were done at the Institute for Material Science and Technology, Technical University Berlin. The purpose is to determine the extrudability of AZ alloys and the application of the extrudates in space frame structure for automobile constructions.

4:15 PM

Influence of the Thermomechanical Treatment on the Microstructure of Magnesium Alloy AZ31: *Jan Bohlen*[1]; A. Hortsmann[1]; F. Kaiser[1]; A. Styczynski[2]; Dietmar Letzig[1]; Karl Ulrich Kainer[1]; [1]GKSS Research Centre, Ctr. for Mg Tech., Max-Planck-Strasse 1, Geesthacht 21502 Germany; [2]Technical University Hamburg-Harburg TUHH, Eiflendorfer Str. 42, Hamburg 21073 Germany

Magnesium wrought alloys offer a large potential for structural applications due to improved mechanical properties and microstructural homogeneity compared to thin-walled casted magnesium parts. Generally, the process parameters during rolling or extrusion are dependent on the formability of the used material as well as on the dynamic and static recrystallisation. The treatment itself leads to a microstructure that has a significant influence on mechanical properties. However, in the case of magnesium and its alloys the basic mechanisms of this influence are not well understood today. Two basic approaches are derived for the analysis of magnesium wrought alloys. One is the direct analysis of the recrystallisation behaviour during the wrought process. The other one is the determination of mechanical property dependences on the microstructure of rolled magnesium or extruded profiles. Results are shown using the grain size and their homogeneity as well as the precipitation distribution as a measure for the micro structure and mechanical tests for the determination of material properties. The dependences for the process parameters of magnesium wrought is shown in order to optimise mechanical properties.

4:35 PM

Low Temperature Super-Plasticity Response of Severe Plastic Deformed Az31b Magnesium Alloy: *Adi Ben-Artzy*[1]; Avigdor Shtechman[1]; Arie Bussiba[2]; Jacob Salah[1]; Sigalit Ifergan[2]; Moshe Kupiec[2]; Ronen Grinfeld[3]; [1]Rotem Industries, Ltd., Metal Forming Grp., PO Box 842, Metar 85025 Israel; [2]Rotem Industries, Ltd., Mechl. Characteriz. Grp., PO Box 9001, Beer-Sheva 84190 Israel; [3]Ben-Gurion University, Matl. Sci. Dept., PO Box 653, Beer-Sheva 84105 Israel

Commonly, super-plasticity phenomena occurred at low strain rates that applied at elevated temperature in materials with fine grain microstructure. Recently, several efforts have been devoted to study of the unusual super-plastic behavior of sever plastic deformation (SPD) at different parameters, motivated by practical and economical considerations. With this context, super-plasticity deformation in this metallurgical state (SPD) may be achieved at low temperatures with relatively high strain rates. Among other materials, Magnesium alloys exhibits this unique deformation mode mainly due to their tendency to dynamic recovery and recrystallization processes. Alloying elements as Zr stabilizes fine grain size of ZK60, which shows super-plastic behavior at wide strain rates and at relatively low temperature range. In the current research, (SPD) has been applied to as cast AZ31B alloy (200 µm average grain size) using Equal Channel Angular Extrusion (ECAE) technique resulted in 5-10 µm average grain size. The super-plastic response of this alloy was characterized by means of elevated temperature tensile test, using round specimens, at low temperature range with various strain rates. The grain refinement microstructure obtained by ECAE was analyzed using modern image analysis method. The results as reflected by the super-plastic curves (elongation, stress versus strain rate), were found sensitive to the number of passes but less sensitive to the successive rotations by 90° of the sample between the passes. Comparison between the super-plastic response of ECAE fine grain size AZ31B to the same alloy which was grain refined by conventionally hot extruded process, indicated that the maximum elongation of the former specimens was shifted to higher strain rate by magnitude order of two. This dramatic finding, point out the great influence of the ECAE in changing conditions of super-plastic behavior.

Materials Lifetime Science and Engineering - IV

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS)

Program Organizers: Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Raymond A. Buchanan, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; D. Gary Harlow, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015-3085 USA; Dwaine L. Klarstrom, Haynes International, Inc., Kokomo, IN 46904-9013 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Robert P. Wei, Lehigh University, Mechanical Engineering and Mechanics, Bethlehem, PA 18015 USA

Wednesday PM Room: 18
March 5, 2003 Location: San Diego Convention Center

Session Chairs: D. Gary Harlow, Lehigh University, Mechl. Eng. & Mech., Bethlehem, PA 18015-3085 USA; Kwai S. Chan, Southwest Research Institute, Matls. Eng. Dept., San Antonio, TX 78228-0510 USA

2:00 PM Invited

Life Prediction Strategies for Land-Based Gas Turbine Blades: *Kwai S. Chan*[1]; N. Sastry Cheruvu[1]; Gerald R. Leverant[1]; R. Viswanathan[2]; [1]Southwest Research Institute, Matls. Eng. Dept., 6220 Culebra Rd., PO Drawer 28510, San Antonio, TX 78228-0510 USA; [2]EPRI, 3412 Hillview Ave., PO Box 10412, Palo Alto, CA 94303 USA

A wide range of coatings including overlay, diffusion, duplex, and thermal barrier coatings, are used on the first-stage blades of land-based gas turbine machines. Life-limiting mechanisms of the first stage blades include cyclic oxidation, thermomechanical fatigue, and among others, hot corrosion. In this overview paper, the development of a science-based methodology for lifing coated first-stage blades is presented. The potential failure mechanisms in coated gas turbine blades are summarized. The current status of a science-based life prediction methodology, which is implemented into a computer software named COATLIFE, for treating cyclic oxidation and thermomechanical fatigue is then highlighted. The scientific bases of COATLIFE are described. The technical capabilities and accuracy of COATLIFE are illustrated by comparisons against laboratory data and field experience. Extension of COATLIFE to predict the service life of TBC-coated blades will also be discussed. Works supported by Electric Power Research Institute and Department of Energy through Contract No. DE-FC26-01NT41321, Mr. Norman T. Holcombe, Program Manager.

2:30 PM Invited

Analysis of Environmental Effects in the Unified Approach: *Asuri K. Vasudevan*[1]; Kuntimaddi Sadananda[2]; Roland L. Hotlz[2]; I. W. Kang[3]; [1]Office of Naval Research, 800N Quincy St., Arlington, VA 22217 USA; [2]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 20375-0001 USA; [3]Defense Quality Assurance Agency of Seoul, Korea and Naval Research Laboratory, Washington, DC 20375 USA

In the Unified Approach fatigue crack growth rates are analyzed using two-parameter approach involving amplitude *K and peak stress intensity, Kmax. By considering these two parameters it is shown that one can analyze most of the fatigue phenomena without the need of crack closure concept. Effects of environment naturally manifest through the Kmax driving force. These effects are dominant at low *K values or slow crack growth rates. Hence they play a significant role in determining the total fatigue life of a component. A systematic analysis of the effects of superposition of environmental effects on fatigue crack growth is made and the behavior is classified. A detailed discussion will be presented taking examples from the literature.

3:00 PM Invited

Fatigue Crack Growth of Discontinuously Reinforced Aluminum (DRA): *Vasudevan V. Ganesh*[1]; Jason Williams[1]; Nikhilesh Chawla[1]; Asuri Vasudevan[2]; [1]Arizona State University, Dept. of Cheml. & Matls. Eng., Tempe, AZ 85287-6006 USA; [2]Office of Naval Research, Arlington, VA 22217-5660 USA

Discontinuously reinforced aluminum (DRA) is emerging as a lightweight, high performance alternative to monolithic aluminum alloys. Developing a framework for fatigue lifetime prediction of these heterogeneous materials requires a fundamental, microstructure-based understanding of fatigue crack growth. In situ fatigue crack growth studies were conducted using a QuestarTM traveling telescope. Fatigue crack growth studies were conducted at varying R-ratios and the behavior analyzed using the two-parameter approach that relates DKth to Kmax, proposed by Vasudevan et al.1 The interaction between the fatigue crack and reinforcement particles or particle clusters was quantified. Preliminary microstructure-based modeling to understand and predict the effect of particle clusters on fatigue crack rate and stress intensity at the crack tip will be discussed. 1A.K. Vasudevan, K. Sadananda, and N. Louat, Mater. Sci. Eng., (1994) A188 1. Research sponsored by the Office of Naval Research under contract #N00014-01-1-0694.

3:30 PM

Fatigue Fracture Mechanism and Fatigue Life Assessment of Aluminum Castings: *Qigui Wang*[1]; [1]General Motors Corporation, CDVC-Powertrain, 1629 N. Washington Ave., Saginaw, MI 48605 USA

The increasing application of shape-cast aluminum components in both automotive and aerospace industries has drawn great concern in fatigue properties of aluminum castings. In order to enhance the fatigue properties of aluminum castings and to increase the reliability and wider use of aluminum castings in cyclic loading applications, a better understanding of fatigue fracture mechanism that presents in aluminum castings and the factors affecting the fatigue crack initiation and propagation is absolutely necessary. For this propose, a critical review and case study has been carried out to address the fatigue fracture mechanism, the factors affecting the fatigue properties and fatigue life assessment methods and eventually to point out further challenges for the improvement and accurate prediction of aluminum fatigue.

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Fatigue and Fretting Fatigue of Biomaterial, Ti-29Nb-13Ta-4.6Zr, in Air and Simulated Body Environment: *Mitsuo Niinomi*[1]; Toshikazu Akahori[1]; Keita Ishimizu[1]; [1]Toyohashi University of Technology, Dept. of Production Sys. Eng., 1-1, Hibarigaoka, Tempaku-cho, Toyohashi 441-8580 Japan

The fatigue and fretting fatigue characteristics of the newly developed biomaterial, Ti-29Nb-13Ta-4.6Zr, were investigated in both air and simulated body environment, Ringerís solution. The fatigue strength of Ti-29Nb-13Ta-4.6Zr is not degraded in Ringerís solution. The fretting fatigue damage is well correlated to the Youngís modulus. The fretting fatigue strength of Ti-29Nb-13Ta-4.6Zr is greater in Ringerís solution than in air in the low cycle fretting fatigue life region, but smaller in Ringerís solution than in air in the high cycle fretting fatigue life region. The effect of lubrication due to Ringerís solution is greater in the low cycle fretting fatigue life region, but the effect of corrosion is greater in the high cycle fretting fatigue life region.

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Effect of Alpha Grain Size on Low-Temperature Fatigue Property of Ti-5%Al-2.5%Sn ELI Alloy: *Yoshinori Ono*[1]; Tetsumi Yuri[1]; Hideshi Sumiyoshi[1]; Saburo Matsuoka[1]; Toshio Ogata[1]; [1]National Institute for Materials Science, Matls. Info. Tech. Sta., 1-2-1, Sengen, Tsukuba, Ibakaki 305-0047 Japan

The effect of alpha grain size on low-temperature fatigue property has been investigated in Ti-5%Al-2.5%Sn ELI alloy used for fuel turbo pumps of Japanese domestic space rocket. Grain size of specimens was controlled to be about 30 micron or 80 micron. Tensile strengths of both specimens are almost same and are increased with a decrease of temperature. However, low-temperature fatigue property is quite different between both specimens. In the specimen with 30 micron grains, 106 cycles fatigue strength at 4K and 77K is 1.6 and 1.5 times higher than that at 293K, respectively. On the other hand, in the specimen with 80 micron grains, 106 cycles fatigue strength at 4K and 77K gets lower to the same level as that at 293K. Hence, it is concluded that refinement of alpha grains plays a very important role to obtain the good low-temperature fatigue property for Ti-5%Al-2.5%Sn ELI alloy.

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Re-Consideration of Fatigue Crack Closure Effect in the Lifetime Prediction of Structural Materials: *Daolun Chen*[1]; [1]Ryerson University, Dept. of Mechl., Aeros. & Industl. Eng., 350 Victoria St., Toronto, Ontario M5B 2K3 Canada

The integrity of some engineering structures, e.g., riveted and welded components, depends predominantly on the lifetime spent in the crack growth, resulting in the development of a defect-tolerant approach. This approach relies on an integration of the crack growth expression, based upon an accurate description of the effective driving force by considering fatigue crack closure effect. However, many researchers have questioned the validity of the conventional crack closure concept. In view of the discrepancies and confusions reported in the literature, we have re-modeled and re-defined the fatigue crack closure effect by taking into account the role of the lower portion of fatigue cycles below the opening point, so that this concept can be used to predict better the fatigue life of materials/components. The aim of this paper is to review the fatigue crack closure effect and to present further the experimental evidence on the modified crack closure definition.

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Characterization of Fatigue Crack Origins in 2024-T3 Aluminum Alloys: *Jonathan Tsang*[1]; Ali Merati[1]; [1]Institute for Aerospace Research Structures, Materials, and Propulsion Laboratories, Bldg. M-13, 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada

There is a high probability of interaction between corrosion and fatigue and the probability increases as aircraft age. New analytical tools and procedures for structural integrity analysis are based on a holistic life assessment that uses the Initial Discontinuity State (IDS) of the material, as the initial condition for crack growth analysis. NRC-IAR is participating in a collaborative research and development project with the objective of producing a reliable corrosion/fatigue life prediction methodology. The work is part of the USAF-Lockheed Martin Corrosion Fatigue Structural Demonstration (CSFD) project. This extensive study has been carried to physically measure the IDS for pristine 2024-T3 aluminum alloys. The type of discontinuity being characterized in this study is limited to constituent particles. The

overall IDS data were measured by metallographic sectioning as well as fractography of fatigue coupons. The two distinctive controlling factors responsible for crack nucleation will be discussed. It was found that for bare 2024-T3 materials, the constituent particles were the main microstructural discontinuity origination the cracks. Comparatively, the fatigue crack origins in clad 2024-T3 samples were located at the surface of the clad. No constituent particles were found to be associated with crack nucleation. Multi-nucleation sites were observed in this material. This paper summarizes the result of tests for the clad and bare 2024-T3 aluminum alloys.

Materials Processing Fundamentals: Thermodynamics and Reaction Kinetics

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee, EPD-Process Fundamentals Committee

Program Organizers: Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA; Princewill N. Anyalebechi, Grand Valley State University, L. V. Eberhard Center, Grand Rapids, MI 49504-6495 USA

Wednesday PM Room: 1A
March 5, 2003 Location: San Diego Convention Center

Session Chair: Seshadri Seetharaman, Royal Institute of Technology, Div. of Metall. Dept. of Matls. Sci. & Eng., Stockholm SE-100 44 Sweden

2:00 PM

Thermodynamic Studies of FeO-Containing Slag Systems: *Patrik Fredriksson*[1]; Seshadri Seetharaman[1]; [1]Royal Institute of Technology, Div. of Metall., Dept. of Matls. Sci. & Eng., Brinellvagen 23, Stockholm SE-100 44 Sweden

Thermodynamic data concerning FeO-containing slags is of importance in ladle refining of steel. With a view to generate a set of reliable and self-consistent thermodynamic data for these slags, experimental determination of the activities of FeO in binary and ternary slags was carried out using the gas equilibration method involving CO-CO2-Ar gas mixtures at steelmaking temperatures. The slag samples in Pt crucibles were quenched after the equilibration and subjected to chemical analysis. The thermodynamic activities of ìFeOî in the slags were calculated from the experimental data. The results are incorporated into a thermodynamic description of silicate melts in the present laboratory. The model is based on a Temkin-Lumsden approach and is able to compute the thermodynamics of higher order systems from the lower order ones.

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Kinetics of Chlorination and Oxychlorination of Hematite: *N. Kanari*[1]; D. Mishra[1]; I. Gaballah[1]; [1]Laboratoire Environnement et Mineralurgie, Rue du Doyen M. Roubault, BP 40, Vandoeuvre, Cedex 54501 France

Kinetics of Fe2O3 reactions with Cl2 and Cl2+O2 gas mixtures were investigated up to 1025 C using thermogravimetric technique. Effects of gas flow rate, temperature and partial pressure of Cl2, O2 and Cl2+O2 on the reaction rate were evaluated. Data were processed to predict the reaction rate mechanism. The values of the apparent activation energy ëEaí of Fe2O3 chlorination were respectively 180 and 74 kJ/mol for the temperatures lower and higher than 875 C. The oxychlorination of hematite proceded with an Ea of 148 kJ/mol between 600 and 1025 C. The increase of the oxygen partial pressure in the Fe-O-Cl system led to low reaction rate of hematite chlorination. The apparent reaction orders with respect to Cl2, O2 and Cl2+O2 at 750 C were respectively 1.44, -0.61 and 0.71.

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Hydrothermal Precipitation and Thermal Transformation of Boehmites: *D. Mishra*[1]; N. Kanari[1]; I. Gaballah[1]; S. Anand[2]; R. P. Das[2]; [1]Laboratoire Environnement et Mineralurgie, Mineral Processing and Environmental Engineering Team, Rue du Doyen M. Roubault BP 40, Vandoeuvre, Cedex 54501 France; [2]Regional Research Laboratory, CSIR, Bhubaneswar, Orissa 751 013 India

Boehmites (gamma-Al2O3.xH2O) are the oxide-hydroxides of aluminum and find extensive use for manufacture of high surface area catalysts, coatings, aluminum and alumina derived materials. The present paper describes a novel hydrothermal precipitation route to obtain high surface area boehmites and their thermal transformation sequences to alpha-Al2O3. Hydrothermal precipitations were carried out in autoclave with inorganic aluminum salt solutions [Al2(SO4)3, AlCl3, Al(NO3)2] and urea as the neutralizing agent. Effects of residence time and temperature on the crystallization extent of boehmites obtained either from Al2(SO4)3 had a lower water content and a higher surface area than the boehmites obtained either from AlCl3 or Al(NO3)2. Calcination of these boehmites between 450-550 C produced gamma-Al2O3 of high surface area (280-330 m2/g). This compound was converted into alpha-Al2O3 at about 1400 C.

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A Novel Method for Extraction of TiO2 from Ilmenite Ores: Vilas D. Tathavadkar[1]; *Animesh Jha*[1]; [1]University of Leeds, Inst. of Matls. Rsrch., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK

Three different types of processes, namely the sulfate process, the chloride process, and the chloride-ilmenite process, are used worldwide for production of titanium dioxide. Of these three methods of production, the chloride process is more popular and is therefore widely used. The sulfate process creates large amounts of dilute acid effluent, whereas the chloride route yields a hazardous waste than the sulphate process. However, the volume produced is much smaller. For example, one-ton production of titanium dioxide generates 12 tons of wastes material from the sulfate process while the chloride process generates only 4 tons wastes. Iron chloride, generated in the chloride route is acidic and hazardous. We have investigated a novel route for the separation of titanium dioxide from ilmenite ores. In this process, ilmenite ore is roasted with alkali carbonate in the temperature range of 800 to 1100°C to form alkali titanates. The roasted mass was leached with water and then with dilute acid solution to remove the impurities. The physical and chemical properties and phase equilibria of roasting reactions were investigated. The results of isothermal roasting, water and acid leaching experiments are discussed in details in this paper. Nearly 95% pure TiO2 was obtained via this method, which can be further improved by acid leaching treatment.

3:30 PM

The Polymorph Transformations of Antimony White in Hydro-Process: *Xiao Songwen*[1]; Yan Xiaohui[2]; Xiao Xiao[3]; Zhang Duomo[4]; [1]Changsha Research Institute of Mining & Metallurgy, Changsha, Hunan 410012 China; [2]Hunan Sunrise Nanometer Material Company Ltd., Changsha, Hunan 410014 China; [3]Central South University, Changsha, Hunan 410083 China; [4]Central South of University, Col. of Metallurgl. Sci. & Eng., Changsha, Hunan China

On grounded of the theoretical models of growth unites with coordination polyhedron structure of anion, the polymorph transformation mechanisms of antimony white Sb2O3 in hydro-process is presented. The new findings has been verified in the commercial-scale test, and it shows that adding a small amount of tratrate ions is effective for transformation of antimony white in hydro-process.

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

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee

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

Wednesday PM Room: 14A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: K. Linga Murti, National Science Foundation, Arlington, VA 22230 USA; Joanna R. Groza, University of California-Davis, Cheml. Eng. Matl. Sci. Dept., Davis, CA 95616 USA

2:00 PM Invited

Real Time Measurements of the Effect of Electric Fields on Grain Growth in Nanocrystalline Silver Thin Films: *E. A. Stach*[1]; T. Freeman[1]; X. Phung[2]; L. Stanciu[2]; J. R. Groza[2]; K. Hukari[3]; R. Dannenberg[4]; [1]National Center for Electron Microscopy, Lawrence Berkeley Natl. Lab., Berkeley, CA 94720-0001 USA; [2]University of California at Davis, Dept. of Matls. Sci. & Eng., Davis, CA 95616 USA; [3]AFG Development Corporation, Petaluma, CA USA; [4]Chahaya Optronics, Fremont, CA USA

One method for creating materials with nanometer grain sizes is by sintering of nanoparticles. However, a critical step in the nanopowder consolidation process concerns the ability to achieve full densification with minimal microstructural coarsening. In this work, we have chosen to study coarsening in a model system, that of nanocrystalline silver thin films, to determine the effects of temperature and electric field on the process. We have developed a methodology that allows us to determine in real time and at high spatial resolution the effects of initial grain size, temperature and applied electric field on the grain growth characteristics. To do this, we have constructed a combined heating/electrical bias stage for a transmission electron microscope. This allows us to heat samples to temperatures in excess of 1000°C as well as apply electric fields in excess of 30 V/m to the sample. This can all be done within the objective lens of the electron microscope, allowing quantitative, real time characterization of the activation energy of grain growth and development of texture in these silver thin films. In this presentation, we will emphasize the details of this new methodology, discuss its strengths and limitations, and present quantitative data concerning the effects of applied electric fields on grain growth.

2:30 PM

Unique Facility for Materials Processing in High Temperatures and High Magnetic Fields: *Sastry V. Pamidi*[1]; Ulf P. Trociewitz[1]; Hiroshi Maeda[1]; Saleh Hayek[2]; Askar D. Sheikh-Ali[2]; Cristiane B. Bacaltchuk[2]; Marwan Elkawni[2]; Shi Shen Yan[2]; Hamid Garmestani[3]; Justin Schwartz[1]; [1]Florida State University Center for Advanced Power Systems, Natl. High Magnetic Field Lab., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA; [2]Florida A&M University & Florida State University College of Engineering, Tallahassee, FL 32310 USA; [3]Florida A&M University & Florida State University College of Engineering, Natl. High Magnetic Field Lab., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA

Processing of materials under the influence high magnetic fields has been reported to cause significant enhancements in useful properties of many materials. The mechanisms of influence of magnetic field on texture development, grain growth, and recrystallization need to be understood in order to optimize the processes and to develop commercial applications of in-field heat treatment processes. We have designed, built, and tested several furnaces for the 20 T resistive magnet at the National High Magnetic Field Laboratory. The furnaces can be used for experiments in magnetic fields up to 19 T and temperatures up to 1600C. All the furnaces are equipped with temperature programmers and gas flow systems to control the atmosphere during heat treatments. The volume in the uniform magnetic field and temperature zone is up to 60 mm in diameter and 100 mm in height to accommodate large samples. Some of our experiments focused on enhancing the texture of Bi2212 high temperature superconducting tapes and understanding r ecrystallization, grain growth, phase transformations, and solidification from the melt in NdFeB, Fe-Si, Zn-Al and terfenol-D systems. The paper describes the unique features of our facilities and some of the experiments that have been performed with the facilities.

2:50 PM

Application of High Magnetic Field to Texture Development in Zn Alloy: *Askar D. Sheikh-Ali*[1]; *Dmitri A. Molodov*[2]; Hamid Garmestani[1]; [1]National High Magnetic Field Laboratory, Lab. for Micromech. of Matls., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA; [2]Institut f¸r Metallkunde und Metallphysik, RWTH Aachen, Aachen D-52056 Germany

Highly textured Zn-1.1%Al alloy with fine-grained microstructure are annealed in a high magnetic field of 32T. The texture of the samples is characterized by the two 0002 components tilted at 15-20° from the normal to the rolling direction of the sheet. The annealing of samples parallel to the field preserves the maximum intensity of texture components and redistributes the intensity closer to the normal direction. The annealing of samples parallel to the field preserves the maximum intensity of texture components and redistributes the intensity between original orientation of 0002 components and normal direction. The obtained results are interpreted in terms of selective grain growth due to the anisotropy of (dia)magnetic susceptibility in Zn.

3:10 PM

Grain Boundary Dynamics in High Magnetic Fields: *Dmitri A. Molodov*[1]; [1]Aachen University of Technology, Inst. of Physl. Metall. & Metal Physics, Kopernikusstr. 14, Aachen 52056 Germany

The movement of grain boundaries is one of the fundamental mechanisms of microstructure evolution during heat treatment of metallic materials. Therefore, control of grain boundary motion means control of microstructure evolution, which is a key for the design of advanced materials. Grain boundary motion can be affected by a magnetic field, if the anisotropy of the magnetic susceptibility generates a gradient of the magnetic free energy. In contrast to curvature driven boundary motion, a magnetic driving force also acts on flat boundaries so that the motion of crystallographically fully defined boundaries can be investigated, and the true grain boundary mobility can be determined. By appropriate positioning and repositioning of the specimen in the magnetic field the energy gradient can be changed and even inverted for the same boundary. This allows for the first time to study asymmetry effects of boundary dynamics. We will report in detail results obtained on Bi and Zn bicrystals. Selective grain growth in deformed Zn monocrystals and texture changes in Zn alloy sheet in high magnetic fields will be discussed.

3:30 PM Break

3:45 PM

Experiments on Dynamic Behavior of Molten Metal and Solid Particles Under Direct Current Passing: *O. I. Raychenko*[1]; V. P. Popov[1]; O. V. Derevíyanko[1]; T. I. Istomina[1]; A. I. Repenko[1]; [1]IPMS, NAS of Ukraine

At electrodischarge sintering of powder mixtures by electric direct current some essential displacements of molten metal and solid particles were revealed. Such displacements at conventional sintering are not known for us. The objects were powders (mixtures): Sn-Cu (dispersivity 100-500 mkm); Sn - high-temperature alloy on nickel base or electrocorundum; coper alloy - electrocorundum (dispersivity 63-500 mkm); Ni (dispersivity 10 mkm). The electric direct current was passed in vertical direction parallel with the axis of cylindrical mould or through the series circuit steel-powder nickel layer-hard alloy. If cylindrical countainer (mould) had not the upper punch (a load on top was absent) then sample after solidification had form of a ìbulletî. This sample became convex on top and concave on bottom. At presense of pre-pressing (start pressing) on top a movement of suspension (molten matrix and solid particles) occured in the similar way. Under action of electro-magnetic forces a redistribution of solid partic les in volume of suspension occurs. Particles with more high conductivity than liquid phase begin to cluster at center. In the case of lower conductivity the motion of the particles to outer side surface of mould prevails. At current passing through series circuit steel-nickel powder layer-hard alloy three named parts of circuit were sintered into single block. After destruction at testing the interlayer had the form of a ring. This shows non-uniformity of passing electric current through interlayer. The cause of displacements of solid (non-molten) particles may be simultaneous influence of thermic convection and squeezing out by the electromagnetic Lorentz-forces, which are directed radially. The phenomena described may be applied at production of objects with gradient (variable in volume) concentration of particles added specially. This refers to products of their interaction with molten matrix as well.

4:05 PM Invited

Theory of Motion of Inclusions in a Viscous Medium Under Joint Influence of Electromagnetism and Thermocapillarity: *O. I. Raychenko*[1]; [1]IPMS, NAS of Ukraine

The motion of a fine inclusion in a viscous medium is subjected to the theoretical examination. This motion can be qualified as an elementary act of the motion of fine phase particles in mixtures: suspensions, emulsions, airsols, foamed liquid materials. The erroneous character of the theory of thermocapillary motion of inclusions, which is universally recognized now, is revealed. It is proposed to take into account the ìenergy of locationî of the inclusion in a non-uniform temperature field, in order to formulate more correctly the Stokes equation describing the velocity field inside the fluid inclusion. The solution of the set of the Stokes equations referring to a liquid particle and a surrounding medium has allowed to obtain the formula for the motion velocity of a particle under joint influence of electromagnetism and thermocapillarity. This formula is suitable for theoretical calculations of transfer processes in suspensions, emulsions and gaseous media containing various inclusions. The calculation on inclusions in molten metals (for example, in Cu, Al, Fe, Ag) shows that the exertions caused by electromagnetism and thermocapillarity are comparable.

4:25 PM
Aspects of Magnetic Field Use for Treatment of Water and Water Systems: *V. V. Honcharuk*[1]; V. V. Malyarenko[1]; [1]Ukraine National Academy of Science, Dumansky Inst. of Colloid Chem. & Chem. of Water, 42, Vernadsky Ave., Kyiv-03142 03680 Ukraine

In aqueous solutions at magnetic field (MF) induction 0,108 T and liquid velocity in cell 0,51,5 m/s the electric driving force (EDF) is approximate to it calculated. As it is elicited, the EDF cause a current for which as electrodes and as external load double the conducting inner wall of the cell. MF action on the system is analogic to electric current energize. At MF-treatment some surface electrode processes (solution or precipitation metal, gassing) cause a running renovation of the surface, that decrease of scale. At the same time composition of the solution change under effect of the electrochemical processes. After choice of the MF-treatment regime these factors can use for water demineralizing, for act on microorganisms, for change of surface charge and colloids coagulable, for galvanic solution and/or precipitating of metals.

Materials Prognosis: Integrating Damage-State Awareness and Mechanism-Based Prediction: Microscale Modeling & Simulation

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

Wednesday PM Room: 16A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: J. Wayne Jones, University of Michigan, Col. of Eng. Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; David Alan Johnson, Air Force Research Laboratory, WPAFB, OH 45433-7817 USA

2:00 PM
Micron- and Nano-Scale Mechanical Testing for Model Parameterization: *Dennis M. Dimiduk*[1]; Michael Uchic[1]; Triplicane A. Parthasarathy[2]; Yoon-Suck Choi[2]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, WPAFB, OH 45432 USA; [2]UES, Inc., Dayton, OH 45432 USA

A necessary part of achieving a materials-based prognostics capability is the development, parameterization, and validation of continuum materials behavior models. Such models should be appropriately informed from microstructural information and knowledge of mechanistic behavior of the material. Continuum crystal plasticity methods are at the forefront of techniques that incorporate first-level microstructural information. However, a deficiency in parameterizing those models lies with the inability to obtain single crystal property information from individual grains, especially when such parameters must reflect the subtleties of material process history. Our effort has sought to measure single-crystal critical resolved shear stresses and strain hardening rates from micro and nano-scale samples extracted from relevantly processed structural alloys. In this presentation we present the experimental methods, challenges associated with those methods, and early results for this new approach toward structural property assessment.

2:30 PM Invited
Modeling the Physics of Microstructure Failure in a Nickel Alloy: *Donald A. Shockey*[1]; Jeffrey W. Simons[2]; Brian D. Peterson[2]; Takao Kobayashi[2]; [1]SRI International, Ctr. for Fracture Physics, 333 Ravenswood Ave., Menlo Park, CA 94025 USA

SRI International is constructing a failure prognostic algorithm for engineering materials that attempts to overcome limitations of current continuum approaches. The algorithm is based on the physics of material failure at the grain and subgrain level in a nickel-based superalloy. The evolution of microstructure deformation and failure, as observed and measured in interrupted fatigue tests is described mathematically, quantified with data generated in the tests, and implemented into a finite element code. The algorithm is demonstrated by simulating damage developing at the tip of a fatigue crack. The model is being extended to include microcrack coalescense and enable simulations of crack propagation in a turbine disk.

2:55 PM Invited
Enhanced Damage-State Awareness Through Full-Field Measurement Techniques: *David Alan Johnson*[1]; [1]Air Force Research Laboratory, AFRL/MLLMN, Bldg. 655, Ste. 1, 2230 Tenth St., WPAFB, OH 45433-7817 USA

A key to integrating mechanism-based prediction and damage-state awareness is better understanding of the fundamental physics of damage. In order to gain this physical understanding, much more efficient means of studying actual material behavior through experimentation must be developed. One very promising area for enabling this efficient experimentation is through full-field deformation mapping accomplished with machine vision. With full-field deformation mapping, it is possible to efficiently study damage development under a wide variety of conditions and at a wide variety of spatial scales. It is also possible to efficiently calibrate and evaluate probabilistic damage models through automated and extensive sampling. An example of the use of deformation mapping in the study of the failure physics of a large-colony, fully lamellar intermetallic will be presented, along with a discussion of the many possible future uses of full-field measurement techniques in studying damage progression, both quantitatively a nd quantitatively. The future real-time integration of modeling and experimentation made possible by full-field techniques will also be discussed.

3:20 PM Break

3:50 PM
Mechanisms of Elevated Temperature Fatigue Crack Growth of Ti-6Al-4V: Tarun Goswami[1]; David S. Gelles[2]; Russell H. Jones[2]; [1]Arkansas Tech University, Mechl. Eng., Russellville, AR 72801 USA; [2]Pacific Northwest National Laboratory, Matls. Sci., PO Box 999, Richland, WA 99352 USA

The Ti-6Al-4V alloy is used as a fan disk material because of its high strength to weight ratio, toughness and fatigue strength; however, very little is known about its elevated temperature fatigue crack growth mechanism. The fatigue crack growth mechanism of Ti-6Al-4V has been evaluated with transmission electron microscopy (TEM) of fatigue crack growth specimens tested at an R of 0.1, frequency of 10 Hz at 175, 230, 290 and 345 C. TEM samples were removed at various locations along the crack wall with special care to locate the foil within the crack tip plastic zone. A major proportion of the dislocations was concentrated in a-grains with only a small fraction present in b-grains. Within the a-grains a combination of both <c> and < a> dislocations formed with some twinning. The <c> type dislocations in planar arrays could be demonstrated with g=0002 near [1210]. Within these grains, dislocation density increased as the temperature was increased. The relevance of these observations to fatigue crack growth mechanisms will be described.

4:15 PM
Integrating Damage State: From Micro-Mechanism to Macroscopic Behavior: *Xijia Wu*[1]; [1]National Research Council of Canada, Inst. for Aeros. Rsrch., M-13, 1200 Montreal Rd., Ottawa, Ontario K1A 0R6 Canada

Fatigue and creep are the two most important failure modes in a high performance gas turbine. Integrated constitutive equations are presented for gas turbine metallic materials under fatigue and creep conditions. These equations are derived from the understanding of the physical mechanisms governing deformation and crack growth in metallic materials. For example, fatigue damage is known to accumulate by restricted slip reversal, which eventually leads to a mode of transgranular crack propagation. On the other hand, the accumulation of creep damage is more complex in a polycrystalline material: it occurs by competition of grain boundary sliding, volume diffusion and intragranular dislocation mechanisms. Recognizing each physical component of deformation would provide an improved life prediction methodology and accurate feedback to the design of the alloy for better performance. A universal equation has been derived that the rate of crack propagation (under either fatigue or creep conditions) is proportional to the accumulation of plastic strain, e_p (or strain range, Delta e_p) and the plastic zone size, l_p, at the crack-tip, as expressed by the formula: da/dt (or da/dN) = e_p (or Delta e_p) . l_p. Using this type of relationships, damage accumulation and the materialís life can be evaluated through integration of the understanding of the microphysical processes. Such physically based relationships are also

useful to enhance the damage state awareness, in coupling with advanced NDI techniques.

4:40 PM Invited
Creep Life Prediction Using a Multiple-Damage State Variable Model: Application to Aluminium Alloy 2650: Alex Djakovic[1]; Hector Basoalto[1]; *Brian F. Dyson*[1]; Malcolm McLean[1]; [1]Imperial College of Science, Technology and Medicine, Matls., Prince Consort Rd., London SW7 2BP UK

A generic Continuum Damage Mechanics approach to creep of particle-strengthened alloys has been developed to account for the influence of the concurrent evolution of a number of types of damage. The approach identifies three broad categories of damage that are fully interactive through being coupled with the rate of creep deformation. The damage mechanisms considered are (a) dislocation accumulation, (b) thermal changes in particle size/distribution and (c) grain boundary cavitation. Primary creep is due to progressive load transfer from the deforming matrix to the particles as creep strain evolves and not to ìstrain hardeningî since dislocations weaken rather than strengthen the alloy in this model. The relative contributions of the different types of damage are material specific, but the formalism allows these differences to be accommodated parametrically within the model. These parameters are constrained within physically realistic limits determined theoretically or by independent experiment, rather than being fitted empirically to creep data. The model will be illustrated in relation to the creep behaviour of the Al-Cu-Mg precipitation strengthened alloy 2650-T8. The principal strengthening precipitate in this alloy is -phase. Account has been taken of its rod-like morphology in accounting for its obstacle strength; the extent of primary creep due to stress transfer from the matrix; and to the asymmetric coarsening of the rods in contributing to tertiary creep. In representing the effects of creep cavitation, it is shown that modelling the transition from unconstrained to constrained cavity growth captures the form of the minimum creep rate and rupture life as functions of stress over a range of temperatures. The multi-damage model also provides a good representation of shapes of creep curves and provides a basis for identifying the damages that have most influence on the creep behaviour under various loading conditions.

5:05 PM Invited
Crystal Rotations During Multiaxial Creep in Single Crystal Superalloys: Characterisation and Modelling: Mamoud G. Ardakani[1]; Hector Basoalto[1]; Barbara A. Shollock[1]; *Malcolm McLean*[1]; [1]Imperial College of Science, Technology and Medicine, Matls., Prince Consort Rd., London SW7 2BP UK

Life prediction procedures for single crystal superalloys, which are now state of the art for advanced gas turbine blades, are largely based on the interpretation of uniaxial creep and low cycle fatigue data. However, turbine blades in service experience significant multiaxial stresses at the blade roots and in the neighbourhood of cooling channels. It is appreciated that uniaxial stresses along non-symmetric orientations cause crystal rotations during creep. It is less well known that multiaxial stressing of symmetric single crystal superalloys can also lead to large crystal rotations that can be spatially heterogeneous. In the present study, the creep behaviour of the single crystal nickel-based superalloy CMSX-4 with <001>, <111> and <011> nominal orientations has been studied on cylindrical specimens with circumferential notches at 850∞C and net-section stresses between 600 and 850 MPa. The distribution in deformation across the specimen diameter has been characterised by mapping the changes in crystal or ientation using electron backscatter diffraction (EBSD) and comparing this with lattice rotations observed in uniaxial creep specimens. Photogrammetry experiments have also been carried out within the notch to study strain distribution. An anisotropic creep model based on a continuum damage mechanics (CDM) formalism that was developed and validated for uniaxial loading of nickel-base superalloys has been used to simulate creep in multiaxial loading in notched specimens. The predictions of crystal rotation resulting from creep deformation are compared with the experimental results.

Materials Research to Meet 21st Century Defense Needs

Sponsored by: TMS, National Materials Advisory Board, *Program Organizers:* Arul Mozhi, National Materials Advisory Board (NA-966), National Research Council, Washington, DC 20001 USA; Harvey W. Schadler, Naskayuna, NY 12309 USA

Wednesday PM Room: 5B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Robert L. Rapson, Air Force Research Laboratory, Non-Metallic Matls. Div., WPAFB, OH 45433-7750 USA; Alan M. Lovelace, General Dynamics Corporation, Commercial Launch Services, Cape Canaveral, FL 32920 USA; Arul Mozhi, National Research Council, Natl. Matls. Advisory Board, Washington, DC 20001 USA

2:00 PM Opening Remarks Robert L. Rapson

2:10 PM Invited
Overview of Defense Materials Needs and Crosscutting Research Priorities: *Harvey W. Schadler*[1]; [1]General Electric, Corporate R&D Ctr., 4024 Windsor Dr., Niskayuna, NY 12309 USA

The Department of Defense (DoD) requested the National Materials Advisory Board to study critical materials and processing research and development (R&D) required to meet 21st-century defense needs. The study identified DoD materials needs and explored R&D priorities in five classes of materials: Structural and multifunctional materials, Energy and power materials, Electronic and photonic materials, Functional organic and hybrid materials, and Bioderived and bioinspired materials. This paper integrates the R&D priorities from all five materials areas and presents the studyís R&D recommendations. The study committee recognized that realizing the revolutionary new defense capabilities that materials science and engineering offer will depend on more than just R&D; innovative management will also be needed to reduce risks in translating fundamental research into practical materials, and to promote cross-fertilization of scientific fields. This paper also discusses these issues and presents the studyís recommendation for needed innovations in management.

2:40 PM Invited
Structural and Multifunctional Materials: *Harry A. Lipsitt*[1]; [1]Wright State University, Matls. Sci. & Eng., 1414 Birch St., Yellow Springs, OH 45387 USA

The Structural and Multifunctional Materials Panel focused on emerging materials and the processes used for their fabrication, with special attention to the types of multifunctionality that could be designed into a material. This paper discusses DoD structural materials development approaches and goals. It highlights the importance of lighter, stiffer, and stronger materials, and the need for materials to operate for long periods at high temperature with predictable degradation. These materials are necessary to improve vehicle mobility, maneuverability, transportability, and survivability. The panel identified four areas of R&D opportunity. These four opportunities are expanded upon, with emphasis on design of structural materials that are truly multifunctional. Investments in these research areas should result in advances that would yield many of the necessary new DoD materials. Such advances will: reduce development time and costs, modernize design criteria, predict and verify functionality, continuously monitor in-service health, and predict residual life.

3:10 PM Invited
Energy and Power Materials: *James Baskerville*[1]; [1]General Dynamics Company, Bath Iron Works, 700 Washington St., MS 6570, Bath, ME 04530 USA

The Energy and Power Materials Panel examined advanced materials and processes in this area. DoD needs for energy and power materials are many, among them, batteries for energy storage; capacitors for storage and release of pulsed power; fuel cells for efficient direct conversion of chemical to electrical energy; photovoltaics for harvesting energy; explosives for enhanced and tailorable lethality; and microturbines for powering unmanned aerial vehicles. The panel identified key materials aspects of each major application, and derived broad themes for materials research. Areas identified were those where DoD funding would be needed due to the lack of commercial interest. Successful pursuit of these themes will provide numerous benefits to the DoD, including: reduced development time and cost; increased energy density in storage devices; improvements in lethality of muni-

tions; practical energy-harvesting devices; and reduced weight of energy and power systems, which will reduce soldier and system payload.

3:40 PM Break

3:55 PM Invited

Electronic and Photonic Materials: *Julia M. Phillips*[1]; [1]Sandia National Laboratories, Physl. & Cheml. Scis. Ctr., Organization 1100, MS 1427, PO Box 5800, Albuquerque, NM 87185-1427 USA

The Electronic and Photonic Materials Panel examined research needs for defense systems in electronics, optoelectronics, photonics, and microsystems (including sensors). The innovation of the private sector allowed the panel to consider which defense needs could be met by making use of commercial developments and which require DoD investment. The panel examined the following military needs that would benefit from electronic and photonic materials: detection, identification, and defense against or avoidance of threats; high fidelity imaging signals; communications systems; compact systems to transmit at very high power and high frequency; enemy identification and monitoring; dynamic camouflage/stealth; and health monitoring of equipment and personnel. While this panel considered a wide variety of military needs from several vantage points, ranging from individual devices or components to entire miniature systems, a number of common themes emerged that point to important areas for research. These research areas will be presented.

4:25 PM Invited

Functional Organic and Hybrid Materials: *Frank E. Karasz*[1]; [1]University of Massachusetts, Dept. of Polymer Sci. & Eng., Conte Rsrch. Ctr., Amherst, MA 01003 USA

The Panel on Functional Organic and Hybrid Materials addressed general concepts that will emerge in the next two decades. The panel predicts that organic materials of high and low molar mass will continue to increase their penetration of military materials applications because of the clear advantages they have in terms of functional flexibility, low weight, and facile processibilityóall leading to economic gain over the life cycle. The panel identified a number of research opportunities which will be presented. If these opportunities are pursued, the panel expects that: modeling will become a routine first step in organic materials development; synthesis and processing of organic materials will tend to converge; polymers of high purity with controlled microstructure will become available; aggregates of organic materials on the nanometer scale will yield new opportunities; combinations of low- and high-molar-mass organic molecules with inorganic materials will become widespread.

4:55 PM Invited

Bioderived and Bioinspired Materials: *Michael Jaffe*[1]; [1]NJ Center for Biomaterials and Medical Devices, 111 Lock St., Newark, NJ 07103 USA

The Panel on Bioinspired and Bioderived Materials focused on how the integration of biology and the physical sciences could result in greatly improved, lightweight, multifunctional materials for DoD. Materials derived from biology, for example, biological molecules as the active element in sensors, and materials inspired by biology were considered. The potential impact of applying biological paradigms to the development of materials to meet DoD requirements was reviewed indepth. This paper will present specific DoD opportunities in the areas of structural materials, functional materials, materials for chemical and biological warfare, wound healing, and human performance enhancement. The panel concluded that: biological toughening mechanisms offer a route to next generation lightweight, tough materials; preservation of biological function of biological molecules is a key driver for next generation of biologically-enabled devices; and in-vivo detection strategies to identify toxins and pathogens, including masked agents, may make it possible to detect a single agent molecule.

5:25 PM Closing Remarks Alan M. Lovelace

Measurement and Interpretation of Internal/ Residual Stresses: Composites

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

Wednesday PM Room: 17B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Don Brown, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; David C. Dunand, Northwestern University, Matls. Sci. & Eng. Dept., Evanston, IL 60208-3108 USA

2:00 PM Invited

Load-Sharing in Cemented Carbide Composites: *Aaron D. Krawitz*[1]; Jon W. Paggett[1]; [1]University of Missouri, Dept. of Mech. & Aeros. Eng., Eng. Bldg. E., Columbia, MO 65211 USA

Load-sharing characteristics of WC-Ni and WC-Co cemented carbides (particulate) composites were studied under uniaxial compressive loads using neutron diffraction on the SMARTS instrument at the Los Alamos Neutron Science Center. Cylindrical samples with volume fractions of 0.08, 0.16 and 0.30 Ni and 0.16 Co were employed. The strain response in each phase was monitored using compressive load cycles of 0-500-0, 0-1000-0, and 0-2000-0 MPa, some of which were sequentially repeated. The very substantial thermal residual stresses were measured before and after loading. In addition, the distribution of strain in each phase was monitored as a function of load. Measurements were made in the axial and transverse directions of the samples.

2:30 PM

Measurement and Modelling of the Strain Dependence of Phase Stresses During Plastic Straining of Aluminium/Nickel Composites: *Francis Delannay*[1]; FrÈdÈric Lani[1]; Laurence Ryelandt[1]; Catherine Salmon[1]; [1]UniversitÈ catholique de Louvain, PCIM, Place Sainte Barbe 2, Louvain-la-Neuve B-1348 Belgium

Composites consisting of a pure Al matrix reinforced with a network of sintered Ni-base fibres were prepared by squeeze casting. The composites inherit the in-plane transverse symmetry of the fibre orientations in the preform. The large ductility of these composites allows using them as model systems for assessing current models for predicting the plastic behaviour of ductile multiphase solids. The evolution of the phase stresses during tensile straining of two composites with 20 vol% and 30 vol% of Ni fibres was measured in the 3 principal directions by neutron diffraction. The results were compared to predictions of an elasto-plastic model of the composites based on the equivalent inclusion method and a mean field approach. The model aims at accounting for both the symmetry of the composite and for the connectivity of the reinforcing phase. These factors are shown to drastically affect the partition of plastic strains between the two phases.

2:50 PM

Processing Effects on Residual Stresses and Stress Partitioning in Al/AlCuFe Composites: *Fei Tang*[1]; Thomas Gnaupel-Herold[2]; Henry J. Prask[2]; Iver Eric Anderson[1]; [1]Iowa State University, Ames Lab., Metal & Ceram. Scis. Prog., Rm. 223, Metals Dvlp. Bldg., Ames, IA 50011 USA; [2]NIST Center for Neutron Research, 100 Bureau Dr., Stop 8562, Gaithersburg, MD 20899-8562 USA

A simple metal matrix composite (MMC) system was designed to investigate the effect of consolidation processing methods on residual stress strengthening. An unalloyed Al matrix was reinforced by Al-Cu-Fe quasicrystal (QXL) particulate, using either commercial purity (99.7%) or high purity (99.99%) powders (dia.<10μm) and consolidated by either vacuum hot pressing or quasi-isostatic forging. By neutron diffraction, the residual stresses were measured in composites reinforced by 15, 20, and 30vol.% QXL particles. Also, matrix/reinforcement load transfer was studied using an in-situ tensile frame. This tensile stress partitioning was studied at stress levels from 10 MPa to 150 MPa for the 30vol.% QXL composites. The ambient environment tensile properties of all MMC types were tested, while the elastic modulus was measured by an ultrasonic method. Microstructures and fractography of the composites were also characterized by SEM. Fund-

ing of this project is from DOE Basic Energy Sciences under contract number W-7405-Eng-82.

3:10 PM
Dynamic Response of Residual Stresses Due to Rapid Temperature Changes in Saffil Reinforced AA339 Aluminum: G. Langelaan[2]; *S. Saimoto*[1]; W. J. Baxter[3]; [1]Queenís University at Kingston, Matls. & Metallurgl. Eng., 60 Union St., Rm. 207, Nicol Hall, Kingston, Ontario K7L 3N6 Canada; [2]K. U. Leuven, Dept. MTM, Heverlee Belgium; [3]Formerly of General Motors, R&D Ctr., Warren, MI USA

The residual stresses in the matrix of an AA339 aluminum 15% (vol.) Saffil composite were measured by X-ray diffraction at 100∞C and 400∞C , after temperature cycling were performed. A special heating stage was developed with a very low thermal expansion to minimize systematic errors due to specimen alignment and a very low thermal mass to permit rapid temperature changes. A linear position sensitive detector enabled diffraction peak profiles to be measured in 5 seconds. The slow thermal cycling was at 10∞C/min for both heating and cooling whereas that for rapid cycling was 200∞C/min between 100∞C and 400∞C. These conditions nearly simulates the temperature responses for the piston head on changing between idling and acceleration. The residual stress responses to thermal cycling for the as-cast and samples in the T5 condition were compared by means of a stress-temperature plot. The greater stability of the T5 specimens is attributed to the associated microstructural reactions to the heating/cooling cycles in the distribution of precipitates.

3:30 PM
Strain Evolution After Fiber Failure in a Single Fiber Al/Al2O3 Composite: *Jay C. Hanan*[1]; Sivasambu Mahesh[2]; Ersan Ustundag[1]; Irene J. Beyerlein[2]; Donald W. Brown[3]; Mark A.M. Bourke[3]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Theoretl. Div., Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

The evolution of elastic strain with applied cyclic tensile loading was monitored for each phase of an Al-matrix/Al2O3-fiber composite using neutron diffraction. The Al2O3 fiber fractured during the first loading while the matrix deformed plastically. A finite element model was developed to interpret the diffraction strain data. The model was shown to account for micromechanical phenomena such as fiber breaking, matrix plasticity and interfacial slipping.

3:50 PM Break

4:05 PM Invited
Measurement and Modeling of Internal Stresses in a Ti-6Al-4V/SiC Composite: *Hahn Choo*[1]; Partha Rangaswamy[2]; Mark A.M. Bourke[3]; [1]The University of Tennessee, Matls. Sci. & Eng., Knoxville, TN 37996 USA; [2]Los Alamos National Laboratory, ESA-DE, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

Internal stresses in a Ti-6Al-4V alloy reinforced with 35 volume % SiC continuous fibers were systematically studied using in-situ high temperature neutron diffraction. First, we present the thermal residual stresses (TRS) in the matrix and fibers within the composite measured from axial and transverse directions during heating from room temperature to 1170K. Second, the anisotropic thermal expansion behavior of the constituent phases in the composite as well as the bulk expansion behavior will be discussed in terms of the TRS and its inelastic relaxation at elevated temperatures. Finally, we will present results from a recent study on the load partitioning in the composite during uniaxial tensile loading at 700K. The neutron diffraction results, coupled with a finite element analysis, will provide insights to the micromechanics of the TRS evolution, thermal expansion behavior, and load-partitioning in a continuous fiber reinforced composite at elevated temperatures.

4:35 PM
Deformation of Fiber Reinforced Bulk Metallic Glass Matrix Composites: *Bjorn Clausen*[1]; Seung-Yub Lee[1]; Ersan Ustundag[1]; Mark A.M. Bourke[2]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, MST-8, PO Box 1663, MS H805, Los Alamos, NM 87545 USA

Bulk metallic glasses (BMG) have superb mechanical properties: yield strength of above 2 GPa, fracture toughness of up to 55 MPa.m$^{1/2}$ and elastic limit of 2%. However, monolithic BMGs fail catastrophically under unconstrained deformation. To overcome this problem, BMG matrix composites with fiber reinforcements were proposed. We have recently investigated the deformation behavior of composites with W, Mo, Ta and Fe fibers. Loading measurements were performed using in-situ neutron diffraction to determine the lattice strains in the fibers. The diffraction data were then combined with finite element modeling to determine behavior of the matrix. It was shown that usually the reinforcements yield first, then start transferring load to the matrix. The reinforcements possess different yield strengths, elastic constants, interface strengths and coefficient of thermal expansion (CTE) values. The effects of these parameters on the overall composite deformation will be described.

4:55 PM
Micro and Macro Residual Stresses in a Two-Phase Material: *Mohammed Belassel*[1]; [1]Proto Manufacturing, Ltd., 2175 Solar Crescent, Oldcastle, Ontario N0R 1L0 Canada

Residual stresses are generated during processing such as shaping, machining, rolling grinding, heat treatment, etc. These residual stresses can be defined as: Micro-stresses and Macro-stresses and be measured using X-ray diffraction technique, separately in each phase. In this study, the residual stresses are generated in a two-phase material, pearlite and measured after heat treatment and plastic deformation. The results obtained were compared to other experimental techniques and theoretical models such as Self-Consistent. The results also have showed that the second phase cementite can play an important role in the generation of both types of stresses. A methodology for stress measurement in a two phase material have been developed.

5:15 PM
High-Temperature Deformation of Silicon Nitride and its Composites: *Geoffrey A. Swift*[1]; Ersan Ustundag[1]; Bjorn Clausen[1]; Mark A.M. Bourke[2]; Hua Tay Lin[3]; Chien Wei Li[4]; [1]California Institute of Technology, Matls. Sci., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; [4]Honeywell Corporation, Morristown, NJ 07962 USA

The deep penetration of neutrons in most materials allows in-situ studies in extreme environments. This advantage of neutron diffraction was utilized in the investigation of strain and texture evolution during high-temperature deformation of monolithic Si3N4 and its composites with SiC particulates. Tension experiments were performed near 1400∞C using the new SMARTS diffractometer at the Los Alamos Neutron Science Center. The diffraction data provided information about thermal expansion coefficients and elastic constants at high temperature. In particular, the hkl-dependent strains were interpreted by employing self-consistent modeling.

Mercury Management: Mining Operations

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizer: Larry Twidwell, Montana Tech of the University of Montana, Metallurgical and Materials Engineering, Butte, MT 59701 USA

Wednesday PM Room: 1B
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Courtney Young, Montana Tech of University of Montana, Sch. of Mines & Eng., Butte, MT 59701 USA; Kumar Ganesan, Montana Tech of University of Montana, Sch. of Mines & Eng., Butte, MT 59701 USA

2:00 PM Invited
Update: Environmental Assessment of Gold Processing by Mercury Amalgamation in a Developing Country: *Courtney A. Young*[1]; Christopher H. Gammons[1]; Rich L. McNearny[1]; Darell G. Slotton[2]; Fernando Quispe[3]; Estaben Aquino[3]; Alfredo Camac[3]; [1]Montana Tech of the University of Montana, Sch. of Mines & Eng., 1300 W. Park, Butte, MT 59701 USA; [2]University of California-Davis, Dept. of Environml. Sci. & Policy, 2132 Wickson Hall, One Shields Ave., Davis, CA 95616 USA; [3]Universidad Nacional del Altiplano, Mining & Geologl. Eng., Puno Peru

Gold ores mined in developing countries are being processed by mercury amalgamation, a technique long banned in the US due to mercury toxicity and its effects on the environment unless it includes a retort operation for mercury recovery. Amalgamation is a simple process that involves contacting a ground, slurried concentrate or ore with mercury in a slightly agitated vessel usually for several hours. The amalgamated mercury is separated by gravity and then filtered through a fine cloth to collect the gold amalgam as a sponge which is then fired

to remove final remnants of mercury and produce high quality gold. However, in Peru and other developing countries in South America, tailings are discarded into uncontrolled drainages and ponds and the sponge is fired in open air. Clearly, in this case, amalgamation is not environmental friendly and, to make matters worse, is typically conducted by families without proper protection. Furthermore, the technique is inefficient. For the past two years, faculty members from Montana Tech and the Universidad Nacional del Altiplano in Puno, Peru have teamed to study the La Rinconada Mine Complex located in southern Peru. The mine is over 5,000 meters above sea level and exists at the foot of a receding glacier in the Andes Mountains. Sediment and water samples were taken for mercury analysis from (1) around the mine, (2) an old mercury furnace site, (3) one of approximately fifty amalgamation operations, (4) the main drainage and tailings pond, (5) various downstream water bodies including a small lake and river three kilometers away, and (6) a site in the river four kilometers north of Lake Titicaca. In addition, a representative suite of predator fish in Lake Titicaca were analyzed for Hg concentration. Results are presented and discussed with relevance to safety and health concerns for the miners as well as the people in the downstream communities.

2:30 PM Invited
Reduction of Mercury Emissions in Small Scale Mining Using Advanced Gravity Concentration: *Hermann Wotruba*[1]; Wolfram M¸ller[1]; [1]University of Technology-Aachen, Aachen Germany

Amalgamation is widely used to extract especially fine gold from primary and secondary deposits in small scale mining operations. To avoid the environmental impact by the use of mercury in gold processing pure gravity concentration can be used. If a final step to upgrade the gold concentrate with mercury is necessary, a retort can be used which leads to a minimization of mercury emissions up to 90% compared to traditional methods. The authors have experience in processing gold from deposits of different origin with high recoveries. Even ultrafine and interlocked gold of primary deposits could be treated successfully. The equipment used therefore can be adapted to the traditional processing units. One of the main advantages of the tested method is the full acceptance by the small scale miners. In the first part of the paper different methods applying mercury in gold processing in South American small scale operations and their impact to the environment are described. In the second part successful, mercury free, operations based on gravity concentration only that have been carried out in Venezuela, Bolivia and Brazil are presented. Recoveries up to 90% could be achieved and the method was accepted by the small scale miners.

2:55 PM Invited
Mobility of Mercury in Aged Gold Mine Tailings: *Kumar Ganesan*[1]; [1]Montana Tech of the University of Montana, Dept. of Environml. Eng., Montana Sch. of Mines, 1300 W. Park, Butte, MT 59701 USA

Elevated mercury levels are expected in aged gold mine tailings due to residual mercury from amalgamation process. The fate of mercury in tailings is not well understood. Its potential health risk depends mainly on the degree to which mercury can be mobile in soil. Mercury is removed from soil by evaporation, plant uptake, microbial activities, leaching, and other physico-chemical processes. In this research the amount of mercury lost by evaporation was evaluated. Several aged gold mine tailings in Montana were sampled for total mercury and its flux into the atmosphere. Mercury levels in tailings were higher than the general background levels in most sites. The mercury levels in some cases were as high as 10,000 ug/g. The field measurements using flux chambers indicated a wide range of mercury flux from aged gold mine tailings. The mercury flux ranged from 0-11,000 ng/m2/hr. The mercury loss by evaporation helps the soil to rid of mercury but the mercury vapor then will participate in the atmospheric mercury cycle. The low solubility of elemental mercury in water helps to minimize mercury leaching from tailings. However, conversion of elemental mercury to more soluble forms by bio-geo-chemical processes can enhance its mobility posing increased health risk.

3:20 PM Invited
Mercury Extraction from Precious Metals Ores: *Charles Washburn*[1]; Charles Gale[1]; [1]Summit Valley Engineering and Equipment, 450 E. 1000 N., Salt Lake City, UT 84054 USA

The authors review mercury scources, and contaminent streams, with particular emphasis on mercury that is extracted from precious metal ores. The design and operation of both large and small mercury distillation facilities are detailed. The relevent properties and characteristics of mercury and mercury vapor are presented. Facility engineering with respect to industrial hygene, area ventilation, and material handling is presented.

3:45 PM Break

4:00 PM Invited
Alkaline Sulfide Treatment of Mercury: *Suzzanne Nordwick*[1]; [1]MSE Technology Applications, 2000 Tech. Way, Butte, MT 59701 USA

The metallurgical treatment of mercury-containing materials for precious or base metals recovery imposes many processing and environmental challenges. Mercury is usually present as elemental or cinnabar. Historically, mercury has been removed from mined ores pyrometallurgically by roasting; but, hydrometallurgical methods have also been employed. For example, alkaline sodium sulfide solution acts as an universal solvent for most mercury compounds. While, most other metals are highly insoluble in this solution. This allows for a high degree of selective separation of mercury from other metals with the exception of tin, antimony and arsenic. Mercury leached with a solution of sodium hydroxide and sodium sulfide or sulfur can be recovered by cementation or stabilized through sulfide precipitation. Hydrometallurgical technology for the pretreatment of mercury removal from gold, silver, and base metal ores using alkaline sulfide leaching will be discussed with a focus on alkaline sulfide leaching of mercury sulfide-containing materials.

4:25 PM Invited
The Treatment of Mercury Bearing Tetrahedrites from Solvakia: *Corby G. Anderson*[1]; [1]Montana Tech of the University of Montana, Ctr. for Adv. Minl. & Metallurgl. Procg., 1300 W. Park, Butte, MT 59701 USA

In the Rozonava deposit in Slovakia tetrahedrite mineralization has historically been processed pyrometallurgically for its silver and copper values. These tetrahedrites contain large amounts of mercury which pose both a processing and environmental challenge. This paper outlines the processing of these materials by industrial alkaline sulfide hydrometallurgical methods. Relevent economics will be discussed.

4:55 PM Invited
Small Scale Artisanal Gold Operation in the Nambija District (Ecuador): *David F.J. Y. Bastin*[1]; Vicente Mata Balseca[1]; Jean Frenay[1]; [1]University of Liege, Metall. & Minl. Procg., Chemin des Chevreuils 1, Bldg. 52, Liege 4000 Belgium

The Nambija District is one of the gold district of Southeastern Ecuador mainly operated by artisanal and small scale mining. This paper describes a typical small scale (50 to 80 t/d) artisanal operation encountered in this region. The flowsheet of operations is described with special emphasis for the Hg participation. A metallurgical balance of the gravimetric circuit was calculated. Mineralogical observations of some of the amalgamation tails were realized. The different tails were subjected to orientative gravimetric tests to recover Au and Hg. Preliminary results are discussed.

Microstructural Processes in Irradiated Materials: Stainless Steels, Radiation Induced Segregation, Ion Irradiation and Other Materials II

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

Wednesday PM Room: 11A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Jeremy Busby, University of Michigan, Nucl. Eng. & Rad. Scis., Ann Arbor, MI 48108 USA; Lance Snead, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37830-6138 USA

2:00 PM
Role of Irradiated Microstructure and Microchemistry in Irradiation Assisted Stress Corrosion Cracking: *Gary S. Was*[1]; Jeremy Busby[1]; Matthew Sowa[1]; Ryan Dropek[1]; Mark Hash[1]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., 1921 Cooley Bldg., Ann Arbor, MI 48109-2104 USA

Irradiation has a profound effect on the stress corrosion cracking propensity of austenitic alloys in high temperature water. Irradiation

assisted stress corrosion cracking (IASCC) has been well documented both in the laboratory and in service over the past two decades. Numerous studies have shown that the degree of intergranular stress corrosion cracking increases with dose. However, the microstructure is simultaneously changing in several ways (dislocation loops, voids, segregation and hardening) and, not surprisingly, they all correlate with increased cracking susceptibility. As a consequence of their simultaneous development, the attribution of IASCC to one or more of these features has been difficult to establish. This paper will examine the changes in microstructure, microchemistry and hardening in austenitic alloys as a result of irradiation and how they can affect stress corrosion cracking individually and collectively. Potential mechanisms by which such changes could impact stress corrosion cracking susceptibility in high temperature water will be presented.

2:45 PM
Interaction of Defects with Grain Boundaries and Implications for Microstructure Evolution in Irradiated Materials: *Maria J. Caturla*[1]; Alison Kubota[1]; Jaime Marian[1]; Brian D. Wirth[1]; [1]Lawrence Livermore National Laboratory, Chem. & Matls. Sci., PO Box 808, L-353, Livermore, CA 94550 USA

Void denudation close to grain boundaries has been observed in irradiated materials. In some cases grain boundary migration also occurs during irradiation. To gain basic understanding of these phenomena we have performed atomistic simulations of the interaction between defects and grain boundaries for different defect types (vacancies and interstitials) and sizes as well as different grain boundary types. The effect of He in these interactions is also discussed. We study changes in grain boundary configuration as the defect density increases. A simple kinetic Monte Carlo model of defect evolution close to grain boundaries is developed. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

3:05 PM
Ab Initio Atomic-Scale Modeling of the Zr-I Interactions in Conditions of Stress-Corrosion Cracking: *Christophe Domain*[1]; Alexandre Legris[2]; [1]EDF R&D, MMC, Site des Renardieres, Route de Sens, Ecuelles, Moret sur Loing 77818 France; [2]LMPGM, USTL, Bat C6, Villeneuve DíAscq 59655 France

We have investigated the iodine-zirconium interactions using ab initio atomic-scale calculations in order to elucidate possible mechanisms of stress corrosion cracking. Our calculations show that for the gas pressures estimated during the reactor power transients, the reduction of the zirconium effective surface free energy induced by the gas adsorption is significant (up to 50%). In particular, for a given iodine partial pressure, the surface reduction for the basal planes is higher than for the prismatic ones, in agreement with the experimental observation of a cleavage along the basal planes. We have also estimated the iodine surface diffusion coefficient, and its high value (about 10-6 cm2/s at 600K) indicates that iodine is mobile enough to follow the crack tip during the cracking experiments reproduced in laboratory conditions. Our results clearly rules out the influence of absorbed iodine during the cracking process, its concentration being totally negligible.

3:25 PM
Effect of Irradiation on the Microstructure and Microchemistry of Grain Boundary-Engineered Austenitic Alloys: *Ryan Dropek*[1]; Gary S. Was[1]; James I. Cole[2]; Jian Gan[2]; Todd R. Allen[2]; [1]University of Michigan, Nucl. Eng. & Radiologl. Scis., 2355 Bonisteel Blvd., 2940 Cooley Bldg., Ann Arbor, MI 48109-2104 USA; [2]Argonne National Laboratory, Nucl. Tech. Div., Idaho Falls, ID 83403 USA

Changes to the composition and structure of grain boundaries have the potential to improve the resistance to irradiation assisted stress corrosion cracking of austenitic stainless steels. Steels of composition Fe-18Cr-8Ni-1.25Mn and Fe-16Cr-13Ni-1.25Mn were chosen for modifications of the grain boundary composition and structure. Composition changes included alloying additions of Zr to the 18Cr-8Ni alloy and Mo and P to the 16Cr-13Ni alloy, followed by the application of special heat treatments designed to segregate Cr to the grain boundary prior to irradiation. Structure changes entailed enhancing the coincident-site lattice boundary fraction over that in an annealed structure by a combination of deformation and thermal treatment. Composition and structure changes were applied individually and collectively to selected alloys that were then irradiated with 3.2 MeV protons to 1 dpa at 400∞C. The effect of grain boundary engineering on microchemistry, along with associated changes in microstructural development due to bulk composition changes are reported.

3:45 PM Break

4:15 PM
The Importance of Chemical Disorder in Radiation-Induced Amorphization of SiC: *Xianglong Yuan*[1]; Linn W. Hobbs[1]; [1]Massachusetts Institute of Technology, Depts. of Matls. Sci. & Eng. & Nucl. Eng., Rm. 13-4054, 77 Massachusetts Ave., Cambridge, MA 02139-4307 USA

Structural freedom considerations based on rigidity theory argue that the multiply corner-sharing network of silicon carbide (four tetrahedra sharing each corner) should not be easily amorphized, contrary to experimental results from electron and heavier particle irradiation experiments. We have used a combination of molecular dynamics simulation (with an empirical potential) and topological analysis to show that SiCís facile amorphizability is controlled by the potential for chemical disorder in this material, with a chemical disorder threshold (defined by the ratio of homonuclear to heteronuclear bonds) about 0.3. Furthermore, we have shown that fully chemically-disordered SiC (random site occupation) undergoes a reversible glass transition, with a MD-model glass transition temperature about 3000 K. The implications for radiation-induced amorphization of SiC and other isostructural compounds are addressed.

4:35 PM
Effect of Dislocation Network on Void Swelling: *Sergei L. Dudarev*[1]; A. A. Semenov[2]; C. H. Woo[2]; [1]EURATOM/UKAEA Fusion Association, Culham Sci. Ctr., Oxfordshire OX14 3DB UK; [2]Hong-Kong Polytechnic University, Dept. of Mechl. Eng., Hung Hom, Kowloon Hong-Kong

We found that competition between nucleation of voids and (three-dimensional) diffusion of vacancies, interstitial atoms and interstitial atom clusters in the presence of heterogeneously distributed dislocations in the vicinity of a grain boundary in an alloy under irradiation can be responsible for the experimentally observed effect of heterogeneous void swelling. The new model is able to describe a number of significant features characterising the effect, including the formation of void denuded zones, the variation of the density of dislocations as a function of distance to the grain boundary and the formation of unusually large voids in the immediate vicinity of grain boundaries. Profiles of void swelling calculated using the new model are found to agree quantitatively with experimental observations. This finding shows that the spatial distribution of the dislocation component of microstructure may play a determining part in the formation of heterogeneous profiles of voids swelling near grain boundaries.

Products, Applications, and Services Showcase: Reduction Technology & Laboratory Analysis

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizer: David V. Neff, Metaullics Systems Company, Solon, OH 44139 USA

Wednesday PM — Room: 5A
March 5, 2003 — Location: San Diego Convention Center

Session Chair: TBA

2:00 PM
CLAYBURN DRI-BARRIER MIXÆñIts Application as a Barrier Lining in Reduction Cells: *Ron Smith*[1]; [1]Clayburn Industries, Ltd., Al Prod. Div., 33765 Pine St., Abbotsford, British Columbia V2S 5C1 Canada

CLAYBURN DRI-BARRIER MIXÆ is a unique, penetration resistant dry vibratable barrier lining developed specifically for use below the cathode in aluminum electrolytic cells. In our presentation we will discuss the developmental history as well as the technical application and installation of Dri-Barrier MixÆ. In addition, we will discuss Clayburnís successful production and installations of Dri-Barrier MixÆ at smelters in the emerging Chinese aluminum industry.

2:20 PM
New Busbars for Smelters: *Serge Risser*[1]; Gerard Hudault[1]; [1]Aluminium Pechiney Lannemezan, Lannemezan F-65300 France

With increasing number of cells in state of the art aluminum smelting technology, smaller busbars are being replaced by bigger ones, which allows an accurate electrical balance and better cooling efficiency. Aluminium Pechiney has improved its welding technology and has developed a new busbar production route for its AP18, AP30 & AP50 technology. The recently upgraded horizontal continuous casting ma-

chine at Lannemezan plant supplies busbars that meet customersí highest quality requirements. Other achievements at Dunkerque, Tomago and Alouette smelters are expected to develop further applications of this technology. The presentation will review updated market information, the use of busbars, the different aluminium grades for conductors, alloys for stems, and key parameters for a safe and successful cast. Furthermore, technical information about facilities will be given.

2:40 PM
The MOELLER Direct Pot Feeding System for a Smooth and Constant Pneumatic Transport of Secondary Alumina to the Electrolyte Cells: *C. Duwe*[1]; [1]Moeller Materials Handling GmbH, Pinneberg Germany

The MOELLER Direct Pot Feeding is a high efficient and full automatically facility for a smooth and wear-resistant constant pneumatic transport of secondary alumina to the electrolyte cells. This pneumatic transport system is a combination of a dense phase feeding from a storage silo to intermediate bins at the electrolyte cells and a SFS (Super feeding system) air slide system directly to each of the electrolyte cells. The TURBUFLOW dense phase feeding as well as the SFS (Super feeding system) air slide system are approved systems which preserves the particle size distribution and the flow ability of the alumina. The main conveying air volume is separated in the intermediate bin and do not influences the situation of feeding secondary alumina from intermediate bin via point feeders into the electrolytic cell. The mass flow of secondary alumina is blocked automatically when the bunker of an electrolytic cell is full and the bulk material cone level has reached the fill spout discharge opening. The fluidising of the bulk material inside the SFS air slide system still works and ensures no remarkable variations of the bulk density. When secondary alumina is removed from the bunker of the electrolytic cell, the pneumatic transport starts again automatically and a constant and reliable mass federate to the pots is ensured. This most competitive system for the pneumatic feeding of secondary alumina to electrolytic cells is one of the components to ensure the quality of the aluminium production on the highest possible level.

3:00 PM
New Generation of Tapping Vehicles: *Knut Prestnes*[1]; [1]HMR Group AS, Prestnesvegen 68, Husnes 5460 Norway

In effort to increase productivity, industry looks for more effective ways to deliver smelted aluminium to the cast house. Capacity of tapping is the key. But in tapping vehicles, capacity increase is limited by two factors: maximum load per axle and lifting height with vacuum tapping. A new tapping concept elaborated and tested in one of the smelters in Norway, reduces these obstacles. A new generation of tapping vehicles has been developed. World patented system allows tapping up to 13 tons (theoretically up to 18-20 tons) of aluminium per one cycle. With this capacity one tapping vehicle can tap from the pots and deliver to the cast house, located at 1 km distance, 12 tons of smelted aluminium per hour. This is industry worldwide record. Additionally the whole process is one-man operated and controlled. Together with simplified crucible cleaning system and quick tapping-, siphon-tube changing they constitute important cost reduction factors.

3:20 PM Break

3:40 PM
Rockwell, Vickers or Nanoindentation?: *Seth Downs*[1]; Dehua Yang[1]; Oden Warren[1]; Thomas Wyrobek[1]; [1]Hysitron, Inc., 5251 W. 73rd St., Edina, MN 55439 USA

Recent trends in miniaturization of devices and demands for high-performance materials dictate a change in the way that materials are characterized. Understanding of the structure, properties and role of processing at the nanoscale is crucial to achieving the desired performance, regardless of the size scale of the final product. Nanomechanical characterization is presented as a new solution for materials testing, expanding on the range of capabilities of traditional hardness testing. Nanoindentation provides superior lateral and vertical resolution, allowing testing of surface properties or single phases of multi-phase materials. Typical nanoindentation and nanoscratching provide measurement of mechanical properties such as modulus, hardness, fracture toughness, friction coefficients and wear resistance. New techniques, such as acoustic emission monitoring and stiffness and modulus mapping, allow investigation of the initial stages of fracture and characterization of interfaces. An overview of the capabilities and techniques offered by Hysitronís nanomechanical characterization instruments will be presented. Comparison and interrelationships of Rockwell, Vickers and nanoindentation will also be discussed.

4:00 PM
Management, Molten Metal and Protective Clothing: *Larry Stinson*[1]; [1]Silver Needle Inc., 402 Main St., Kellogg, ID 83837 USA

This presentation will combine all the strategic and economic goals that work together to make a safety program not only succeed for a company but to actually be a benefit and asset as well. Topics to be covered include: 1. Liability; 2. Location; 3. Personnel; 4. Clothing Committee and 5. Economics.

4:20 PM Cancelled
Pulsar: Total Analytical Flexibility for Solids, Powders and Solutions: *Charles Belle*[1]; Phil Bennett[1]; Bruce MacAllister[1]; [1]Leeman Labs, Inc., 6 Wentworth Dr., Hudson, NH 03051 USA

Recycling - General Sessions: General Recycling

Sponsored by: Light Metals Division, Jt. LMD/EPD-Recycling Committee
Program Organizers: Han Spoel, Spalco Metals Inc., Toronto, Ontario M5R 1W8 Canada; Paul Crepeau, General Motors Corporation, MC/486-710-251, Pontiac, MI 48340-2920 USA

Wednesday PM Room: 3
March 5, 2003 Location: San Diego Convention Center

Session Chair: Han Spoel, Spalco Metals Inc., Toronto, Ontario M5R 1W8 Canada

2:00 PM Announcements

2:10 PM
PickingñAn ìOldî Process for Separation of Non-Ferrous Metals?: *Stefan Mutz*[1]; Thomas Pretz[1]; [1]Aachen University of Technology, RWTH Aachen, Wuellnerstr. 2, Aachen D-52062 Germany

The term ìSORTINGî implies the separation of a blended material into two or more products, all or both of which may be classified by the different material characteristics in the preparation technology. The oldest sorting procedure, by handpicking, is today used infrequently to separate different material mixtures, because of the considerable cost of labour and the strict hygiene regulations at the workplaces in Europe. In principle, manual sorting is only used to solve separation tasks, in which mechanised sorting could not be utilised. It should be noted that picking is one of the most selective processing routes. Current developments in the fields of computers systems and sensor engineering makes the creation of automatic picking devices possible, especially where efficiencies far beyond the realms of handpicking are desired. In the future therefore, automatic picking applications will not only be utilised in the already common applications, such as waste glass assortment by colour, but also in a variety of complex material mixtures e.g. old shredded cars. Thus, automatic picking, supported by high performance computer systems and new sensor technology, becomes an attractive alternative in many areas of waste processing. However, in order to merge the application of the automatic sorting procedures with the complex material flows of the recycling industry, a substantial research and development need still exists. The demands of the users are to sort increasingly larger mass flows, which themselves constitute larger material varieties. A further demand is the need to accurately identify and sort the single grains as the mass flow rises. This lecture compares the traditional sorting procedures, such as hand picking with todayís state of the art methods of classification within the range of automatic picking. Apart from the potential technological developments, some operational issues and future opportunities for this technology are illustrated especially in the fields of NF-metals.

2:40 PM
Recycling Rates of Waste Home Appliances in Taiwan: *Esher Hsu*[1]; Chen-Ming Kuo[2]; [1]National Taipei University, Dept. of Stats., 67 Sec., 3 Min-Sheng E. Rd., Taipei 104 Taiwan; [2]I-Shou University, Dept. of Mechl. Eng., 1 Sec., 1 Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

On July 5, 1997, Environmental Protection Administration of Taiwan publicized the recycling regulation of waste home appliances that include four items, namely, television, refrigerator, washing machine, and air conditioner. It is believed that this regulation pioneers the law enforcement of waste home appliances in the world. The objective of this study is to provide helpful base for amending recycling rates of waste home appliances. This study investigates the collection and recycling costs of waste home appliances in accordance with these newly established routes and facilities, respectively. Cost

survey was conducted among collectors and recycling plants of waste home appliances; consequently, the collection and recycling costs were analyzed, correspondingly. Results provide some suggestions regarding recycling route, recycling system, recycling rates paid by manufactures and importers, recycling subsidies to collectors and recyclers. Those suggestions may provide useful policy implications to EPA for the future decision-making regarding the recycling of waste home appliances.

3:10 PM
Economic Assessment of the European Union Directive for Recycling of End-of-Life Vehicles: *Randolph E. Kirchain*[1]; Adriana Diaz-Triana[1]; [1]Massachusetts Insitute of Technology, Matls. Sys. Lab., 77 Massachusetts Ave., E-40 Rm. 202, Cambridge, MA 02139 USA

The Materials Systems Laboratory at the Massachusetts Institute of Technology (MSL) is conducting research on the economic and environmental implications of End-of-Life Vehicle (ELV) recycling, in the context of the recent European Community Directive 00/53/EC. This Directive sets explicit recycling targets for automakers. Specifically, recycling/recovery must be increased to 80/85% by 2006 and to 85/95% by 2015. Two key goals for the Directive are: 1) to prevent waste, preferably through improved design, but also through more intensive recovery and 2) to improve the environmental performance of those operators involved in the treatment of ELVs. MSL has developed a framework for the analysis of vehicle recycling in the US, through the use of process-based Technical Cost Models (TCMs). TCMs describe the recycling system, with different arrays for operators and their interactions, taking into account technical information about all relevant processes. TCMs have proven to be powerful tools for evaluating cost implications when looking at different materials and/or processing alternatives, providing the decision-maker with an accessible means for comparing distinct approaches as to inform on technical decision. MSL has recently developed recycling TCMs to reflect the economic and environmental dimensions of vehicle recycling in two different European Member States: Portugal and The Netherlands. The infrastructure for ELV processing in Portugal is based on dismantling and limited shredding and materials separation activities, while the more mature Dutch system promotes increased dismantling of materials for recycling. The activity level of the dismantling, shredding and materials separation operators and the associated costs are forecasted in the context of each country. The analysis of these different ELV recycling infrastructures through the development of appropriate TCMs is a valuable approach to understand key economic factors often overlooked when considering the implementation of recycling policies.

3:40 PM Break

3:55 PM
Radionuclide Surrogate Decontamination of Metal Using Electroslag Remelting: *David K. Melgaard*[1]; Gregory J. Shelmidine[2]; James A. Van Den Avyle[2]; Martin A. Molecke[3]; [1]Sandia National Laboratories, Div. 1835, MS 0889, PO Box 5800, Albuquerque, NM 87185-0889 USA; [2]Sandia National Laboratories, Div. 1835, MS 1134, PO Box 5800, Albuquerque, NM 87185-1134 USA; [3]Sandia National Laboratories, Div. 6141, MS 718, PO Box 5800, Albuquerque, NM 87185-0718 USA

A significant emerging recycling, waste minimization problem stems from production of power using nuclear fuel. Premium quality metals used in nuclear plants become contaminated and are unsuitable for reuse. Consequently decontamination studies were conducted to evaluate the decontamination capacity and partitioning effectiveness of Electroslag Remelting (ESR). Eight laboratory melts were conducted to evaluate the effectiveness of using ESR to transport radionuclides from the liquid metal phase to the liquid flux or slag phase. To determine the decontamination effectiveness, several surrogate compounds for uranium and plutonium were added to the melts. During the melts, the melting rate was varied, typical slag additives were used, and two different slag compositions were evaluated. The resultant post-test ingots and slag were then analyzed to determine the partitioning of the surrogates. ESR generally proved very effective in removing contaminates from the metal with a few noted exceptions.

4:25 PM
A Process to Recycle Glass Fibers from Glass Manufacturing Waste: *B. J. Jody*[1]; J. A. Pomykala[1]; E. J. Daniels[1]; J. C. Wells[2]; C. E. Davis[2]; [1]Argonne National Laboratory, Energy Sys. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Saint-Gobain Vetrotex America, 4515 Allendale Rd., Wichita Falls, TX 76310-2199 USA

This paper describes a one-step thermal process, developed at Argonne National Laboratory, to clean and purify the glass waste that is generated during the glass forming process so that the waste could be recycled into new glass fibers or new glass products without adversely impacting the operations of the industry or the quality of its products. The process involves shredding the waste to a manageable size and then processing it at temperatures below its melting point to remove the polymer-coating layer. Preliminary cost analysis of the process shows a potential payback of less than 2 years. The glass industry generates more than 60,000 tons of glass manufacturing waste annually in the glass forming process alone. Recycling of this waste stream, can keep this non-biodegradable waste out of the landfills. In addition use of recycled glass lowers energy costs, by an average of about $5 per ton compared to using virgin raw materials to make virgin glass fibers. This will also reduce the emission of CO_2 by 17,400 tons a year and will also reduce the emission of nitrogen oxides.

4:55 PM
Para-Eco Incinerator Ash Processing System for Cleaning Slag with Recovering Metals: *Kiyoshi Takai*[1]; Ken-ichi Ohkura[1]; Masao Suzuki[2]; [1]Rasa Corporation, Chitose Bldg., 2-9-4 Nihonbashi Kayabacho, Chuo-ku, Tokyo 103 Japan; [2]AI-Tech Associates, Tokura 3-38-13, Kokubunji-shi, Tokyo 185-0003 Japan

Para-Eco Incinerator Ash Processing System for cleaning slag has been developed through a pilot plant study of 7 years. The major part of the system is an electric furnace, heating to 1773K. The feed material consists of a municipal solid waste incinerator ash mixture with the reducing agent of coke and inorganic flux of calcium and magnesium. With this furnace system three kinds of products are produced. They are an inorganic molten salt that is slag, molten iron alloy absorbing non-ferrous metals, and fly ash. Due to a reducing atmosphere in the furnace, the slag does not contain any metal components as well as chlorine component just like a natural rock and/or aggregate. A retreatment plant modifies this slag into a practically useful aggregate. The plant consists of reheating step followed by a slow cooling step for crystallizing the slag. The iron components in the ash are reduced to molten iron droplets, which concentrate at the furnace bottom. The non-ferrous metal components such as copper, chromium, and others are reduced to each individual metallic state. Accordingly, they are absorbed into the molten iron but not into the molten slag. Volatile metals of lead, zinc, cadmium and others as well as sodium, potassium, and chlorine go into the fly ash again. A solvent extraction method and/or other appropriate procedures are applicable to extract metal components from this fly ash.

Science and Technology of Magnetic and Electronic Nanostructures: Functional Nanostructures: Ferroic Materials

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Ramamoorthy Ramesh, University of Maryland, Department of Materials and Nuclear Engineering, College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Wednesday PM Room: 15A
March 5, 2003 Location: San Diego Convention Center

Session Chair: Timothy D. Sands, Purdue University, Sch. of Matls. Eng., W. Lafayette, IN 47907 USA

2:00 PM
Bringing Oxides into the Silicon World: Strategies to Integrate Functional Oxides Epitaxially with Silicon: *Darrell G. Schlom*[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., 108 MRI Bldg., Research Park, University Park, PA 16802-6602 USA

Oxides possess many unparalleled properties, but we live in the silicon age. Hybrid structures in which the functional properties of oxides can be exploited in combination with silicon offer exciting opportunities for devices. The question is how to integrate such chemically different materials without degrading either material to create an electrically-useful interface. Which oxides are better suited for integration and how does one go about integrating them with silicon? This question is the focus of this talk. A methodology will be described, based on thermodynamics and oxidation kinetics, for the integration of functional oxides with silicon with the goal of achieving an abrupt heteroepitaxial interface. Examples illustrating functional oxides that

can be grown epitaxially on silicon in a low temperature/excess oxidant regime will be presented, as well as functional epitaxial oxide/silicon heterostructures that make use of the integration of epitaxial oxide layers with silicon.

2:45 PM
Interfacial Structure and Properties of Nanoscale Ferroelectrics: *Xiaoqing Pan*[1]; Haiping Sun[1]; Wei Tian[1]; Jeffrey Haeni[2]; Darrell G. Schlom[2]; [1]University of Michigan, Matls. Sci. & Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA; [2]Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16803-6602 USA

In this talk, we will present our recent work on the interfacial structure and properties of nanoscale ferroelectrics. Well-controlled ferroelectric thin films and heterostrucures were grown by reactive molecular beam epitaxy (MBE). The strain relaxation and misfit dislocation formation mechanisms of epitaxial BaTiO3 on (001) SrTiO3 substrates were studied using transmission electron microscopy (TEM) techniques. In-situ TEM studies showed that the threading dislocations climb toward the film/substrate interface during annealing at hight temperatures and form extended misfit dislocations to relax the strain in the epitaxial ultra-thin film. Quantitative high-resolution TEM was employed to examine the atomic positions of cations and anions in the coherent BaTiO3/SrTiO3 heterostructures. It was found that the relative static displacement of cations to anions is much greater than that of bulk BaTiO3, indicating the strain-induced elevation of spontaneous polarization in BaTiO3 thin films that may responsible for the theoretical prediction of giant dielectric constants for layered ferroelectric structures.

3:30 PM
Atomic Layer Controlled Growth of Epitaxial Magnetic Oxide Heterostructures by PLD with In Situ High Pressure RHEED: *Chang-Beom Eom*[1]; [1]University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., 1500 Engineering Dr., Madison, WI 53706 USA

Complex oxide materials possess an enormous range of electrical, optical, and magnetic properties. For instance, insulators, high quality metals, dielectrics, ferroelectrics, semiconductors, ferromagnetics, colossal magnetoresistance materials, superconductors, and nonlinear optic materials have all been produced using oxide materials. A major challenge is the atomic layer controlled heteroepitaxial growth of various complex oxide materials so that these properties can be fully utilized in novel devices. We have grown SrRuO3-SrTiO3-SrRuO3 epitaxial ferromagnetic oxide heterostructures on TiO2- terminated (001) SrTiO3 substrates using pulsed laser deposition including in-situ high pressure RHEED. SrRuO3 is a negatively spin polarized ferromagentic oxide with a lattice parameter of 3.93≈ i.e., a lattice mismatch with (001) SrTiO3 substrates of 0.64%. Our RHEED intensity data and AFM images suggest that the SrRuO3 films on SrTiO3 substrate grow in the step-flow mode with a transition from 2-dimensional layer-by-layer mode into step-flow mode after covering one monolayer of SrRuO3. The origin of the growth mode transition can be attributed to a change in mobility of ad-atoms and switching of a surface termination layer from the substrate into the film. Transmission electron microscopy images of a cross-sectional SrRuO3-6 unit cell SrTiO3-SrRuO3 heterostructure show that the trilayer is single domain with atomically sharp interfaces between SrRuO3 and SrTiO3. Such an atomic layer control of the interfaces and barrier layers in ferromagnetic oxide (FM-I-FM) trilayer junctions allows for quantitative studies of spin-polarized transport across the ferromagnetic oxide (FM-I-FM) junction interfaces.

4:15 PM
Nano and Micro Elastic Domains in Constrained Ferroelectric Films: *Alexander L. Roytburd*[1]; [1]University of Maryland, Matls. & Nucl. Eng, College Park, MD 20742 USA

We present results of experimental and theoretical studies of 90∞ domains and their effect on electro-mechanical properties of PZT tetragonal films. Engineering of constraint is used to design different polydomain structures of elastic domains. Mechanical interaction between a FE film and a substrate is engineered through changing misfit between a FE film and different substrates, creating vicinal structures of the substrate surface, patterning FE films with formation of stripes and islands. Important role of elastic domains in switching of 180∞ FE domains as well as in relaxation of piezostress created by 180∞ domains is discussed. The mobility of elastic domains and their switching in nano-islands resulting in giant direct and converse piesoeffects are demonstrated. The possibility to obtain nanoscale domain structures in constrained films and bulk crystals is explored.

Surface Engineering in Materials Science - II: High Temperature Materials and Surface Alteration

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

Wednesday PM Room: 7A
March 5, 2003 Location: San Diego Convention Center

Session Chairs: Y. Sohn, University of Central Florida, MMAE/AMPAC, Orlando, FL USA; P. Kuppusami, National Institute for Materials Science, High Temp. Matls. Grp., Tsukuba Science City, Ibaraki Japan

2:00 PM
Development of Oxidation Resistant Coatings Based on Gamma TiAl: *Antonis Zaroulias*[1]; Guosheng Shao[1]; Panos Tsakiropoulos[1]; [1]University of Surrey, Sch. of Eng. (H6), Mechl., Matls. & Aeros. Eng., Guildford, Surrey GU2 7XH England

The development of gamma TiAl base alloys has addressed their oxidation and mechanical properties. The selected alloying additions to gamma and gamma + a2 alloys do not result in continuous Al2O3 scale formation in air. An intermixed Al2O3/TiO2 scale continues to form in the more complex alloys, but the rate of growth of this scale has been reduced. Alloys with Cr additions at a level generally above 8 to 10 at% can form a two-phase (gamma + Laves phase) microstructure and are capable of continuous Al2O3 scale formation in air. Furthermore, the Ti-Al-TM tau phases form protective Al2O3 in air, with the best oxidation behaviour exhibited by TM = Cr. Thus, the Ti-Al-Cr system offers unique opportunities for the design of coatings that are mechanically compatible with the substrate and also oxidation resistant. The paper will report results of our experimental study of Ti-Al-Cr coatings applied by PVD on gamma base substrates. The coatings have been designed for oxidation resistance using thermodynamic databases and microstructure models developed at the University of Surrey. A distinct feature of our study is the deposition of amorphous coatings with high crystallisation temperatures. The design and processing of coatings will be presented. The microstructures and properties of the as deposited and nanocrystalline coatings will be discussed.

2:20 PM
Long-Term Oxidation Behavior of Aluminized CMSX-4 Superalloys: *Nan Mu*[1]; Yongho Sohn[1]; Irene L. Nava[2]; [1]University of Central Florida, Adv. Matls. Procg. & Analysis Ctr., PO Box 162455, 4000 Central Florida Blvd., Plando, FL 32816-2455 USA; [2]Solar Turbines, Inc., PO Box 85376, 2200 Pacific Hwy., San Diego, CA 92186-5376 USA

Thermally grown oxide (TGO) scale on aluminized CMSX-4 single crystal superalloy has been studied after long-term oxidation testing to examine the spallation behavior of TGO using photo-stimulated luminescence spectroscopy (PSLS) and microscopy. Twelve disk-shaped (2.54 cm dia. and 0.32 cm thick) specimens were oxidized at 788∫C (1450∫F), 871∫C (1600∫C), 954∫C (1750∫F) and 1010∫C (1850∫F) for 5,000, 7,500 and 10,000 hours. Spallation of TGO scale along the NiAl(B2) grain boundary ridges was observed for specimens exposed to higher temperature and longer time. In contrast, at the lowest temperature and the shortest time of oxidation, a significant amount of metastable gamma- and theta-alumina was observed by PSLS along with some spallation. This type of spallation did not occur along the grain boundary ridges, but within the grains themselves.

2:40 PM
Enhancing the Corrosion Resistance of AISI 304 Stainless Steel by Laser Surface Melting: *S.-H. Wang*[1]; J.-Y. Chen[1]; G. Campbell[1]; L. Xue[1]; [1]National Research Council of Canada, Integrated Mfg. Tech. Inst., 800 Collip Cir., London, Ontario N6G 4X8 Canada

AISI 304 stainless steel is widely used in industry for numerous applications requiring corrosion resistance. Despite its good corrosion

resistance, the steel is prone to sensitization by heating, which significantly reduces its resistance to localized corrosion, especially intergranular corrosion. In this study, a thin layer of sensitized AISI 304 stainless steel surface was melted using a CW CO2 laser, to study the effect of laser surface melting on its corrosion resistance. Laser surface melting produced a thin protective surface layer with extremely fine microstructure, homogeneous distribution of alloying elements and clean microstructure boundaries, which contributes to the desensitization of the stainless steel and enhances its resistance to pitting and intergranular corrosion as well as general corrosion. The laser-melted layer also showed resistance to the sensitization and deterioration of the enhanced corrosion resistance when subjected to post heating.

3:00 PM Break

3:20 PM
Optical Emission Spectroscopy Correlations to Plasma Nitriding Surface Treatments for Process Optimization and Control: R. B. Young[1]; S. Patankar[1]; K. A. Prisbrey[1]; *F. H. (Sam) Froes*[1]; [1]University of Idaho, Inst. of Matls. & Adv. Processes, Col. of Eng., Matls. Sci. & Eng. Dept., Moscow, ID 83844-3024 USA

Optical emission spectroscopy is inexpensive, and gives reliable information about chemical states and operating parameters during plasma nitriding surface treatments of titanium, tool steels, and stainless steels. The objective was to correlate plasma nitriding variables, optical emission spectra, material property improvement, and metallographic, SEM, and XPS surface characterization. The procedure was to measure these variables during and after nitriding in inductively coupled radio frequency plasma. Plasma variables included plasma power, electron temperature, ion density, pressure, gas flow, and others. Pattern recognition algorithms coupled with ab initio molecular modeling calculations helped identify key wavelengths in optical emission spectra. Neural networks and fuzzy logic methods allowed implementation of practical control methods and online optimization of process parameters. Our results included correlations of plasma state parameters with photon emissions, and correlations of nitriding performance with process parameters. These results yielded methods for better process optimization, design, and control.

3:40 PM Moved to Materials Lifetime Sciences Symposium
Physically-Based Models for the Prediction of Fatigue and Dwell Fatigue Crack Growth in Ti-6242: Comparison of Models with Experiments: F. McBagonluri[2]; Chris Mercer[1]; E. Akpan[1]; W. Shen[1]; *W. O. Soboyejo*[1]; [1]Princeton University, Princeton Matls. Inst., D404 E. Quad., Princeton, NJ 08544 USA; [2]University of Dayton, Dept. of Matls. Eng., Dayton, OH 45390 USA

4:00 PM
Influence of Ta Content on Microstrucure and Mechanical Properties of Ir-Ta as Diffusion Barrier Coatings on Nickel Base Single Crystal Superalloy TMS-75: *Parasuraman Kuppusami*[1]; Hideyuki Murakami[2]; Takahito Ohmura[3]; [1]National Institute for Materials Science, High Temp. Matls. Grp., High Temp. Matls. 21 Proj., 1-2-1, Sengen, Tsukuba Science City, Ibaraki 305-0047 Japan; [2]The University of Tokyo, Dept. of Matls. Eng., Sch. of Eng., 7-3-1, Hongo, Bukyo-ku, Tokyo 113-8656 Japan; [3]National Institute for Materials Science, Steel Rsrch. Ctr., 1-2-1, Sengen, Tsukuba Science City, Ibaraki 305-0047 Japan

Ir- Ta alloy coatings have been proposed as a novel diffusion barrier material on nickel base single crystal superalloys owing to their high yield strength at high temperatures. This investigation has been undertaken to generate the database of mechanical and oxidation resistance properties of Ir-Ta alloy coatings with varying Ta contents. DC magnetron sputter deposition of Ir-Ta coatings from a composite target of Ir-Ta was carried out at 573 K on <100> oriented Ni- base single crystal superalloy TMS-75 substrates. The influence of tantalum contents of 16.5, 23.9, 39.8 and 65.1at % in Ir-Ta coatings on the microstructure and mechanical properties has been investigated. Nanohardness and microhardness measurements indicated that addition of Ta alters the mechanical properties such as hardness and Youngís modulus significantly. Further, a comparative study on the thermal cyclic oxidation resistance of the Ir-Ta modified aluminide coatings is presented.

4:20 PM
Sulfidation Behavior of Fe20Cr Alloys: *Lalgudi V. Ramanathan*[1]; Marina F. Pillis[1]; [1]Instituto de Pesquisas Energeticas e Nucleares (IPEN), Matls. Sci. & Tech. Ctr., Av. Prof. Lineu Prestes 2242, Cidade Universitaria, Sao Paulo 05508-000 Brazil

High temperature corrosion of structural alloys in sulfur bearing environments is many orders of magnitude higher than in oxidizing environments. Increase in oxidation resistance of various alloys with addition of reactive elements is well known. The effect of yttrium on sulfidation behavior of Fe-Cr alloys is still inconclusive. This paper reports the effect of yttrium on sulfidation behavior of Fe-20Cr alloys in H2-2%H2S environment at 700∞C. In the presence of Y, the sulfidation rate of the alloy decreased by an order of magnitude, the sulfide grain size was smaller and the sulfide growth mechanism predominantly anionic, after an initial cation diffusion controlled process. The sulfide layer on the FeCrY alloy was significantly thinner than that formed on the FeCr alloy. The alloy/sulfide interface on the FeCr alloy was planar where as that on the FeCrY alloy was undulated, with large regions of internal sulfidation.

The Mike Meshii Symposium on Electron Microscopy: Its Role in Materials Research: Electronic, Magnetic, Optical, Piezoelectric Materials, Surface Treatment

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

Wednesday PM Room: 9
March 5, 2003 Location: San Diego Convention Center

Session Chairs: A. K. Vasudevan, Office of Naval Research, Matls. Sci. Div., Arlington, VA 22217 USA; P. W. Voorhees, Northwestern University, Matls. Sci. & Eng. Dept., Evanston, IL 60208 USA

2:00 PM Invited
Relation Between Stress-Induced Voiding and Film Texture in Cu Thin Films: *Junichi Koike*[1]; Atsuko Sekiguchi[1]; Makoto Wada[1]; Kouichi Maruyama[1]; [1]Tohoku University, Dept. of Matls. Sci., 02 Aobayama, Aoba-ku, Sendai, Miyagi 980-8579 Japan

Cu thin films are new materials for interconnect lines in advanced integrated circuits. Because of thermal expansion misfit between Cu and its surroundings, thermal stress arises and causes stress-induced voiding that leads to device failure. Since the elastic property of Cu is highly anisotropic, voiding tendency is expected to depend on local crystallographic texture. We investigated the relationship between the voiding tendency and the film texture using transmission and scanning electron microscopy and electron back scattering analysis in conjunction with finite element calculation of stress distribution. We found that voids were formed at interfaces and corners of incoherent twins under a large stress concentration in a (111) oriented films. By changing texture to a random or a (100) oriented texture, stress-induced voiding could be avoided because of the absence of the incoherent twins. Possible ways to control the film texture and the voiding tendency will also be presented.

2:30 PM
TEM Observation of Phase Transformation and Magnetic Structure in Ferromagnetic Shape Memory Alloys: *Koichi Tsuchiya*[1]; [1]Toyohashi University of Technology, Production Sys. Eng., Tempakucho Hibarigaoka 1-1, Toyohashi, Aichi 441-8580 Japan

Ni-Mn-Ga ferromagnetic shape memory alloys have been attracting much attention as a new class of magnetic actuator materials. It was reported that in Ni-Mn-Ga strain up to 5% can be obtained by magnetically-induced twinning in the martensite phase. This report describes the results of in-situ TEM observations of martensitic transformation and preceding intermediate phase transformation. It was revealed that the intermediate phase is characterized by the formation of satellite spots at 1/6g220 positions, which suggests the formation of micro domains accompanied by the modulation of a {110}<1-10> type with 6 layes period. Transformation from the intermediate phase to the martensite phase was continuous. The Lorentz microscopy was also used to investigate the magnetic domain structures in the martensite and its relation to the martensite microstructures such as the variant and microtwins. It was revealed that microtwins form the

magnetic domains and the twin boundaries have a character of 90∞ walls.

2:50 PM
Mapping of Magnetization Patterns Through Phase Reconstructed Lorentz Microscopy: *Marc De Graef*[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

Lorentz microscopy has for many years been a qualitative observation technique, providing images of magnetic domain walls (through the out-of-focus or Fresnel mode) and magnetic domains (through the Dark Field or Foucault mode). Recently, it has become possible to use the out-of-focus Fresnel images to compute the phase of the electron wave. From this phase, the magnetization pattern can be computed by a simple gradient operation. The method relies on the so-called Transport-of-Intensity Equation (TIE), which relates the spatial variation of the phase to the longitudinal derivative of the image intensity. Simple and fast solution algorithms are now available to solve the equation almost in real-time. In this presentation we will describe the foundations of the method, and provide several illustrations using the following material systems: patterned Permalloy islands, ferromagnetic shape memory alloys, and hard magnetic materials.

3:10 PM
Electron Microscopy of Combined Reaction Processed L10 FePd Based Ferromagnets: *Amal A. Al-Ghaferi*[1]; *Anirudha R. Deshpande*[1]; Jorg M. Wiezorek[2]; Huiping Xu[1]; [1]University of Pittsburgh, MSE, 864, Benedum Hall, Pittsburgh, PA 15213 USA; [2]University of Pittsburgh, MSE, 848, BEH, Pittsburgh, PA 15213 USA

The Extraordinary magnetic properties in FePd based (L10) intermetallic alloys depend strongly on the microstructure and defect structure. The tetragonal ordered L10 phase forms from an FCC (Fe,Pd) solid solution via a first order transformation between 650-790°C (Tc) depending on Pd content. In conventionally processed FePd alloys dodecahedrally conjugated polytwin morphologies develop with submicron dimensions. The presence of polytwins is detrimental for the technical magnetic properties resulting in considerably lower MR and HC than theoretically predicted for (L10) FePd. Combined reaction processing exploits the competition between recrystallization and the ordering transition during annealing of cold worked Fe-Pd alloys and enables microstructural control to taylor technical properties. Combined reaction processing has been used to produce FePd alloys with fourfold increased coercivity with respect to the conventionally processed material. Here SEM and TEM are used to identify the microstructural origins of the enhanced magnetic properties and study the microstructures of combined reaction processed FePd permanent magnet alloys. Support from NSF (DMR-0094213) with Dr. Murthy as program manager is acknowledged.

3:30 PM Invited
Electrolytic Graining of Aluminum Lithographic Printing Plates: *Chao-Sung Lin*[1]; Gee-Chen Chiou[1]; [1]Da-Yeh University, Dept. of Mechl. Eng., 112 Shan-jiau Rd., Da-Tsuen, Chang-hwa 51505 Taiwan

AA1050 Al lithographic plates were electrograined in nitric and hydrochloric acids using various ac current waveforms. Detailed etch pit morphology and etch film microstructure were characterized by cross-sectional transmission electron microscopy (TEM). After electrograining, an etch film deposited on the Al surface and masked its real topography, which exhibited a pitted surface after removing the etch film. The etch film morphology strongly depended on the electrolyte composition, and the frequency and waveform of the ac current. For example, the etch film formed in nitric acid exhibited a layered structure, whereas that formed in hydrochloric acid was a single layer. Moreover, the layer thickness of the etch film decreased with increasing frequency and anodic/cathodic charge ratio of the ac current, indicating that the etch film formed during the cathodic half cycle. The layered etch film thus developed when the etch film overlaying the aluminum surface underwent dissolution/peeling during successive cycles.

4:00 PM
The Role of Analytical Electron Imaging in Semiconductor Device Development and Manufacturing: *Jerzy Gazda*[1]; Peter Liu[1]; [1]Advanced Micro Devices Corporation, Process Characterization & Analytl. Lab., Austin Wafer Fab Div., MS-613, 5204 E. Ben White Blvd., Austin, TX 78741 USA

In this overview we will show examples of the three most important roles of electron and ion microscopy to semiconductor device manufacturing. We will illustrate the current role of SEM tools in Fab manufacturing process verifications, concentrating on discussion of challenges faced by this technique when used for inspections of 65 nm-node and smaller devices. Secondly, we will illustrate the role of focused ion beam (FIB) tools in defect inspection and specimen preparation for SEM and TEM. Finally, we will discuss the currently occurring changes affecting the role of TEM-based techniques as they move from predominantly defect inspection to a larger role in thin film metrology.

4:20 PM
Field-Driven In-Situ TEM Observations of Domain Boundary Cracking in Relaxor-Based Piezoelectric Crystals: *J. K. Shang*[1]; X. Tan[1]; Z. Xu[2]; [1]University of Illinois at Urbana-Champaign, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA; [2]City University of Hong Kong, Dept. of Physics & Matls. Sci., Tat Chee Ave., Kowloon, Hong Kong China

Relaxor-based piezoelectric crystals have recently received much attention because of their unusually large piezoelectric strains. However, these crystals are highly susceptible to field-induced cracking after repeated electric cycling.In this study, micromechanisms of electric-field induced cracking were examined in relaxor-based piezoelectric crystals by in-situ transmission electron microscopy and in-situ polarized optical microscopy. Field-induced cracking of domain boundaries was observed as incompatible piezoelectric deformations developed in the domains of different polarizations. Under repeated bipolar electric cycling, domain colonies developed near the crack tip and electric fatigue crack grew along the colony boundaries.

4:40 PM
Observation of GeO2 Redistribution in the Oxidized Poly-Si1-xGex Films by Electron Beam Irradiation: *Han-Byul Kang*[1]; Jun-Ho Lee[1]; Cheol-Woong Yang[1]; [1]Sungkyunkwan University, Sch. of Metallurgl. & Matls. Eng., 300 Chunchun-dong, Jangan-Gu, Suwon, Kyunggi-Do 440-746 Korea

We have observed the irradiation effect in oxidized poly-Si1-xGex films under electron induced in the Transmission Electron Microscope. Poly-Si1-xGex films with various compositions and about 1000≈ in thickness were deposited on Si wafers. The oxidation were carried out in a conventional tube furnace at 800°. Cross sectional samples of oxidized poly-Si1-xGex films were irradiated with LaB6 cathode operated in the TEM. In order to analyze the redistribution of elements, we used the Energy Dispersive X-ray Spectroscopy. The amount of current densities measured by GATAN slow scan CCD camera. After irradiation, we can know that the changing of the GeO2 distribution in the oxide layer. For the oxidized poly-Si0.6Ge0.4 films, the accumulation of GeO2 was observed at the surface. And, for the oxidized poly-Si0.4Ge0.6 films, the crystallization of GeO2 was occurred in the oxide layer. Ge lattice fringes and twinning were observed in the oxide layer.

5:00 PM
Electron Backscatter Diffraction for Studies of Localized Deformation: *R. H. Geiss*[1]; R. R. Keller[1]; [1]NIST, Matls. Reliability Div., Boulder, CO 80305 USA

Electron backscatter diffraction (EBSD) was used to study localized deformation in constrained volume materials. We will present a study of deformation in narrow aluminum interconnects after low frequency, high current density AC cycling. Joule heating and differential thermal expansion caused cyclic thermal straining, resulting in thermomechanical fatigue. By quasi in situ AC testing, we determined the evolution of the crystallography of all grains and boundaries in the lines, allowing us to formulate a mechanistic understanding of the deformation process, including slip line formation and grain growth. In a second study, we present an analysis of diffraction patterns from selectively oxidized, multilayered AlGaAs/GaAs structures. Elastic strains accompanying AlGaAs oxidation were characterized by EBSD. Image quality maps revealed the resulting strain field about oxide growth fronts which compared with FE simulations. Quantitative strain measurements were made comparing diffraction band width measurements of processed images.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Alloys Properties

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday PM Room: Solana
March 5, 2003 Location: San Diego Marriott Hotel

Session Chairs: Shigeatsu Nakazawa, Tohoku University, Dept. of Metall., Sendai 980-8579 Japan; Adolf Mikula, Institute of Iorganic Chemistry, Waehringerstrasse 42, Vienna A-1090 Austria

2:00 PM Keynote
ZincñA Possible Component in Lead-Free Solders: *Adolf Mikula*[1]; [1]Institute of Iorganic Chemistry, Waehringerstrasse 42, Vienna A-1090 Austria

Lead-tin solders are commonly used in electronic packaging due to their unique combination of electrical, chemical, physical, thermal and mechanical properties. Since lead and lead containing alloys cause great environmental concern and health hazards it is necessary to replace lead in solder materials. One of the main driving forces to eliminate lead in solder joints is the fact that the disposal of great amount of electronic equipment in landfills can cause lead to leach out and contain the underground water and subsequently find its way into the human body. Besides environmental concerns also new technological developments require new solders with a variety of improved or new properties. The miniaturisation of electronic devices need smaller and smaller interconnections and a reliable joining must be achieved with smaller volumes of solder materials. In most cases these new materials will be ternary tin or indium alloys. The second element could be Bi, Cu, Ag, and Au and as third elements Zn, Ni, Sb, Al and P must be considered. All these elements have an influenze on certain properties. Small amount of zinc will improve in most cases the mechanical properties and the strength of such solders. We investigated the thermodynamic properties and the oxidation behavior of several ternary zinc systems: Al-Sn-Zn, Cu-In-Zn, Ag-Sn-Zn and In-Sn-Zn.

2:35 PM
Activity Measurement of the Constituents in Molten Sn-Ag-In and Sn-Zn-Mg Ternary Lead Free Solder Alloys by Mass Spectrometry: *Takahiro Miki*[1]; Naotaka Ogawa[2]; Tetsuya Nagasaka[1]; Mitsutaka Hino[1]; [1]Tohoku University, Dept. of Metall., Grad. Sch. of Eng., Aoba-yama 02, Aoba-ku, Sendai 980-8579 Japan; [2]Tohoku University (Now at NKK Corporation), Sendai 980-8579 Japan

The increase of demand for smaller and lighter portable electronic devices has made interconnecting densities and packaging technologies more important. Soldering material widely used is Sn-Pb alloy, which have low melting point and excellent electrical, strength properties and wettability. However, Pb is one of the toxic elements, which is undesirable due to environmental and safety reasons, thus Pb-free alternative alloy is preferred for new soldering material. Sn-Ag and Sn-Zn based alloys were viewed as very promising candidates, among many potential substitutes. Addition of third element to these alloys will decrease the melting point of the alloy. Hence, Sn-Ag-In and Sn-Zn-Mg alloys are expected to be suitable for replacing Sn-Pb solder alloy. In order to design new Pb-free soldering materials, precise understanding of thermodynamic properties and phase diagrams for alloy systems are crucial. In the present work, ion current ratios of Ag to In and Mg to Zn were measured for Sn-Ag-In and Sn-Zn-Mg alloy by mass spectrometry, respectively. Also, the authors reviewed the thermodynamic properties of terminal binary alloys determined by other researchers and evaluated a thermodynamic function to express the excess Gibbs free energy of each binary alloy. Thermodynamic function to express the excess Gibbs free energy of liquid Sn-Ag-In and Sn-Zn-Mg ternary alloy were determined, utilizing the assessed Gibbs free energy of terminal binary alloys with the measured ion current ratios using mass spectrometer.

3:00 PM
Effect of Additives on Viscosity of Molten Nickel Base Alloys: *Yuzuru Sato*[1]; Koji Sugisawa[2]; Daisuke Aoki[2]; Masayoshi Hoshi[2]; Jong-Il Kim[2]; Tsutomu Yamamura[2]; [1]Tohoku University, Dept. of Metall., Aramaki aza Aoba, Sendai 980-8579 Japan; [2]Inha Technical College, Dept. of Metallurgl. Eng., Inchon 402-752 Korea

Viscosity has been measured for various binary alloys of nickel with iron, cobalt and chromium to clarify the behavior of viscous flow of molten nickel alloys. Entire concentration range for iron and cobalt, and 0-60at% for chromium were studied. Method for the measurement was an oscillating cup viscometry using an alumina crucible that was precisely machined. Temperature was up to about 1880K for all the measurements. A furnace used was carefully controlled to keep best temperature uniformity that was within 0.5K for entire length of the crucible to prevent the convection in the melt. All the results represented good Arrhenius relationship although some measurements included overcooled temperatures. The isothermal viscosities increased monotonously with an addition of iron and chromium into nickel. On the other hand, the addition of cobalt showed slight decrease, and then viscosity increases in the progress of addition of cobalt.

3:25 PM
Microstructure Analysis of ZA Alloy Rod Directionally Solidified by Heated Mold Continuous Casting: *Ying Ma*[1]; Yuan Hao[1]; [1]Gansu University of Technology, Matls. Sci. & Eng., 85 Langongping Rd., Lanzhou, Gansu 730050 China

The as-cast and heat treatment microstructure of ZA alloy rod directionally solidified by continuous casting has been analyzed. The results show that the microstructure of the ZA alloy lines is the parallel directional dendritic columnar crystal. Every dendritic crystal of eutectic alloy ZA5 is composed of many layer eutectic , and Á phase. The microstructure of hyper eutectic ZA alloys is primary dendritic crystal and interdendritic eutectic structure. The primary phase of ZA8 and ZA12 is , phase, but the primary phase of ZA22 and ZA27 is · phase.

3:50 PM Break

4:10 PM Invited
Activities of Bi and In in the Bi-In Liquid Alloy Measured by Using Vacuum-Sealed Quartz Cell/Atomic Absorption Spectrophotometer Combination: *Shigeatsu Nakazawa*[1]; Minoru Sunada[2]; Takeshi Azakami[3]; Tetsuya Nagasaka[1]; [1]Tohoku University, Dept. of Metall., Grad. Sch. of Eng., Aoba-yama 02, Sendai 980-8579 Japan; [2]Sunada Kogyo Company, Ltd., Kosugi 406, Tonami 939-1357 Japan; [3]Saitama Institute of Technology, Dept. of Matls. Sci. & Eng., Grad. Sch. of Eng., 1690 Fusaiji, Okabe-machi, Osatogun 369-0293 Japan

Activities of Bi and In in the Bi-In liquid alloy were measured over the whole range of composition at the temperature from 850K to 1050K. An alloy was vacuum-sealed in a quartz cell and heated at the temperature of interest. The absorption for 307nm radiation from Bi lamp was measured for Bi atom vapor in the cell. By heating a pure metal as a standard and measuring the absorbance as a function of the temperature, an analytical curve for Bi atom vapor was constructed and used for conversion of the absorbance to the vapor density. Bi activity was determined as the ratio of the Bi atom vapor density over the alloy to that over a pure metal. The same procedure was applied to In vapor and In activity was determined independently of Bi. Results were used to reevaluate the thermodynamics of the Bi-In system.

4:40 PM
Thermodynamic Properties of Several Ternary Zinc Alloys: Adolf Mikula[1]; *Sabine Knott*[1]; [1]University of Vienna, Inst. f‚r Anorganische Chemie, W‰hringerstrasse 42, Vienna A-1090 Austria

In this presentation I would like to show the importance of good, reliable and consistent thermodynamic data for industry and for research. The thermodynamic properties of several ternary liquid zinc alloys were determined using emf and calorimetric measurements. The Au-Sn-Zn, Ag-Sn-Zn, Cu-In-Zn and Al-Sn-Zn alloys were investigated because they are possible candidates for new lead-free solder materials. In these systems the thermodynamic data can be used to improve phase diagram calculations and will be helpful for some theoretical models to improve the calculation of physical or mechanical properties. In the Au-Sb-Zn, Ag-Sb-Zn and Cu-Sb-Zn the temperature dependence of the thermodynamic data was used to look for the existence of Chemical Short Range Order (CSRO) in the melt of these alloys.

5:05 PM
Defect Formation and Mechanism of ZA Alloy Made in Continuous Casting by Heated Mold: *Ying Ma*[1]; Feng-yun Yan[1]; Hongjun Liu[2]; [1]Gansu University of Technology, Sch. of Matls. Sci. & Eng., Lanzhou 730050 China; [2]Huazhong University of Science and Technology, Sch. of Matls. Sci. & Eng., Wuhan 430074 China

The continuous directional solidification technique of five kinds of special ZA alloys with eutectic, peritectic and eutectoid transformation under the condition of continuous casting by heated mold was studies. The mechanism of surface defects appearing in ZA alloy line was discussed and the structure of some defects were analyzed by SEM. The results show that only when the fitting of various technique factors, pressure head, outlet temperature, pulling speed and cooling condition is reasonable under a certain range, can the ZA alloy smooth line be continuously pulled out. Unreasonable technique will result in hot tear, rough surface, mush outlet, leaking and other defects. The shape and location of solid-liquid zone have the importance influence on the forming of above defects. When the solid-liquid zone is located in the mould outlet or inner mould, the surface of solidification will protrude into the inner mould. If the left liquid cannot counteract the solidification contract, the rough surface will appear. If the solidification surface further into the mould, the ingot in mould will have a big friction force when pulling and form hot tear. When the solid-liquid zone is moved out of mould, the solidification surface will turn into a plane, which made it easy to form leaking. The vibration or unstable operation will make the ingot winding during pulling.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Materials Processing Technologies: General II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday PM Room: Point Loma
March 5, 2003 Location: San Diego Marriott Hotel

Session Chair: Thomas P. Battle, Dupont, Edge Moor, DE 19809 USA

2:00 PM Invited
Development of Closed Recycling Process for Low Grade Scrap of Al-Based Composite Materials: *Hiroyuki Sano*[1]; Shinichi Kato[2]; Tatsuya Motomura[2]; Toshiharu Fujisawa[1]; [1]Nagoya University, Rsrch. Ctr. for Adv. Waste & Emission Mgt., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan; [2]Nagoya University, Dept. of Matls. Sci. & Eng., Furo-cho, Chikusa-ku, Nagoya 464-8603 Japan

Closed recycling process for low grade scrap of Al-based composite materials was developed. Flux treatment with water soluble halide is a key technique in this process. In the present work, flux treatment conditions were discussed from the viewpoint of separation and recovery of matrix, reinforce and flux materials. Optimum separation condition was obtained for the NaCl-KCl-KF flux treatment. Recovered aluminum alloy has enough quality for using various materials. Used flux is recyclable for the process by adding a small amount of KF.

2:35 PM
Two-Way Shape Memory Effect of Ferromagnetic Ni-Mn-Ga Sputter-Deposited Films: *Makoto Ohtsuka*[1]; Minoru Matsumoto[1]; Kimio Itagaki[1]; [1]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., 2-1-1, Katahira, Aoba-ku, Sendai 980-8577 Japan

The ternary intermetallic compound Ni_2MnGa is an intelligent material, which has a shape memory effect (SME) and a ferromagnetic property. Use of shape memory alloy films for an actuator of micromachines is very attractive because of its large recovery force. The Ni-Mn-Ga films with a thickness of nearly 5 μm were deposited with a radio-frequency magnetron sputtering apparatus. They were heat-treated at 1073 K for 36 ks for homogenization and ordering. The purpose of the present study is to investigate the effects of fabrication conditions on the properties of the films such as chemical composition, crystal structure, microstructure and transformation temperature. Furthermore the effect of plastic deformation and of constraint aging on SME is also investigated. The two-way SME was shown after plastic deformation. The intensity of X-ray diffraction peaks from stress-induced martensitic phase was found. The constraint-aged films also showed the two-way SME by thermal cycling.

3:00 PM
Fabrication of BaNd2Ti4O12/Bi4Ti3O12/BaNd2Ti4O12 Laminated Ceramics by Spark Plasma Sintering: *Yong Jun Wu*[1]; Naofumi Uekawa[1]; Kazuyuki Kakegawa[1]; [1]Chiba University, Ctr. for Frontier Elect. & Photonics, Dept. of Matl. Tech., Chiba 263-8522 Japan

Laminated ceramics have received wide scientific and commercial attention because its properties, such as mechanical properties and electrical properties, can be tailored by adjusting the structure, thickness and composition of the different layers. For example, ferroelectric Bi4Ti3O12 (BIT) which has a positive temperature coefficient of dielectric constant (te) can be used to modify the negative te of typical microwave dielectric ceramic BaNd2Ti4O12 (BNT). However, BaNd2Ti4O12/Bi4Ti3O12/BaNd2Ti4O12 (BNT/BIT/BNT) laminated ceramics could not be prepared by the general method because the optimal sintering temperature of BaNd2Ti4O12 ceramic (around 1350°C) was much higher than that of Bi4Ti3O12 ceramic (around 1100°C). In this research, a new method of spark plasma sintering (SPS) combined with post-sintering was successfully proposed to prepare BNT/BIT/BNT laminated ceramics. It was divided into three steps. In first step, the BaNd2Ti4O12 ceramics were prepared by conventional sintering method. In second step, the calcined and prepressed Bi4Ti3O12 powders were sandwiched between BaNd2Ti4O12 ceramic pellets and spark plasma sintered at 900°C for 10min to synthesize BNT/BIT/BNT composite ceramics. In last step, a post-sintering was employed to re-oxidize the partially reduced BNT/BIT/BNT composite ceramics. The results of scanning electron microscopy (SEM) and electron probe micro-analysis (EPMA) showed that BNT layer and BIT layer were well bonded and no significant interfacial infiltration between them was observed. The sandwiched BNT/BIT/BNT ceramic with 10.7% BIT in volume has a dielectric constant of 92.8, a low dielectric loss of 0.0068 and a small temperature coefficient of dielectric constant of 35 ppm°C at 1 MHz.

3:25 PM
Preparation of Infrared Materials from Ultrafine Quartz Powder: *Huaming Yang*[1]; Jianhong Chao[1]; Weiqin Ao[1]; Guanzhou Qiu[1]; [1]Central South University, Dept. of Minl. Eng., Changsha 410083 China

Infrared material is a special material widely used in military, biological engineering and chemical industry, but the problems, such as unstable property, high cost and higher thermal expansion coefficient, seriously restrict the development and wide application of traditional infrared materials with SiC or ZrSiO4 matrix. Ultrafine quartz powder was used to prepare infrared material by Hot-pressing technology. The aim of the paper is to investigate the thermal expansivity and effects of SiO2 size, addition of Al2O3 or Fe2O3 on infrared emissivity of quartz matrix infrared material(QMIM) in 8-25μm wavelength range. The results indicate that 89% of infrared emissivity and 0.4◊10-5/ of thermal expansion coefficient of QMIM can be obtained. Affecting mechanism of Al2O3 and Fe2O3 on infrared emissivity of QMIM were also discussed.

3:50 PM Break

4:10 PM
Deposition of Multilayered Titanium Thin Film by Nd: YAG Laser Ablation: *Takahiro Nakamura*[1]; Hideyuki Takahashi[2]; Katsutoshi Yamamoto[3]; Nobuaki Sato[3]; Atsushi Muramatsu[3]; Eiichiro Matsubara[2]; [1]Tohoku University, Dept. of Matl. Sci., Katahira 2-1-1, Aoba-ku, Sendai 980-8577 Japan; [2]Tohoku University, Inst. for Matl. Rsrch., Katahira 2-1-1, Aobaku, Sendai 980-8577 Japan; [3]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls., Katahira 2-1-1, Aobaku, Sendai 980-8577 Japan

The thin film of TiO2 by means of laser ablation has the nanostructure and would appear the new properties. Multilayered TiO2/Ti thin film on quartz substrate was synthesized by laser ablation of Ti and TiO2 targets using Nd: YAG pulse laser under the established conditions. Namely, Ti and TiO2 film was successively deposited on the substrates by the irradiation using laser light of the energy 500 and 400 mJ and the repetition rate 50 Hz for 15 and 6 sec with Ti and TiO2

target, respectively, in a high vacuum reaction chamber under the oxygen pressure of 10-8 Torr. Thicknesses of Ti and TiO2 in as-prepared film are 150 and 210 nm with growth rates of about 10 and 35 nm/sec, respectively. Preparation of TiS2/TiO2/Ti multilayered film was also examined in the presence of oxygen and H2S (or CS2) with Ti and/or TiO2 target.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Aqueous and Electrochemical Processing V

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday PM Room: Pacific
March 5, 2003 Location: San Diego Marriott Hotel

Session Chairs: Paulo von Kr¸ger, Federal University of Ouro Preto, Minas Gerais Brazil; U. B. Pal, Boston University, Dept. of Mfg. Eng., Boston, MA 02446 USA

2:00 PM Invited
Zirconia-Based Inert Anodes for Green Synthesis of Metals and Alloys: C. P. Manning[1]; A. Krishnan[1]; *U. B. Pal*[1]; [1]Boston University, Dept. of Mfg. Eng., Boston, MA 02446 USA

The research work demonstrates the technical viability of employing zirconia-based inert anodes for environmentally sound and cost-effective production of metals such as magnesium, tantalum, aluminum, etc., directly from their oxides. The inert anode consists of the oxygen-ion-conducting stabilized zirconia membrane in intimate contact on one side with a catalytically active electronic phase. The opposite (other) side of the zirconia membrane is placed in contact with an ionically conducting solvent phase containing the oxides of the desired metals. An inert cathode is placed in the solvent and an appropriate electric potential is applied between the electrodes to synthesize the metals from their oxides in the solvent. The full-benefit of the process can be realized if it is conducted at temperatures between 1200-1400∞C. At these temperatures the ohmic resistance drop across the stabilized zirconia membrane are low and therefore high current densities on the order of 1 A/cm2 or greater can be obtained. In addition, the process efficiency can be further increased by directly reforming hydrocarbon fuel over the anode. It should be noted that several attempts have been made earlier to employ this concept at temperatures below 1000∞C. However, these efforts have not been successful mainly because sufficiently high current densities could not be obtained through the zirconia membrane. This paper reports the recent progress of a continuing laboratory-scale investigation involving different types of zirconia-based inert anodes employed at temperatures between 1200-1400∞C. The topics covered include: stability of the zirconia membrane in the selected molten solvent (flux), volatility of the flux, potentiodynamic sweeps, electrolysis experiments, and analysis of the metals produced.

2:35 PM Invited
Hydrometallurgical Process for Recycle of Spent Nickel-Metal Hydride Secondary Battery: *Toshihiro Kuzuya*[1]; Takayuki Naitou[2]; Hiroyuki Sano[1]; Toshiharu Fujisawa[1]; [1]Nagoya University, Rsrch. Ctr. for Adv. Waste & Emission Mgmt., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan; [2]Nagoya University, Dept. of Matls. Sci. & Eng., Furo-cho, Chikusa-ku, Nagoya, Aichi 464-8603 Japan

A hydrometallurgical process has been developed for recovery of metal values such as Co, Ni and rare earths from the electrode materials of spent nickel metal-hydride(Ni-MH) secondary battery. MmNi5 intermetallic compound could be separated from the electrode materials mixture by sedimentation. A typical chemical composition of MmNi5 was approximately, in mass%, 56.0%Ni, 13.4%Ce, 10.6%La and 7.9%Co. The time dependency of leaching intermetallic compound with sulfuric acid solution was examined with processing factors such as sulfuric acid concentration, temperature and agitation intensity. The leaching of rare earth metals proceeded very rapidly, reaching completion in less than 1.8ks. On the other hand, the slow leaching of nickel was observed. The percent of nickel leached at 328K could only reach about 70%.

3:05 PM
Rare Earth Separation and Recycling Process Using Rare Earth Chloride: *Testsuya Uda*[1]; Masahiro Hirasawa[2]; [1]California Institute of Technology, Matls. Sci., MC138-78, Pasadena, CA 91125 USA; [2]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls. (IMRAM), 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan

We review our recent achievements of rare earth separation technique and new process idea for rare earth magnet recycling. (1) Binary chloride mixtures of rare earths were separated by new selective reduction-vacuum distillation process. According to our experimental results, apparent separation factors were 8.1 for Pr-Nd chloride mixture and 570 for Nd-Sm chloride mixture. These values are higher than conventional solvent extraction methods. (2) A study of recycling process of magnet sludge was carried out. The rare earths in the neodymium magnet sludge were extracted by chlorination with FeCl2. An activated carbon was used as a de-oxidation reagent. Metallic iron in the sludge was not chlorinated because the iron monochloride is not stable. The extracted rare earth chlorides were easily separated from Fe-alloy and the excess of FeCl2 by vacuum distillation. 96% of neodymium and 94% of dysprosium in the sludge were extracted into chloride phase. By the vacuum distillation, a mixture of neodymium and dysprosi um trichlorides of 99.2% purity was recovered. It was confirmed that the rare earth chlorides were converted to the corresponding oxides by a pyrohydrolysis reaction accompanied by gaseous HCl formation. The HCl gas can chlorinate metallic iron to FeCl2. Therefore, a new recycling process for rare earth magnet waste can be realized as a chlorine circulation type process. During the process, only carbon and water are consumed, and there are no toxic pollutants. Moreover, the obtained rare earth oxide can be directly used as raw material in the conventional oxide electrolysis process.

3:30 PM
A Novel Application of Electrorefining in a Membrane Cell to Reclaim Zinc from the Bottom Dross of Hot Dip Galvanization: *Levente Becze*[1]; Tam·s I. Tˆrˆk[1]; [1]University of Miskolc, Dept. of Nonferrous Metall., Miskolc-Egyetemvaros, Miskolc 3515 Hungary

The zinc bottom dross is obtained as a by-product from hot dip galvanizing operations and contains at least 92% zinc, which is recycled mainly by pyrometallurgical processes. The overall costs of such a treatment, however, are often relatively high, and the very strict environmental protection regulations might also cause additional difficulties for the high temperature procedures in the future. Therefore, a novel hydo-eletrometallurgical process has been developed in our laboratory, which could reclaim zinc of high purity, while attempting to achieve low energy consumption and making allowances for all the environmental restrictions. This aqueous processing technique, based on electrolytic refining in a cell equipped with anion-exchange membrane to separate the anode and cathode compartments, allows the indirect purification of the anolyte. Zinc is dissolved from the anode along with some of the impurity elements, while the intermetallic compounds (Fe-Zn, Fe-Al-Zn) are accumulated in the anode slime.

3:55 PM Break

4:05 PM Invited
Fundamental and Applied Research on Tin Electrorefining, Employing Stainless Steel Cathodes: *Paulo von Kr¸ger*[1]; Erivelto Luis de Souza[2]; [1]Federal University of Ouro Preto, Dept. of Met. Eng., Sch. of Mines, Minas Gerais Brazil; [2]FundaÁ„o Gorceix, Ouro Preto, Minas Gerais Brazil

This paper deals with the cell design and operation of a tin electrorefing 1:5 pilot plant. The purpose was the fitting of the basic parameters to be used in a full scale tankhouse, for a major Brazilian tin producer. The main difference was the use of permanent, stainless steel cathodes, instead of the conventional tin starting sheets. The results on both product quality and operating parameters are discussed.

4:40 PM
Separation of Elements in Stainless Steel by Electrorefining Process: *Toshihide Takenaka*[1]; Masahiro Kawakami[1]; Masao Kawaguchi[1]; [1]Toyohashi University of Technology, Dept. of Produc-

tion Sys. Eng., Hibarigaoka 1-1, Tempaku-cho, Toyohashi, Aichi-Prefecture 441-8580 Japan

Stainless steel is one of the major materials in a nuclear reactor. The management of radioactivated stainless steel will become an important subject in the near future. The half-life of radioactive elements of Fe and Cr are short in general, whereas those of Ni is very long. Therefore, the separation of the elements in stainless steel should be effective to reduce the management cost of the waste radioactivated stainless steel. In this study, the separation of the elements in stainless steel was investigated by an electrorefining technique in an aqueous solution. The electrochemical reactions of the elements in stainless steel were studied by voltammetry, and potentio-static electrolysis was carried out. A plate of austenitic stainless steel, SUS304, was used as an anode and a cathode. The anode was dissolved electrochemically with good anodic current efficiency in a chloride solution, while the anodic dissolution did not occur in a sulfate solution. The smaller the anodic overpotential was, the lower the Ni concentration in the solution was after electrolysis. The residue where Ni was concentrated was found on the anode. At the cathode, metallic electrodeposit was obtained by potentio-static electrolysis, and only a small amount of H2 gas was evolved simultaneously. The electrodeposit at the cathode consisted of Fe and Cr mainly, and the Ni content was less than 0.5wt% under the suitable condition. It is concluded that the electrorefining technique in a chloride solution can be applied for separation of the elements of stainless steel.

5:05 PM

Preparation of Fibrous Nickel Oxide Powder by Wet Chemical Precipitation Combined with Pyrolysis: *Zhang Chuanfu*[1]; Jing Zhan[1]; Xueyi Guo[1]; Masazumi Okido[2]; [1]Central South University, Col. of Metallurgl. Sci. & Eng., Yuelu Dist., Changsha, Hunan 410083 China; [2]Nagoya University, Ctr. Integrated Rsrch. in Sci. & Eng., Furocho, Chikusa-ku, Nagoya 464-8603 Japan

In this study, the process of wet chemical precipitation of nickel ions from the solution with oxalic acid followed with precursor pyrolysis has been employed to prepare the fibrous NiO powder. The influences of the pH value, reaction temperature, concentrations of reactants, the order of reagents adding and the addition of surfactant on the morphology of precursor were investigated for the wet chemical precipitation stage; and that of the morphology of precursor, temperature and time for decomposition, air flow, and the rate for heating on the morphology, particle size and specific surface area of the final obtained NiO particles during pyrolysis were addressed respectively. The results show that the fibrous NiO particles with 100~120 for axis-ratios and about 6.0 m2/g for specific surface area can be obtained under certain experimental conditions. Spherical nanometer NiO powder particles were obtained after further grinding of fibrous nickel oxide particles.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Non-Ferrous Production Technologies and Industrial Practice: Copper III

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors

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

Wednesday PM Room: Santa Rosa
March 5, 2003 Location: San Diego Marriott Hotel

Session Chairs: Lauri Holappa, Helsinki University of Technology, Lab. of Metall. FIN-02015 HUT Finland; F. Kongoli, FLOGEN Technologies Inc., Montreal, Quebec Canada

2:00 PM

Effect of Oxygen Enrichment on the El Teniente Converter Productivity at Ilo Smelter, Per*: *Jose Bengoa*[1]; Jose Palacios[2]; Mario Sanchez[3]; [1]Southern Peru Copper Company, Ilo Smelter, PO Box 35, Ilo Peru; [2]University of Atacama, Dept. Met. Eng., PO Box 240, Copiapo Chile; [3]University of Concepcion, Dept. Met. Eng., PO Box 53-C, ConcepciÛn Chile

Low metal prices and environmental restrictions are always a challenge for copper smelters operation and its productivity. Thus, in order to reduce production costs and control gas emissions, improvements of El Teniente Converter operation were made at Ilo Smelter Plant of Southern Peru Copper Co., Ilo, Peru, and the main objective was to increase its capacity for smelting copper concentrates without affecting normal operation. The present work corresponds to a recent study conducted during the normal industrial operation of El Teniente Converter in this plant and, it shows the effect of increasing air oxygen enrichment from 28 to 34% on its productivity. The results show an increasing of concentrate smelting rate from 800 to 1370 tpd and hence a notorious improving in copper production, and practically no effect on the refractory consumption. Additionally, the increment of the concentrate throughput increases SO2 concentration in the exhausting gas and the sulfuric acid production, showing the effort done by this operation in order to accomplish the environmental Peruvian regulation.

2:30 PM

Oxidation of Copper at Different Temperatures: *Gabriel Plascencia*[1]; Torstein Utigard[1]; [1]University of Toronto, Matls. Sci. & Eng., 184 College St., Toronto, Ontario M5S 3E4 Canada

Copper was oxidized in the temperature range from 300 to 1000∫C under different partial pressures of oxygen. In the range from 300 to 500∫C, copper oxidizes following the logarithmic rate law; while in the range from 600 to 1000∫C copper oxidizes following the parabolic rate law. Transition from logarithmic to parabolic growth occurs at 525∫C. X-ray diffraction was performed on oxide scale to account for the CuO/Cu2O ratio at different temperatures. Activation energies found in this work are in good agreement with those already reported.

2:55 PM

YCC ISASMELT ProjectñThe First Chinese ISASMELT Furnace: *Yun Li*[1]; Philip Arthur[2]; [1]Yunnan Copper Corporation, Wangjiaqiao, Western Hill Dist., Kunming, Yunnan 650102 China; [2]MIM Process Technologies, ISASMELT, Level 2, 87 Wickham Terrace, Brisbane, QLD 4000 Australia

In 1999 Yunnan Copper Corporation made a decision to carry out technical modification on the copper smelter and change the existing sinter plant/electric furnace process to ISASMELT/electric furnace slag cleaning. The ISASMELT furnace has been running smoothly since heatup in May 2002. The design capacity for the ISASMELT furnace is 600,000 tonnes of dry copper concentrate per year. The main aim of the project was to improve environmental performance and decrease energy consumption. Generally the project is going smoothly at the current stage, due to selection of reliable and successful technology. Good preparation work was done. The extensive training program for key people in Mount Isa Copper Smelter improved the technical peopleís understanding of the process greatly and ensured successful hand-over of the process technology. The paper describes the plant layout and initial operating data.

3:20 PM

Modelling of Chalcocite Concentrate Flash Smelting: *Zdzislaw Miczkowski*[1]; Jozef Czernecki[1]; Zbigniew Smieszek[1]; [1]Institute of Non-Ferrous Metals, SowiÔskiego 5, Gliwice 44-100 Poland

KGHM Polska Miedz S.A. applied single-stage process for copper production from chalcocite concentrates in flash furnace. The paper contains model of complex oxidation process of single concentrate grain smelted at KGHM. Description takes into consideration main chemical reactions, heat and mass transfer phenomena, inside the grain as well as between the grain and gas environment. Two zones of process course inside the grain were distinguished. The first zone moves faster within the grain and is caused by increase of temperature inside. Liberation of carbonate and organic carbon occurs there. Second zone penetrates the grain at lower rate. The rate is determined by oxygen presence. Second zone is the place where sulphur is eliminated and vapours of organic compounds are oxidised. On the basis of lab tests results, coefficients characterising oxygen transfer within the grain and determining the rate of chemical reactions were calculated. Model calculations results for single concentrate grain processed in the environment of various oxygen content and model calculations results for concentrate flux smelted in the lab reactor system are presented.

3:45 PM Break

3:55 PM Invited
Copper Converting Versus Steel ConvertingñA Critical Comparison: *Lauri Holappa*[1]; Heikki Jalkanen[1]; [1]Helsinki University of Technology, Lab. of Metall., PO Box 6200, Hut 02015 Finland

Copper converting is mostly performed in Peirce-Smith converters. The process is still very similar to the original one from the late 19th century: a batch process in a horizontal cylindrical reactor with air blowing via tuyeres along the length of the reactor side. This converter is sometimes called Bessemer converter as Bessemer proposed a similar converter to steelmaking. However, finally he came to a vertical pear-shaped converter with air bottom-blowing. In steel converting, a clear continuum can be perceived from air-blowing Bessemer &Thomas processes to oxygen converter processes (LD or BOP) using top-blowing via a lance, to oxygen bottom-blown converters (OBM or Q-BOP) with special tuyeres, and finally to nowadays combined blowing converters utilizing the advances of both top and bottom blowing. In copper converting similar ideas were missing or at least did not lead to large scale success, with the exception of top-blown rotary converters (TBRC or Kaldo) which were used in small extent both for steel and non-ferrous converting. On one respect copper converting has taken a leading position i.e. in the progress of continuous converting. Mitsubishi process as well some new processes like Ausmelt make continuous converting of matte to blister copper by oxygen-air blowing via top lances. A different principle for continuous converting is flash converting by Kennecott-Outokumpu which fully utilizes the exothermic reactions in smelting and converting solid fine matte particles in a reactor shaft similar to Outokumpu matte flash smelting. Although numerous ideas and principles have been tested for continuous steelmaking it seems to be still far from commercial breakthrough. The paper discusses principal physico-chemical and process technical similarities and differences in copper and steel converting. The main processes are reviewed and compared, and the trends are discussed in respect of eventual exchange of knowledge between ferrous and non-ferrous metallurgy.

4:20 PM
Effect of Electric and Magnetic Fields on Metallic Inclusions in a Liquid Slag: *Andrzej Warczok*[1]; *Gabriel Riveros*[1]; [1]Universidad de Chile, Av. Tupper 2069, Santiago 2777 Chile

Liquid slags produced in smelting of copper concentrates contain copper in a dissolved form as well as mechanically entrained inclusions of copper matte. Coalescence and sedimentation of inclusions during slag cleaning plays a key role in copper recovery. Liquid matte inclusion in a molten slag presents a system of metallic conductor in an ionic solution. Constant electric field induces migration of the matte droplet along current lines. Additional phenomena occur in a system under crossed electric and magnetic fields. Electromagnetic buoyancy force acting on the inclusion affects its settling rate. Electromagnetic buoyancy force of insulating and metal spheres has been measured in a liquid synthetic slag. It was found that depolarisation of metal sphere, related to the electrode reactions occurring at the surface, plays an important role in determination of direction and magnitude of the electromagnetic force. On the basis of existing theories and results of measurements the mathematical model, describing the behaviour of metallic inclusions in the liquid slag under the influence of crossed electric and magnetic fields, was developed. Simulation of behaviour of copper matte inclusions under crossed fields showed trajectory of motion in a liquid slag as a function of the slag properties and the inclusion size.

4:45 PM
Factors Affecting the Rate of Copper Reduction During Copper Refining: *Gabriel Riveros*[1]; Andrzej Warczok[1]; Leandro Voisin[1]; Tanai Marin[1]; [1]Universidad de Chile, Av. Tupper 2069, Santiago 2777 Chile

Fire refining of copper consists of two stages oxidation and reduction of copper. Very frequent the capacity of anode furnace is determined by the rate of copper reduction. Analysis of factors affecting the rate of oxygen reduction from a liquid copper with solid carbon or injected hydrocarbons pointed out the role of mass transfer in a liquid copper in final stage of reduction. Experimental results of copper reduction with graphite and injected natural gas in a crucible scale showed two stages of the reduction process with a sharp change of controlling mechanism. Estimated dynamic surface area of gas bubbles based on measured bubble frequency and gas flowrate allowed for determination of reaction constant as a function of gas injection intensity and degree of partial pre-combustion of air/natural gas mix. The variation of reductant utilisation as a function of oxygen concentration and temperature has been evaluated based on the determined kinetic model. Possibilities of intensification of copper reduction in fire refining has been discussed on the basis of obtained kinetic data and analysis of various factors determining the time of reduction.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Iron and Steel Making Fundamentals II

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors
Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Wednesday PM Room: Leucadia
March 5, 2003 Location: San Diego Marriott Hotel

Session Chairs: Fumitaka Tsukihashi, The University of Tokyo, Dept. of Adv. Matls. Sci. Grad. Sch. of Frontier Scis., Tokyo 113-8656 Japan; Mitsutaka Hino, Tohoku University, Dept. of Metall. Grad. Sch. of Eng., Sendai 980-8577 Japan

2:00 PM Invited
Simulation on the Formation, Dripping and Penetration Behavior of Primary Oxide Melt in the Pyrometallurgical Process: *Mitsutaka Hino*[1]; Atsushi Kumano[1]; Kenko Shimizuno[2]; Tetsuya Nagasaka[1]; [1]Tohoku University, Dept. of Metall., Grad. Sch. of Eng., Sendai 980-8579 Japan; [2]Fujitsu Company, Kawasaki Japan

Primary melt, which appears in the ore during heating, plays an important role in many pyrometallurgical processes. For example, Al_2O_3 is known to be enriched in the primary melt which is formed in the lower part of the ironmaking blast furnace, so that such primary melt could possibly penetrate into micro-porosity of sinter ore and result in the harmful effect on the reducibility of the ore. With keeping such background in view, it has been observed the dripping behavior of Fe_tO-CaO-SiO_2-Al_2O_3-MgO slag from the iron funnel, which has simulated the micro-porosity of sinter ore with the wide range of basicity, Al_2O_3, Fe_tO and MgO contents. The effect of MgO on slag dripping behavior has been summarized in this paper. Premelted slag sample was charged on the iron funnel, suspended in the furnace from thermobalance and heated under Ar stream at 4K/min. Dripping of melt from the funnel and slag hold-up were detected by weight change and its temperature was measured. It was found that the hold-up of the MgO free slag increased with increasing Al_2O_3 content, while this trend became weaker with increasing of the basicity. The addition of 2mass% MgO resulted in large increase of hold-up on the most of the conditions. When FetO content was less than 20mass% and the basicity was lower than 0.8, MgO lowered the hold-up even if the slag contained 10mass% Al_2O_3. Every slag hold-up showed the minimum at the basicity of less than unity. This trend became remarkable when the slag contained 2mass% MgO.

2:30 PM Invited
New Reactor Concepts for Direct Coal-Based Continuous Steelmaking: *Noel A. Warner*[1]; [1]The University of Birmingham, Cheml. Eng., Edgbaston, Birmingham B15 2TT UK

Modern steelmaking is based on direct use of oxygen in high intensity batch reactors. A totally new approach is proposed in this paper, based on the view that pursuit of high intensity is unlikely to lead to successful coal-based continuous steelmaking. By substituting carbon dioxide and water vapor as the oxidants rather than using oxygen directly, the intensity is greatly reduced. Accordingly, subsurface formation of carbon monoxide bubbles can be avoided by careful manipulation of the three participating rate processes, gaseous diffusion, interfacial chemical kinetics and liquid phase mass transfer. Steelmaking can then be carried out without explosive ejection of molten droplets and the generation of micro-spray but rather in a controlled and fumeless fashion. The vital component is the introduction of generic melt circulation technology. Three melt circulation loops in series are envisaged. Starting with a composite feed of iron ore fines, pulverized

coal and lime flux, a metallized solid raft floating on molten iron is propelled out of the ironmaking loop onto the first of two downstream steelmaking loops. In the first, a liquid slag is formed, primary decarburization is undertaken not with oxygen but rather with CO2 and H2O and the melt is desulfurized. The semi-product stream enters the third melt circulation loop, where open-channel decarburization is effected along with dephosphorization to yield a low carbon steel product. Alternatively, the melt leaves the last loop to irrigate a packed tower countercurrent to argon at around 100 mbar to continuously produce ultra-low carbon (ULC) steel.

2:55 PM

Kinetics and Morphological Studies of a Carbon Composite Briquette Aiming the Emergent Ironmaking Technologies: *JosÈ Carlos DíAbreu*[1]; JosÈ Henrique Noldin[2]; Karla de Melo Martins[1]; [1]Catholic University, DCMM, Rua Marques de S„o Vicente 225, Sala 542L, G·vea, Rio de Janeiro, RJ 22453-900 Brazil; [2]Catholic University/Tecnored, Ltd., R. Gal. Garzon, 22, 308, Rio de Janeiro, RJ 22470010 Brazil

The first part of this work presents the effects of temperature, gas flowrate and external atmosphere over the reduction rates of a carbon composite briquette (CCB), aiming its use as a burden in some of the new alternate ironmaking technologies, such as Tecnored and RHFs. Conversions were obtained determining the metallization degree, being the pre-exponential factor and the apparent activation energy calculated using the Arrhenius equation. It is reported that raises in temperature, decreasing in N2 flowrate and the use of a CO atmosphere, increase the reaction rate. Into the second part, a morphological study of the iron metallization process during the reduction of these briquettes is presented. The main objective of this investigation was to assess the morphology of the metallic iron formed into the cross section of the samples, for different temperatures and times of reduction, using Optical and SEM images. It was possible to verify that three types of iron morphology occurred: an external, dense and continuous iron layer, and the presence of iron globules and whiskers at the briquetteís core. The measurements of the carbon content on the globules and the external iron layer are also presented.

3:20 PM

Phase Relations During Sintering of Iron Ore and Fluxing Effect of Minor Components: *Florian Kongoli*[1]; Ian McBow[1]; Akira Yazawa[2]; [1]FLOGEN Technologies, Inc., Matls. Tech. Dept., 5757 Decelles, Ste. 511, Montreal, Quebec H3S 2C3 Canada; [2]Tohoku University, 16-32, Niizaka, Aoba-ku, Sendai 981-0934 Japan

The production of a homogeneous self-fluxing sinter is an important step in iron making processes. A good sinter should have good permeability and reducibility and has to keep those characteristics for a certain time. An early melt down of the sinter in the blast furnace, where its solid state reduction is essential, would cause many problems such as low permeability and reductibility. Nevertheless, the important factors that influence these characteristics, such as the chemistry of the sinter and the fluxing effect during sintering and sinter reduction conditions, have not yet been clarified. In todayís new reality where many new minor components such as Al2O3 and MgO enter to the sinter through raw materials, the quantification of the fluxing effects during sintering becomes even more important. In this work, the fluxing effect of many sinter major and minor components has been quantified through the characterization of phase relations in the CaO-FeO-Fe2O3-SiO2 system at sintering conditions and the quantif ication of the effect of Al2O3 and MgO by the means of some practical diagrams which can directly help the optimization of the sintering processes.

3:45 PM Break

4:05 PM Invited

Thermodynamics of Removal of Tramp Elements from Steel Scrap: *Fumitaka Tsukihashi*[1]; [1]The University of Tokyo, Dept. of Adv. Matls. Sci., Grad. Sch. of Frontier Scis., Tokyo 113-8654 Japan

Increasing amounts of steel scrap are being used as resources for steelmaking. Therefore, there has been a growing interest in the removal of harmful tramp elements such as antimony, zinc, tin, arsenic, bismuth, lead and copper from molten steel. The removal of these elements from molten steel by an oxidative treatment is basically impossible and is feasible by using basic fluxes in a reductive refining process. The problem that should be solved is the removal treatment of tramp elements economically in steelmaking process and production of high quality steel by reducing the effect of contaminated tramp elements with satisfying the environmental issues. In this paper, the available thermodynamic data of the tramp elements in molten steel such as the activities of tramp elements, the partition ratio of them between molten steel and sulfide and calcium based fluxes, and the Gibbs free energy of compounds of tramp elements are summarized. Furthermore, the possibility of the removal of tramp elements from molten steel by using various flux systems is thermodynamically estimated by using these thermodynamic data.

4:35 PM Invited

Thermodynamics of Mold Powder: *Hiroyuki Fukuyama*[1]; Kazuhiro Nagata[1]; [1]Tokyo Institute of Technology, Dept. of Chem. & Matls. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

In continuous casting of steel, mold fluxes play some important roles. In particular, horizontal heat transfer from strand to mold significantly affects surface quality of steel. A crystalline layer in slag films yields larger thermal resistance by scattering infrared radiation from steel and forming air gap at the flux/mold interface. Cuspidine (3CaO∑2SiO2∑CaF2) is one of the most important compounds crystallized in slag films during the casting. In order to discuss the crystallization behavior of cuspidine, the present authors have experimentally determined the CaO-SiO2-CaF2 phase diagram around cuspidine. Moreover, the thermodynamic properties of cuspidine are essentially required to discuss the stability field of cuspidine in multi components systems of mold flux. However, no experimental data are currently available on the Gibbs energy of formation of cuspidine. Thus, the present study aims for experimentally determining the Gibbs energy of formation of cuspidine by both electromotive force method and transpiration method.

5:00 PM

Influence of Reduction-Carburization Conditions on the Rate of Iron Carbide Formation: *Abdel-Hady El-Geassy*[1]; Mahmoud Ibrahim Nasr[1]; Mohamd Bahgat Sedikk[1]; [1]Central Metallurgical Research & Development Institute, Iron-Making Div., PO Box 87-Helwan, Cairo Egypt

High grade iron ore fines rejected from the DRI plants were isothermally reduced in H2/CO gas mixtures at 550-850°C. The freshly reduced reaction products were then subsequently carburised in either H2/CO or H2/CO/S. Thermogravimetric analysis technique was used to follow up both of reduction and carburisation reactions as a function of time. The carburised samples were characterized by X-ray phase identification, Mossbaure and carbon and chemical analyses. The influence of temperature, H2:CO ratio in the gas mixtures and carburisation time were intensively investigated and correlated. A conversion extent of iron to Fe3C was taken as a measurable index for the efficiency of carburisation reaction under the different experimental conditions. The presence of sulfur greatly stabilize the Fe3C formation resulting 96% conversion extent in 40%H2/CO mixture at 650°C.