TMS2004

133rd Annual Meeting & Exhibition

The Minerals, Metals & Materials Society welcomes you to the TECHNICAL PROGRAM for the 133rd TMS Annual Meeting & Exhibition, to be held March 14–18, 2004, in Charlotte, North Carolina.

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.

This document comprises

WEDNESDAY'S TECHNICAL PROGRAM

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

See you in Charlotte!

TMS

AIME FOUNDED 1871

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

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The New On-Line TMS Document Center

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Create Your Own Custom Publication
Gather individual papers and articles from TMS proceedings volumes, *JOM*, *Journal of Electronic Materials*, and *Metallurgical and Materials Transactions A* and *B* to create a one-of-a-kind publication that meets your needs. TMS will compile them in either a softcover book or on a CD-ROM—it's your choice.

Coming in 2004: *TMS Letters*
TMS Letters is a peer-reviewed, on-line-only journal featuring technical updates of hitherto unpublished research presented at TMS meetings. Available free-of-charge to TMS members (and by subscription to nonmembers), the journal comprises two-page technical updates, including text and graphics.
Visit the TMS Document Center for additional information about *TMS Letters*!

See it for yourself!

Visit the new TMS Document Center today.

http://doc.tms.org

AN INTERNATIONAL EVENT IN SCIENCE AND ENGINEERING

During the week of March 14–18, the 2004 TMS Annual Meeting & Exhibition will host approximately 4,000 science and engineering professionals, representing more than 70 different countries. They are convening at the Charlotte Convention Center 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
- International Titanium Association
- International Magnesium Association
- National Science Foundation
- TMS Public & Governmental Affairs Committee

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

- **Tour** the Exhibition of more than 160 Companies Displaying New Products and Services
- **Attend** Special Lectures and Tutorials
- **Participate** in Short Courses on Metal Matrix Composites, Introduction to Nanomanufacturing and Nanotechnology, Technology Transfer Seminar, Smelter Grade Alumina from the Smelting Perspective and Computational Modelling for the Materials Professional
- **Enjoy** Special Luncheons, Dinners, and Social Functions, including events honoring Didier de Fontaine, R.J. Arsenault, A.L. Roytburd and Roger D. Doherty
- **Network** Extensively
- **Experience** the Charm and Amenities of Charlotte

Extensive details about these and all conference-related activities can be found on the 2004 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 16, 2004.** After this date, it will be necessary to register at the meeting site.

The **Westin Charlotte Hotel** is the TMS headquarters hotel. Special conference rates have been contracted with this hotel and others in the area surrounding the **Charlotte Convention Center.** To receive special rates, use the TMS 2004 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 2004. 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 2004 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 2004 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:

ADVANCED MATERIALS FOR ENERGY CONVERSION II
Dhanesh Chandra, Renato G. Bautista, and Louis Schlapbach, editors
ISBN 0-87339-574-3 • Approx. 560 pp., illus., index, softcover
Order No. 04-5743 • Weight 3 lbs
M $112 ✦ S $89 ✦ L $160

ADVANCES IN SUPERPLASTICITY AND SUPERPLASTIC FORMING
Eric M. Taleff, Paul E. Krajewski, and Peter A. Friedman, editors
ISBN 0-87339-564-6 • Approx. 436 pp., illus., index, softcover
Order No. 04-5646 • Weight 2 lbs
M $115 ✦ S $91 ✦ L $164

BULK METALLIC GLASSES
Peter K. Liaw and Raymond A. Buchanan, editors
ISBN 0-87339-573-5 • Approx. 256 pp., illus., index, softcover
Order No. 04-5735 • Weight 2 lbs
M $125 ✦ S $99 ✦ L $179

EPD CONGRESS 2004
Mark Schlesinger, editor
Includes the proceedings from the following symposia: Electrochemical Measurements and Processing of Materials, General Pyrometallurgy, Materials Processing Fundamentals, Solid and Aqueous Wastes, Sustainable Development session of Recent Advances in Non-Ferrous Metals Processing, and General Recycling session of Recycling.
ISBN 0-87339-565-4 • Approx. 1,020 pp., CD-ROM
Order No. 04-5654-CD • Weight 1 lb
M $71 ✦ S $56 ✦ L $101

LATERITE NICKEL SYMPOSIUM 2004
D.M. Lane and W.P. Imrie, editors
ISBN 0-87339-550-6 • Approx. 1,144 pp., illus., index, hardcover
Order No. 04-5506 • Weight 4 lbs
M $119 ✦ S $94 ✦ L $170

LIGHT METALS 2004
A.T. Tabereaux, editor
Includes the proceedings from the following symposia Alumina & Bauxite, Aluminum Can Recycling, Aluminum Reduction Technology, Carbon Technology, Cast House Technology, Reactive Metals session of Recent Advances in Non-Ferrous Metals Processing, Aluminum and Aluminum Dross Processing sessions of Recycling.
ISBN 0-87339-567-0 • Approx. 1,150 pp., illus., hardcover & CD-ROM
Order No. 04-5670-G • Weight 7 lbs
M $150 ✦ S $125 ✦ L $225

MAGNESIUM TECHNOLOGY 2004
Alan A. Luo, editor
ISBN 0-87339-568-9 • Approx. 436 pp., illus., hardcover & CD-ROM
Order No. 04-5689-G • Weight 3 lbs
M $101 ✦ S $80 ✦ L $144

SOLIDIFICATION OF ALUMINUM ALLOYS
Men G. Chu, Douglas A. Granger, and Qingyou Han, editors
ISBN 0-87339-569-7 • Approx. 440 pp., illus., softcover
Order No. 04-5697 • Weight 2 lbs
M $118 ✦ S $93 ✦ L $168

MULTIPHASE PHENOMENA AND CFD MODELING AND SIMULATION IN MATERIALS PROCESSES
L. Nastac and B. Li, editors
Includes the proceedings from the following symposia: Multiphase Phenomena in Materials Processing and CFD Modeling and Simulation of Engineering Processes.
ISBN 0-87339-570-0 • Approx. 760 pp., illus., softcover
Order No. 04-5700 • Weight 4 lbs
M $132 ✦ S $105 ✦ L $189

SOLIDIFICATION PROCESSES AND MICROSTRUCTURES: A SYMPOSIUM IN HONOR OF PROF. W. KURZ
M. Rappaz, C. Beckermann, and R. Trivedi, editors
ISBN 0-87339-572-7 • Approx. 432 pp., softcover
Order No. 04-5727 • Weight 2 lbs
M $112 ✦ S $88 ✦ L $159

THE FIFTH GLOBAL INNOVATIONS SYMPOSIUM ON MATERIALS PROCESSING AND MANUFACTURING: SURFACES AND INTERFACES IN NANOSTRUCTURED MATERIALS AND TRENDS IN LIGA, MINIATURIZATION, AND NANOSCALE MATERIALS
Sharmila M. Mukhopadhyay, John Smugeresky, Sudipta Seal, Narendra B. Dahotre, and Arvind Agarwal, editors
Includes the proceedings from the following symposia: Surfaces and Interfaces in Nanostructured Materials and the Fifth Global Innovations Symposium on Materials Processing and Manufacturing: Trends in LIGA, Miniaturization, and Nanoscale Materials
ISBN 0-87339-566-2 • Approx. 720 pp., illus., softcover
Order No. 04-5662 • Weight 4 lbs
M $118 ✦ S $93 ✦ L $168

ULTRAFINE GRAINED MATERIALS III
Yuntian Theodore Zhu, Terence G. Langdon, and Ruslan Z. Valiev, editors
ISBN 0-87339-571-9 • Approx. 824 pp., illus., index, softcover
Order No. 04-5719 • Weight 4 lbs
M $124 ✦ S $98 ✦ L $177

M /Member ✦ S / Student ✦ L / List

The following proceedings are planned for publication in TMS journals after the meeting:

In the *Journal of Electronic Materials*

Challenges in Advanced Thin Films: Microstructures, Interfaces, and Reactions

Lead-Free Solders and Processing Issues Relevant to Microelectronic Packaging

Phase Stability, Phase Transformation, and Reactive Phase Formation in Electronic Materials III

In *Metallurgical and Materials Transactions*

Beyond Nickel-Base Superalloys

Hume-Rothery Symposium: Structural and Diffusional Growth

Phase Transformations and Deformation in Magnesium Alloys

In *TMS Letters*

Processing and Properties of Powder-Based Materials

Other symposia eligible for *TMS Letters*:

Cost-Affordable Titanium

Dislocations

Educational Issues in Transport Phenomena in Materials Processing

General Abstracts

General Poster Session

Internal Stresses and Thermo-Mechanical Behavior in Multi-Component Materials Systems

Roytburd Symposium on Polydomain Stryctures

Symposium in Honor of Prof. Roger D. Doherty

The Didier de Fontaine Symposium on the Thermodynamics of Alloys

The Role of Grain Boundaries in Material Design

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

ADDITIONAL RESOURCES

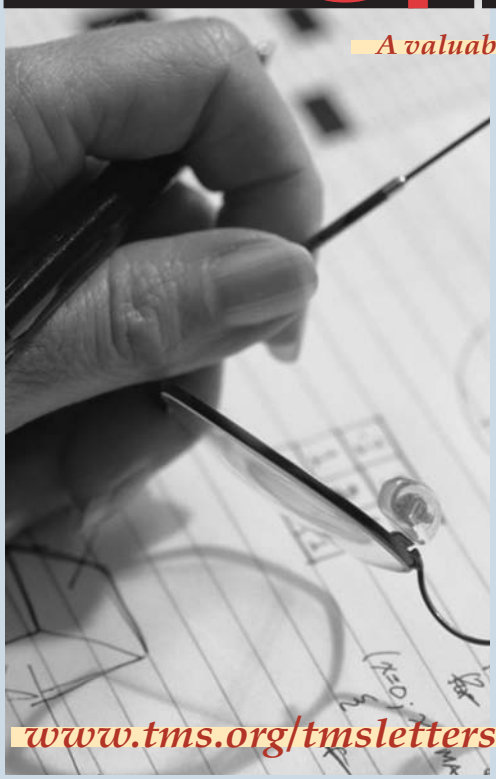
On-line answers to any of your 2003 TMS Annual Meeting & Exhibition questions can be found at

- **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-04/Exhibit2004/Annual04-exhibit-home.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: 1-800-759-4867 (in the U.S. and Canada) or
(724) 776-9000, ext. 243
Fax: (724) 776-3770

TMS LETTERS



A valuable new resource for members

A distinguished publication venue for authors

Timely, relevant, and rigorously reviewed, *TMS Letters* is a unique technical journal that presents cutting-edge research in succinct, informative technical updates.

The peer-reviewed journal will be available exclusively in on-line format through the TMS Document Center (*doc.tms.org*) and will be accessible free-of-charge to all TMS members as a benefit of membership. *TMS Letters* will be composed entirely of two-page technical updates, including text and graphics, of research presented at TMS meetings that are not published in any other book or journal.

The first issue of *TMS Letters* will consist exclusively of technical updates presented at the 2004 TMS Annual Meeting, to be held March 14–18, 2004. Presenters at the 2004 TMS Annual Meeting, whose work will not be published in any other book or journal, may submit their work for publication in the inaugural issue of *TMS Letters*.

To learn more about *TMS Letters* or
to submit a technical update, contact:
Dan Thoma
Editor, *TMS Letters*
c/o TMS
184 Thorn Hill Road, Warrendale, PA 15086
E-mail: tmsletters@tms.org
Web: *www.tms.org/tmsletters.html*

www.tms.org/tmsletters.html

Visit this web site often, as more details will be made available throughout the year, including author instructions for submitting papers to the journal and non-member subscription information.

TMS2004

March 14-18, 2004 • Charlotte, NC

ADVANCE REGISTRATION FORM

Advance Registration Deadline: February 16, 2004

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

AM04-TECH

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

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

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

1.

☐ Dr. ☐ Prof. ☐ Mr. ☐ Mrs. ☐ Ms. _____
Last Name First Name Middle Initial

Member of: ☐ TMS ☐ AIST ☐ SME ☐ SPE Member Number: _____

Informal First Name to Appear on Badge: _____ Date of Birth: _____

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 16, 2004		On-Site Fees after February 16, 2004	
☐ Member	$400	M	$500	ML
☐ Non-member Author*	$490	NMA	$590	NMAL
☐ Non-member *	$550	NM	$650	NML
☐ Student Member ##	$0	STU	$0	STUL
☐ Student Non-member ## *	$25	STUN	$25	STUNL
☐ TMS Senior Member	$250	RM	$250	RML
☐ Exhibit Booth Personnel	$0	E	$0	EL
☐ Exhibit Only	$35	EO	$35	EOL

Registration TOTAL $ _____

** Includes TMS membership for 2004*
Students must attach a copy of their school's student identification card.

3. Social Function Tickets:

		Fee	Quantity	Total
Monday 3/15/04	Didier de Fontaine Honorary Dinner	$60	_____	$_____ FD
	R.J. Arsenalt Honorary Dinner	$60	_____	$_____ JD
	Roger Doherty Honorary Dinner	$60	_____	$_____ DD
Tuesday 3/16/04	TMS-AIME Banquet	$60	_____	$_____ AD
	Tables of 8	$480	_____	$_____ AD8
	Table Sign to Read _____			
	Extraction & Processing Division Luncheon	$35	_____	$_____ EP
	Tables of 8	$280	_____	$_____ EP8
	Table Sign to Read _____			
Wednesday 3/17/04	Light Metals Division Luncheon	$35	_____	$_____ LM
	Tables of 8	$280	_____	$_____ LM8
	Table Sign to Read _____			
	A.L. Roytburd Honorary Dinner	$60	_____	$_____ RD

Social Function TOTAL $ _____

4. Tutorial Luncheon Tickets:

Monday 3/15/04 — Fee — Quantity — Total

The Young Leader Tutorial Lecture is free.
You may purchase the optional box lunch for $25 _____ $_____ EM

5. Publication Orders:

All orders that are not indicated for shipment on this form must be picked up at the meeting.

Order Number	Title	Shipping Weight	Quantity	Subtotal Weight	At-Meeting Price	List Price	Subtotal Price
04-5654-CD	EPD Congress 2004 (CD-ROM)	1	_____	_____	$71	$101	$_____
04-5506	Laterite Nickel 2004	4	_____	_____	$119	$170	$_____
04-5670-G	Light Metals 2004 (Book and CD-ROM Set)	7	_____	_____	$150	$225	$_____
04-5689-G	Magnesium Technology 2004 (Book and CD-ROM Set)	3	_____	_____	$101	$144	$_____
04-5743	Advanced Materials for Energy Conversion II	3	_____	_____	$112	$160	$_____
04-5662	Fifth Global Symposium on Materials Processing and Manufacturing: Surfaces and Interfaces in Nanostructured Materials and Trends in LIGA, Miniaturization, and Nanoscale Materials	4	_____	_____	$118	$168	$_____
04-5719	Ultrafine Grained Materials III	4	_____	_____	$124	$177	$_____
04-5727	Solidification Processes and Microstructures (A Symposium in Honor of Prof. W. Kurz)	2	_____	_____	$112	$159	$_____
04-5697	Solidification of Aluminum Alloys	2	_____	_____	$118	$168	$_____
04-5735	Bulk Metallic Glasses	2	_____	_____	$125	$179	$_____
04-5646	Advances in Superplasticity and Superplastic Forming	2	_____	_____	$115	$164	$_____
04-5700	Multiphase Phenomena and CFD Modeling and Simulation in Materials Processes	4	_____	_____	$132	$189	$_____

Subtotal $ _____

WEIGHT AND ZONE CHART

Weight	USA	Canada	Mexico	Western Europe	J. A. NZ	EE, C/S Am, Pac. Rim,	Middle East, Africa
1	$4.50	$4.00	$5.00	$4.50	$5.00	$5.50	$7.50
2	$5.00	$7.50	$9.50	$8.50	$9.50	$10.50	$14.50
3	$5.50	$11.00	$14.00	$12.50	$14.00	$15.50	$21.50
4	$6.00	$14.50	$18.50	$16.50	$18.50	$20.50	$28.50
5	$6.50	$18.00	$23.00	$20.50	$23.00	$25.50	$35.50
6	$7.00	$21.50	$27.50	$24.50	$27.50	$30.50	$42.50
7	$7.50	$25.00	$32.00	$28.50	$32.00	$35.50	$49.50
8	$8.00	$28.50	$36.50	$32.50	$36.50	$40.50	$56.50
9	$8.50	$32.00	$41.00	$36.50	$41.00	$45.50	$63.50
10	$9.00	$35.50	$45.50	$40.50	$45.50	$50.50	$70.50
11	$9.50	$39.00	$50.00	$44.50	$50.00	$55.50	$77.50
12	$10.00	$42.50	$54.50	$48.50	$54.50	$60.50	$84.50

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

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

One-time $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. ($10.00) + 4 lbs ($6.00) = 16 lbs. ($16.00)]*

Publications TOTAL $ _____

6. Continuing Education Short Courses:

Sunday, March 14, 2004

	Advance Fees until February 16, 2004 Member	Non-member	On-Site Fees after February 16, 2004 Member	Non-member
☐ 1. Metal Matrix Composites	$475	$560	$525	$610
☐ 2. Introduction to Nanomanufacturing and Nanotechnology	$475	$560	$525	$610
☐ 3. Technology Transfer Seminar	$475	$560	$525	$610
☐ 4. Smelter Grade Alumina from the Smelting Perspective	$475	$560	$525	$610
☐ 5. Computational Modeling for the Materials Professionals	$475	$560	$525	$610

Short Course TOTAL $ _____ $ _____

7. 2004 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 USA dollars drawn on a USA 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 16, 2004. A $50 processing fee will be charged for all cancellations. **No refunds will be processed after February 16, 2004.**

TMS2004

133rd Annual International Meeting & Exhibition
March 14-18, 2004 • Charlotte, North Carolina, 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
(in 212, 718, 516, 914, 631 or international call 212-532-1660)
(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 16, 2004

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. _____

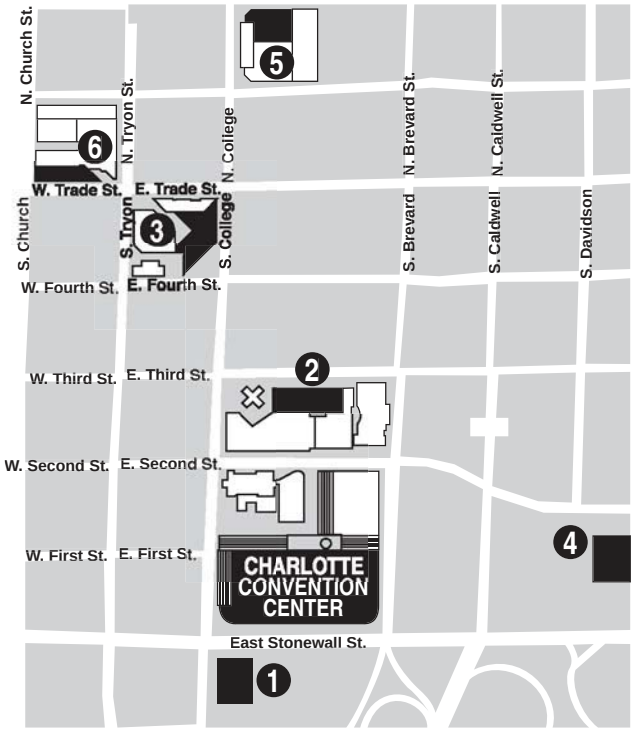
Type of Accomodations: (check one)
☐ Single 1 person/1bed ☐ Double 2 people/1bed ☐ Twin 2 people/2 beds
☐ Triple 3 people/2 beds ☐ Quad 4 people/2 beds

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

TMS has contracted a block of rooms at the headquarters hotel, Westin Charlotte Hotel, 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 133rd TMS Annual Meeting & Exhibition by making your reservation at one of the listed hotels prior to the advance housing deadline. Thank you.

HEADQUARTERS

1. **Westin Charlotte Hotel**
   $179/single • $194/double
2. **Hilton Charlotte Hotel**
   $154/single • $174/double
3. **Omni Hotel**
   $129/single • $129/double
4. **Adams Mark Hotel**
   $125/single • $125/double
5. **Holiday Inn Center City**
   $115/single • $115/double
6. **Marriott City Center Hotel**
   $138/Traditional S/D
   $138/Concierge Level S/D



Confirmations: Confirmations will be e-mailed, faxed or mailed to you from Travel Planners, Inc. once your reservation has been secured with a deposit or credit card. 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. 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 _____

Monday-March 15		Tuesday-March 16		Wednesday-March 17		Thursday-March 18	
AM	PM	AM	PM	AM	PM	AM	
					Materials Analysis: Understanding the Columbia Disaster		Ballroom B
Dislocations: Modeling and Simulation Fundamentals	Dislocations: Simulation and Observation of Fundamental Mechanisms	Dislocations: Dislocation Structures and Patterning	Dislocations: Novel Experimental Methods	Dislocations: Plasticity, Voids, and Fracture	Dislocations: Dislocations in Complex Materials		201A
	Advances in Superplasticity and Superplastic Forming: Dvlp. of Advanced Superplastic Forming Processes	Advances in Superplasticity and Superplastic Forming: Advances in Superplastic Al-Mg Materials	Advances in Superplasticity and Superplastic Forming: Advances in Superplastic Forming of Light Alloys	Advances in Superplasticity and Superplastic Forming: Advd. Superplastic Matls. & the Sci. of Superplasticity	Advances in Superplasticity and Superplastic Forming: Modeling of Superplastic Forming Processes and Materials	General Abstracts: Session IX	201B
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GRID

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5th Global Innovations Symposium: Trends in LIGA, Miniaturization, and Nano-Scale Materials, Devices and Technologies: Properties, Processes, and Modeling

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Powder Materials Committee, MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS), MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD/EPD-Process Modeling Analysis & Control Committee, MPMD-Surface Engineering Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee

Program Organizers: John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA; Steven H. Goods, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Sean J. Hearne, Sandia National Laboratories, Albuquerque, NM 87185-1415 USA; Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA

Wednesday AM Room: 202B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Andy Minor, Lawrence Berkeley National Laboratory, Matls. Sci. Div., Berkeley, CA 94720 USA; Tom Buchheit, Sandia National Laboratories, Albuquerque, NM 87185 USA

8:30 AM
Integrating Scientific Disciplines for Future Nanotechnologies: *John Charles Barbour*[1]; [1]Sandia National Laboratories, Nanostruct. & Semiconductor Physics, MS 1415, Org 1112, Albuquerque, NM 87185-1415 USA

Future technologies will rely on a complex integration of materials and functionalities that bridge several length scales to connect nanometer-scale architectures to the real world of man. The Center for Integrated Nanotechnologies (CINT), which is operated jointly by Sandia National Laboratories and Alamos National Laboratory, has a unique technical vision focused on integrating scientific disciplines and expertise across multiple length scales, and can therefore address the nanoscience challenges of coupling the nano- and micro-length scales. The core disciplines within CINT include: Nanomechanics, Nano-bio-micro Interface, Nanoelectronics/photonics, Complex Functional Materials, and Simulation and Modeling. This talk will present examples from these disciplines where the performance of existing microsystems has been improved by adding nanoscience, and where work is proceeding to understand fundamental mechanisms which govern properties of future nanosystems. This work is supported by the DOE Office of Basic Energy Sciences. Sandia is multiprogram laboratory operated by Sandia Corporation under contract DE-AC04-94AL85000.

9:00 AM
Mechanical Characterization of Nanoscale Gold Beam Structures Using AFM and Nanoindentation Techniques: *Xiaodong Li*[1]; Patrick Nardi[1]; Chang-Wook Baek[2]; Jong-Man Kim[2]; Yong-Kweon Kim[2]; [1]University of South Carolina, Dept. of Mech. Engrg., 300 Main St., Columbia, SC 29208 USA; [2]Seoul National University, Sch. of Elect. Engrg. & Compu. Sci., San 56-1, Shillim-Dong, Kwanak-ku, Seoul 151-742 S. Korea

In the design of micro/nanoelectromechanical systems (MEMS/NEMS) devices, mechanical properties of the MEMS/NEMS building structures are essential because most material properties are known to be size-dependent. Mechanical characterization of nanoscale gold beams with widths ranging from 200 to 500 nm has been carried out. Hardness and elastic modulus of the unreleased beams were measured using a nanoindenter. Bending tests were performed on the released cantilever and fix-fix beams using a nanoindnter in conjunction with an atomic force microscope (AFM). Residual stress in the beams was measured. Effect of residual stress on the mechanical properties is discussed. Deformation behavior of the beams was studied using a scanning electron microscope and finite element analysis.

9:20 AM
Mechanical Behavior and Oxidation of Pt-Ir Alloy Thin Films: *Richard R. Chromik*[1]; Thirumalesh Bannuru[1]; Walter L. Brown[1]; Richard P. Vinci[1]; [1]Lehigh University, Dept. of Matls. Sci. & Engrg., 5 E. Packer Ave., Bethlehem, PA 18015 USA

MEMS applications often require novel electrode materials with both robust electrical and mechanical properties. Stress relaxation and morphological stability present problems, especially at high temperatures. Alloying is sometimes used to address these issues, but its effectiveness in thin films may be reduced from that of bulk alloying. The mechanical behavior of oxidized and unoxidized Pt-Ir thin films have been studied, where oxide dispersion strengthening and solid solution strengthening are expected respectively. Alloy films were fabricated by sputter co-deposition with compositions ranging from 0 to 25at% Ir, as measured by x-ray photoelectron spectroscopy (XPS). Mechanical properties of the films were measured by nanoindentation and wafer curvature. For unoxidized specimens, solid solution strengthening was observed, where films with higher Ir content exhibited higher hardness. Oxidation studies were carried out to form Ir oxide particles and examine the dispersion strengthening mechanisms as well.

9:40 AM
Deformation Mechanisms in Nanostructured Aluminum Alloys Processed by Cryomilling Techniques: Bing Q. Han[1]; *Enrique J. Lavernia*[1]; [1]University of California, Chem. Engrg. & Matls. Sci., Davis, CA 95616 USA

Cryomilling process is proven to be an effective approach to process bulk nanostructured aluminum alloys. In the present study, microstructural characteristics and deformation behavior of bulk nanostructured aluminum alloys processed via consolidation of cryomilled powders were reviewed. The microstructure of supersaturated solid solution with equiaxed grains from 50 nm to 300 nm was observed in several as-extruded cryomilled Al alloys. Tensile behavior was characterized by high strength and low strain hardening. The high strength at room temperature was primarily attributed to three types of strengthening: grain size effect, solid solution hardening and Orowan strengthening. Cryomilled Al-Mg alloys with bimodal microstructure show both high strength and improved ductility.

10:00 AM Break

10:20 AM
Tension/Compression Asymmetry, Anisotropy and Size Effects in the Plastic Deformation of Ti-6242 Single Colonies: *Dave M. Norfleet*[1]; Michael J. Mills[1]; Michael D. Uchic[2]; Mike F. Savage[3]; Joe Tatalovich[1]; [1]Ohio State University, Matls. Sci. & Engrg., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Air Force Research Laboritory, Matls. & Mfg. Direct., Wright Patterson AFB, Dayton, OH 45433 USA; [3]Pratt & Whitney, E. Hartford, CT 06108 USA

The constant strain rate deformation behavior of individual ??? colonies of the titanium aeroengine alloy Ti-6Al-2Sn-4Zr-2Mo-0.1Si (composition in wt.%) has been studied through an ultra small-scale compression technique. Using a FEI Dual Beam Focused Ion Beam, cylindrical compression samples were micromachined into the grip-ends of sub-millimeter-scale samples that previously had been tested in tension. Thus, one goal of the work is determine if a pronounced tension/compression asymmetry exists for these single colony crystals, while performing the test on the same samples and using the same deformation axis. Six single colony crystal orientations are being explored, associated with single slip in the HCP alpha phase along the three distinct a-type slip systems on both basal and prism planes. The compression samples, having diameters ranging from 10 to 35 microns, were mechanically tested using an MTS Nano Indenter XP fitted with a flat tip to apply uniaxial compression at a constant strain rate. The effect of sample size on the flow properties will be discussed. The resulting data will be correlated with tensile results, and TEM studies will be presented.

10:40 AM Cancelled
Average Stresses in Simulated Thin Films

11:00 AM
Molecular Dynamics Simulation of Dislocation Formation During Vapor Deposition of Multilayers: *Xiaowang Zhou*[1]; Haydn N.G. Wadley[1]; [1]University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA 22903 USA

Molecular dynamics has been used to study dislocation formation during NiFe/Au/NiFe/Au and CoFe/NiFe/CoFe/Cu multilayer deposition. A direct nucleation of misfit dislocations on (111) interfaces was observed in the NiFe/Au/NiFe/Au system. Both dislocation configura-

tion and density were in good agreement with HRTEM experiments. A misfit energy increasing dislocation structure was found in the simulated CoFe/NiFe/CoFe/Cu multilayers. It formed by atomic assembly mechanisms. During deposition on the (111) surface of an f.c.c. lattice, adatoms may either occupy f.c.c. sites or h.c.p. sites. This results in partial dislocations at the f.c.c and h.c.p. domain boundaries. These boundaries tend to have missing atoms, and therefore, a later deposited layer tends to have less planes compared to a previously deposited layer. This effect is negligible for large lattice mismatch systems such as NiFe/Au, but is relatively significant for small lattice mismatch systems such as CoFe/NiFe. Growth conditions affecting dislocations are discussed.

11:20 AM
Molecular Dynamics Simulations of Single Asperity Contacts: Monotonic and Cyclic Loading: *Pil-Ryung Cha*[1]; David J. Srolovitz[1]; [1]Princeton University, Dept. of Mech. & Aeros. Engrg., Princeton Matls. Inst., 70 Prospect Ave., Princeton, NJ 08544 USA

Many state-of-the-art micro-electronic-photonic and MEMS devices are based upon micro/nano-scale contacts. Descriptions of such contacts must account for elastic/plastic deformation, adhesion and contact shape evolution. Plastic deformation is fundamentally different on the nanoscale. We present a molecular dynamics study of single asperity contacts in EAM Au as a function of contact loading and unloading at different frequencies. We monitor the full force-displacement curve, the evolution of the atomic structure/asperity morphology, dislocation nucleation and motion. Plastic deformation causes the disappearance of individual atomic layers with concomitant abrupt jumps in the applied force-displacement curve. Dislocations nucleated upon loading tended to be partials. Stacking fault pyramids are created and then annihilated causing abrupt jumps in force-displacement curves. Upon unloading, dislocations run out of the asperities, leaving dislocation-free structures. The tensile stresses generated during pull-of f produce twin-like structures. The nature of the deformation produced in cyclic loading was extremely sensitive to the deformation rate.

Advanced Materials for Energy Conversion II: Metal Hydrides III

Sponsored by: Light Metals Division, LMD-Reactive Metals Committee
Program Organizers: Dhanesh Chandra, University of Nevada, Metallurgical & Materials Engineering, Reno, NV 89557 USA; Renato G. Bautista, University of Nevada, Metallurgical and Materials Engineering, Reno, NV 89557-0136 USA; Louis Schlapbach, EMPA Swiss Federal, Laboratory for Materials Testing and Research, Duebendorf CH-8600 Switzerland

Wednesday AM Room: 203A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Ricardo B. Schwarz, Los Alamos National Laboratory, MS G755, Los Alamos, NM 87545 USA; Etsuo Akiba, National Institute of Advanced Studies, Energy Elect. Inst., Tsukuba, Ibaraki 305-8565 Japan; R. Tom Walters, Westinghouse Savannah River Co. LLC, Strategic Matls. Tech. Dept., Aiken, SC 29808 USA

8:30 AM Plenary
The Role of Defects on the Hydrogen Storage Capacity of Metals: *Reiner Kirchheim*[1]; Astrid Pundt[1]; Christian Kluthe[1]; Mohamed Suleiman[1]; [1]Universitaet Goettingen, Inst. fuer Materialphysik, Tammannstrasse 1, Goettingen D-37077 Germany

Hydrogen storage alloys used today are multicomponent and structurally disordered containing large fractions of dislocations and grain boundaries. The current status of modelling pressure composition isotherms and H-diffusivity for these hydrogen-metal systems will be reviewed. Recent experimental results on the interaction between hydrogen on the one hand and subsurface sites, dislocations, grain boundaries and phase boundaries on the other hand are presented for model systems. These model systems are small Pd-clusters, nanocrystalline, deformed, and internally oxidized Pd and metallic multylayers. Corresponding experimental results are obtained by small angle neutron scattering, Synchrotron X-ray diffraction and by tomographic atom probe analysis.

9:00 AM Keynote
Hysteresis in the Reversible Storage of Hydrogen in Metals: *R. B. Schwarz*[1]; A. Khachaturyan[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Rutgers University, Ceram. & Matls. Engrg., 607 Taylor Rd., Piscataway, NJ 08854 USA

In many metal-hydrogen systems, the plateau pressure during hydrogenation is higher than that during hydrogen removal, in what is known as hysteresis. Since the plateau pressure reflects equality between hydrogen chemical potentials in the transforming phases, and classical thermodynamics cannot explain lack of ergodicity, early models attributed the hysteresis to the irreversible generation of lattice defects. The recent model of Schwarz and Khachaturyan [PRL 74(1995)2523] attributes the hysteresis to the effect of the coherency strains between the solid-solution and hydride phases. We summarize this model and present evidence supporting it. In particular, we show that hysteresis is also observed between the pressure necessary to form Pd-hydride precipitates in a Pd single crystal and the pressure required to form Pd-crystal precipitates in a Pd-hydride single crystal. Therefore, the generation of dislocations, which cannot be avoided during the transformation due to the large mismatch strain, is not the cause for the hysteresis.

9:25 AM Invited
Diffraction Studies of Alanates: *Hendrik W. Brinks*[1]; Bjørn C. Hauback[1]; Didier Blanchard[1]; Craig M. Jensen[2]; [1]Institute for Energy Technology, Physics Dept., PO Box 40, Kjeller NO-2027 Norway; [2]University of Hawaii, Dept. Chmst., 2545 The Mall, Honolulu, HI 96822 USA

Alanates, metal hydrides based on the AlH_4^- unit, is one of the most promising groups of metal hydrides for reversible hydrogen storage at moderate temperatures. Their storage capacity is very large, e.g. $NaAlH_4$ and $LiAlH_4$ can release 5.6 and 7.9 wt%, respectively, below 200ºC. The pioneering work of Bogdanovic et al. in 1997 revealed that Ti additives increased the desorption kinetics of NaAlH4 and made rehydrogenation possible. In order to get a better understanding of the reactions, detailed studies of the structure is essential. In particular, it is important to understand the nature of the additive in order to improve the kinetics. Our structural studies of $NaAlD_4$ with different additives will be presented. Crystal structures of $LiAlD_4$, $NaAlD_4$, Li_3AlD_6 and $KAlD_4$ will be presented, as well as in-situ diffraction results of the decomposition. $LiAlD_4$ has been shown to decompose completely to LiD, Al and D_2 at 127ºC, releasing 7.9 wt% H_2. Addition of VCl_3 by ball milling increases the reaction rate.

9:50 AM Invited
Advances in Hydrogen Storage: *Arnulf J. Maeland*[1]; Bjorn C. Hauback[1]; [1]Institute for Energy Technology, Dept. of Physics, PO Box 40, Kjeller 2027 Norway

The advancement of hydrogen and fuel cell technologies in transportation as well as stationary and portable applications depend very much on efficient and safe storage of hydrogen. Storage technologies, gaseous and liquid hydrogen storage and chemical storage, will be reviewed and discussed. Particular emphasis will be placed on hydrogen storage in the form of metal hydrides.

10:15 AM Break

10:30 AM Invited
The Use of Hydrogen Driven Metallurgical Reactions (HDMR) to Produce Reactive, Nano-Scale and Nano-Composite Materials: *J. J. Reilly*[1]; J. R. Johnson[1]; J. Wegryzn[1]; [1]Brookhaven National Laboratory, Dept. of Energy Sci. & Tech., Upton, NY 11733 USA

The direct reaction of hydrogen with a metal to form a metal hydride phase results in a large volume change which can result in pulverization of the material. This hydrogen decrepitation (HD) process was exploited some years ago to produce rare earth alloy powders. More recently the HDDR (hydriding, dehydriding, disproportionation, recombination) process was introduced which exploits thermodynamic instability of many alloys at high temperatures in the presence of H2 gas and was used to produce improved magnetic alloys. We have extended this process to produce nanocomposite materials by introducing a reducible metal oxide which greatly increases the number of possible reactions. Such solid state reactions fall into a wide class which may be designated as hydrogen driven metallurgical reactions (HDMR). We will discuss the versatility of this process to produce many types of nanocomposite (or nano-scale) materials, particularly those of interest for use as Li anodes in batteries and for improved hydrogen storage compounds.

10:55 AM
Palladium Composite Membranes for Hydrogen Separation: *Stephen N. Paglieri*[1]; Stephen A. Birdsell[1]; Ronny C. Snow[1]; Vincent B. Hesch[1]; Frank M. Smith[1]; [1]Los Alamos National Laboratory, PO Box 1663, MS-C348, Los Alamos, NM 87545 USA

A palladium membrane reactor can be used to efficiently generate hydrogen from hydrocarbons. Two types of palladium composite membranes were investigated for hydrogen separation. A palladium alloy membrane was prepared by electroless plating a thin (~20 micron) layer of palladium and then copper onto a commercially available porous (nominal 0.2 micron pores) alpha-alumina substrate. The resulting multilayer metal film was annealed at 360ºC for several days to promote metallic interdiffusion and alloy formation. During the heat treatment, a maximum hydrogen flux of 0.15 mol (STP)/m^2·s was observed at 360ºC and a pressure drop across the membrane of 100 psi. The hydrogen/argon ideal separation factor was 68 at these conditions, however, the separation factor decreased upon thermal cycling. The other type of membrane fabricated was a palladium coated vanadium-copper alloy foil. New methods are being developed for sealing the thin (40 micron) foil into modules for testing.

11:15 AM
Hydrogen Storage Properties of Li Related and C Related Materials: *Takayuki Ichikawa*[1]; Hironobu Fujii[1]; [1]Hiroshima University, Natural Sci. Ctr. for Basic R&D, 1-7-1 Kagamiyama, Higashi-Hiroshima, Hiroshima 739-8521 Japan

We are investigating hydrogenating/dehydrogenating properties about the 1:1 mixture of lithium amide and lithium hydride containing a small amount of TiCl3 (1mol %) as a candidate of hydrogen storage materials. The product desorbs a large amount of hydrogen (~5.5 wt.%) in the temperature range from 150 to 250°C under the heating rate condition of 5°C/min and also shows an excellent cycle retention with an effective hydrogen capacity of more than 5 wt.% until at least 3 cycles. In addition, we are also studying the hydrogen desorption properties about hydrogenating graphite made by ball milling under hydrogen atmosphere. The results indicate that a small amount of iron contamination during milling plays a quite important role as a catalyst for hydrogen absorption/desorption properties. Two-peak structure for hydrogen desorption in the TDS profile is due to existence of high dispersing iron on graphite.

Advanced Materials for Energy Conversion II: Thermodynamics, Superconductors & Batteries

Sponsored by: Light Metals Division, LMD-Reactive Metals Committee
Program Organizers: Dhanesh Chandra, University of Nevada, Metallurgical & Materials Engineering, Reno, NV 89557 USA; Renato G. Bautista, University of Nevada, Metallurgical and Materials Engineering, Reno, NV 89557-0136 USA; Louis Schlapbach, EMPA Swiss Federal, Laboratory for Materials Testing and Research, Duebendorf CH-8600 Switzerland

Wednesday AM Room: 204
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Ramana G. Reddy, University of Alabama, Dept. of Metallurgl. & Matls. Engrg., Tuscaloosa, AL 35487-0202 USA; Brent Fultz, California Institute of Technology, Matls. Sci., Pasadena, CA 91125 USA; Olivia A. Graeve, University of Nevada, Metallurgl. & Matls. Engrg., Reno, NV 89557-0136 USA

8:30 AM Invited
Origin of Entropy of Intercalation of Li into Li_yCoO_2: *Brent Fultz*[1]; Yvan Reynier[2]; Tabitha Swan-Wood[1]; Jason Graetz[1]; Peter Rez[3]; Rachid Yazami[2]; [1]California Institute of Technology, Matls. Sci., Mail 138-78, Pasadena, CA 91125 USA; [2]California Institute of Technology/CNRS-LEPMI, Matls. Sci., Mail 138-78, Pasadena, CA 91125 USA; [3]Arizona State University, Dept. of Physics & Astron., Tempe, AZ 85257-1504 USA

We studied the thermodynamics of lithium insertion into LiCoO_2, the cathode material of commercial rechargeable Li batteries. The entropy of lithium intercalation, dS, and enthalpy of intercalation, dH, were determined from measurements of the open circuit voltage of electrochemical half-cells at different temperatures. The present work addresses the entropy. A large change of 2 k_B/atom was observed for the difference in entropy of intercalation of lithium into Li_0.6CoO_2 and Li_1CoO_2. The three sources of entropy of lithium concentration in Li_yCoO_2 are: 1) configurational entropy, 2) electronic entropy, and 3) vibrational entropy. The first two contributions were obtained by calculation, using combinatoric estimates for 1, and electronic structure calculations with the VASP package for 2. Configurational entropy, and surprisingly, electronic entropy, account for most of the entropy difference measured by electrochemical methods. The third contribution, vibrational entropy, was measured by inelastic neutron scattering, and found to be small.

8:55 AM Keynote
Recent Developments in Materials and Design Concepts for Bipolar Plates in Fuel Cells: *Ramana G. Reddy*[1]; [1]University of Alabama, Dept. of Metallurgl. & Matls. Engrg., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

The fuel cell technology is predestined to provide a major breakthrough in the way we power our very existence on the planet, powering virtually everything from cars to mobiles. The widespread commercialization of the technology has still not been made possible due to high costs associated with the fuel cell components. One such component in the fuel cell stack is the bipolar/end plate. This presentation reviews some of the recent developments in the materials, design, and concepts for bipolar/end plates in the polymer electrolyte membrane fuel cell stack from the author's experience at The University of Alabama. Experimental results for use of Fe-, Cu-based alloys for bipolar/end plate as an alternative to the expensive conventionally used graphite material are presented. The developments of the models for optimizing the design parameters in the gas flow-field of these plates are discussed. Based on these simulations results, some of the new concepts for these plates were urbanized. These include: use of met al foam in the gas flow-field and corrugated thin sheet bipolar/end plate. Experimental results with these new concepts are presented and will be compared with the model predicted results. Applications of these new concepts in the development of commercial fuel cell stacks are discussed.

9:20 AM Keynote
Vibrational Energy Scavenging Via Thin Flim Piezoelectric Ceramics: *Elizabeth Kathryn Reilly*[1]; Eric Carleton[1]; Paul Kenneth Wright[1]; [1]University of California, Mechl. Engrg., 2111 Etchverry Hall, Berkeley, CA 94720 USA

This work focuses on constructing a vibrational energy scavenging device with a specific application to MEMS wireless sensor networks. The device utilizes vibrations produced by HVAC ducts, traffic in a room, and even wind hitting a window. The advantages of using thin film (~1 micron) PZT (Pb1.15(Zr0.47, Ti0.53)O3) over a larger scale bimorph will be addressed. The thin films are grown using pulsed laser deposition (PLD) to deposit the film epitaxially on MgO. The PZT is then removed from the MgO using excimer laser liftoff and attached to a metallic shim, thus creating a usable bimorph. Discussion of the constituent equations will reflect that of system with an external force applied perpendicular to the beam at its tip. Characterization and material analysis will illustrate the effectiveness of this technique in creating an energy scavenging device.

9:45 AM
In-Situ Production of Nano-Structured Ceramics by Spray Solution Technique: *Konstantin Krasimirov Konstantinov*[1]; Zheng Wei Zhao[1]; Ling Yuan[1]; Hua Kun Liu[1]; Shi Xue Dou[1]; [1]University of Wollongong, ISEM, Northfields Ave., Wollongong, NSW 2522 Australia

Various nano-structured ceramic materials e.g. LiCo1-xNixO2, CoO, Co3O4 and SnO2 have been prepared in-situ by a spray pyrolysis method. The effect of the temperature and sintering time on nanocrystallinity, phase composition, and different physical and electrochemical parameters have been studied in detail. Different methods including X-ray diffraction, gas sorption analysis (for estimation of BET surface area), ICP-OES analysis, TEM and SEM techniques, combined with EDX analysis and standard battery testing methods have been used to characterized the powders obtained. We have demonstrated that the method used is very flexible and universal, and it permits good control of the crystal size and phase product, allowing in-situ production of simple or complex ceramics possessing specific surface areas that are generally larger than for the corresponding materials obtained via conventional technology. The obtained materials have promising potential applications not only as anode or cathode battery materials, but also as catalysts or capacitors.

10:05 AM Break

10:20 AM Invited
Secondary Lithium Ion Polymer Microbatteries: *Daniel A. Steingart*[1]; James W. Evans[1]; Paul K. Wright[1]; [1]University of California, Dept. of Matls. Sci. & Engrg., 2117 Etcheverry Hall, Berkeley, CA 94720 USA

The next generation of wireless sensor networks requires the energy production and storage mechanism to be integrated on chip. We are designing a micro-scale lithium secondary battery to be deposited

on the backside of a millimeter scale wireless sensor. The battery will continuously be charged by energy scavenging devices such as MEMS piezo-benders or heat pumps. The battery novelty comes from designing the cell around the duty cycle for a given application. The material novelty in the system lies in adaptation of Dunn's V_2O_5 aerogel cathode and Kostecki's pyrolyzed carbon anode for micro scale implementation. We will fabricate the entire cell on campus: the cathode from sol gel processing, the anode from standard micro fabrication photolithography, and combining them in a SiN2 or similar enclosure. Theoretical performance from proposed geometries will be discussed, as well as characterization and optimization of the half-cell reactions.

10:45 AM Invited
Processing and Properties of Nickel Foams for Battery Electrodes: *David S. Wilkinson*[1]; Vladimir Paserin[2]; [1]McMaster University, Dept. of Matl. Sci. & Engrg., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [2]INCO Technical Services, Sheridan Park, Mississauga, ON L5K 1Z9 Canada

Ni-metal hydride foams generally utilize nickel foams as the electrode phase. Amongst several methods for the manufacture of these chemical vapour deposition onto a polymer foam substrate is one of the most attractive. In this paper we will present several issues related to the processing of these foams and their properties in the as-deposited condition. We will show what factors control the high strength and consequent brittleness of the as-deposited foams. We will also present data on the optimum sintering conditions for these materials.

11:10 AM
Surface Modification of Spinel Li1+xMn2O4 Cathode Material in Li-Ion Battery by Li2O-2B2O3 Glass: *Hong Wei Chan*[1]; Shyang Roeng Sheen[2]; Jenq Gong Duh[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Engrg., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan; [2]Coremax Taiwan Corporation, R&D, 11 Wen Hwa Rd., Hsinchu Hsien 303 Taiwan

Lithium borate glass, Li2O-2B2O3, coated on the surface of the lithium manganese oxide cathode material in Li-ion batteries have been synthesized to achieve electrochemical cyclability and structural stability in this study. The heat treatment temperature was evaluated according to TG/DTA analysis. The Li1+xMn2O4 powder derived from co-precipitation method was calcined under various weight percent of Li2O-B2O3 glass to form fine powder of single spinel phase with different particle size, size distribution and morphology. The pure spinel Li1+xMn2O4 coated with LBO glass were successfully obtained by well-mixed solid-state method. The structure was confirmed by XRD along with the composition measured by EPMA and ICP-AES. The lattice parameter decreased with the content of the LBO wt's. From FESEM image and Laser Scattering measurements, the average particle size was in the range of 2-8?Ým. The electrochemical behavior of LiMn2O4 powder was examined by using two-electrode test cells consisted of a cathode, metal lic lithium anode, and an electrolyte of 1M LiPF6 in a 1:1 (volume ratio) mixture of EC/DMC. Cyclic charge/discharge testing of the coin cells, fabricated by both Li1+xMn2O4 and LBO-coated Li1+xMn2O4 cathode material provided high discharge capacity. Furthermore, the LBO-coated Li1+xMn2O4 powder showed better cyclability than un-coated Li1+xMn2O4 after 16 cycles test. The introduction of LBO-coated Li1+xMn2O4 revealed high capacity and apparently decreased the decay rate after cyclic test.

11:30 AM
Synthesis and Electrochemical Characterization of Cobalt - Manganese Oxide: *Nagireddy Ravinder Reddy*[1]; Ramana G. Reddy[1]; [1]University of Alabama, Dept. of Metallurgl. & Matls. Engrg., A-129 Bevill Bldg., 126 Seventh Ave., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

Cobalt - MnO2 was prepared using sol-gel method in ambigel form. Prepared material was studied as electrode material for electrochemical capacitors (EC's). Co - MnO2 was characterized using various techniques like XRD, SEM, TGA and BET. Co-MnO2 was electrochemically characterized using cyclic voltammetric technique. Cyclic Voltametry (CV) experiments were carried out using three electrode system. Saturated calomel electrode, platinum mesh and Co-MnO2 rolled in between titanium meshes were used as reference electrode, counter electrode and working electrode respectively. Non-capacitative behavior was observed in the first cycle and progressive charge enhancement was observed up to 100 cycles. Co-MnO2 electrode showed maximum capacitance of 105 F/g at a scan rate of 5 mV/s in 1M NaCl electrolyte. Capacitance fading was not observed up to 800 cycles. The results were compared with the capacitative behaviour of pure MnO2.

Advances in Superplasticity and Superplastic Forming: Advanced Superplastic Materials and the Science of Superplasticity

Sponsored by: Materials Processing and Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Structural Materials Committee
Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; P. A. Friedman, Ford Motor Company, Dearborn, MI 48124 USA; Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; P. E. Krajewski, General Motors R&D Center; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; J. G. Schroth, General Motors, R&D Center, Materials and Processes Laboratory, Warren, MI 48090-9055 USA

Wednesday AM Room: 201B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Rajiv S. Mishra, University of Missouri, Metallurgl. Engrg., Rolla, MO 65409-0340 USA; Amit K. Ghosh, University of Michigan, Dept. of Matls. Sci. & Engrg., Ann Arbor, MI 48109-2136 USA

8:30 AM
Cooperative Processes in Large Plastic and Superplastic Deformation: *Michael Zelin*[1]; Amiya Mukherjee[2]; [1]The Goodyear Tire & Rubber Company, Akron, OH 44309 USA; [2]University of California, Chem. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

Despite drastic differences in occurrence and mechanisms of normal plastic deformation (ND) and superplastic deformation (SPD), there is close similarity between the two with regard to mechanical aspects of deformed system-reaction to the applied force, non-uniformity of plastic flow, and its dislocation nature. In both ND and SPD, deformation is initiated at surfaces of maximum shear stress causing a long range correlation in grains deformation, i.e. its cooperative manner. This paper focuses on cooperative character of ND and SPD revealed at different microstructural levels. Results of targeted observations of deformation occurring in a drawn eutectoid steel wire and superplastic lead-tin eutectic are presented and discussed invoking different approaches. Observed macroscopic pattern of localized deformation surfaces can be explained from the point of view of solid mechanics. Sequential progress of deformation at localized deformation surfaces can be modeled in terms of cellular dislocations. Microscopic aspects of progress of ND and SPD can be rationalized in terms of lattice and grain boundary dislocations, respectively. Work supported in part by NSF/DMR grant 9903321/0240144.

8:55 AM
Influence of Grain Boundary Structure and Composition on Superplastic Deformation: *John S. Vetrano*[1]; C. H. Henager[1]; R. J. Kurtz[1]; R. G. Hoagland[1]; V. Guertsman[2]; [1]Pacific Northwest National Laboratory, MSIN P8-16, PO Box 999, Richland, WA 99352 USA; [2]CANMET/MTL, 568 Booth St., Ottawa, ON K1A 0G1 Canada

Grain boundary sliding (GBS) plays a large role in superplastic deformation and dictates not only the deformation characteristics but also the final failure. As part of a program to better elucidate the nature of GBS we have studied the interplay between the sliding of grains and their structure and composition in model Al-Mg alloys. Tools such as high-resolution analytical transmission electron microscopy and molecular dynamic computer simulations were utilized to investigate the influence of Mg and Sn on grain boundary structure, and examining how those different structures affect the ability of the boundary to slide. This paper will examine these effects from the atomistic level (grain boundary dislocations, microsegregation) to the micro- and macro-scale (precipitates and cavitation). Work supported by the Materials Science Division, Office of Basic Energy Sciences, U.S. Department of Energy under contract DE-AC06-76RLO-1830.

9:20 AM
Superplasticity in Zr-Based Bulk Metallic Glasses: A Critical Overview: *Martin Heilmaier*[1]; [1]Otto-von-Guericke-University Magdeburg, Inst. of Matls. Engrg. & Testing, PO Box 4120, Magdeburg D-39016 Germany

With the recent advent of multi-component bulk glassy alloys exhibiting wide supercooled liquid region, amorphous metals have ma-

tured from a lab curiosity to potential structural materials with unique mechanical and physical properties. However, applications are often limited by the uncertainty about the appropriate processing window. The compressive deformation behavior of Zr-based BMG matrices with and without second phase precipitates in a wide temperature and strain rate range within the supercooled liquid region is reviewed[1] and compared with tensile data on bulk samples and thin ribbons[2,3]. Truly Newtonian viscous flow with m = 1 and homogeneous deformation at constant viscosity is observed for strain rates as high as 10^{-2} s^{-1}. Application of Spaepens free volume theory clearly indicates that superplastic deformation in BMGs is controlled by atomic diffusion, or more specific by the competing effects of free volume generation and annihilation. [1]A. Reger-Leonhard et al., Scripta Mater. 43 (2000), 459. [2]J.P. Chu et al., Scripta Mater. 49 (2003), 435. [3]Y. Kawamura et al., Scripta Mater. 37 (1997), 431.

9:45 AM
Transformation Superplasticity of Titanium by Reversible Hydrogen Cycling: Heeman Choe[1]; *David C. Dunand*[1]; [1]Northwestern University, Dept. of Matls. Sci. Engrg., Evanston, IL 60208 USA

Commercially-pure titanium was reversibly alloyed and dealloyed with hydrogen at 860°C, thus repeatedly triggering the transformation between hydrogen-free α-Ti and hydrogen-alloyed β-Ti. Under an externally applied tensile stress, the internal mismatch stresses produced by the α-β transformation are biased, resulting in a strain increment accumulated after each chemical cycle in the direction of the applied stress. These strain increments are linearly proportional to the applied stress at small stress levels ($\sigma < \cong 2$ MPa), as previously reported for transformation superplasticity achieved by thermal cycling of hydrogen-free α-Ti. The present study investigates systematically the effect hydrogen partial pressure, cycle time, and external stress upon the value of the superplastic strain increments, as well as the concurrent contribution of creep as a deformation mechanism.

10:10 AM Break

10:30 AM
Research and Development Towards Grain-Boundary-Plasticity from Superplasticity: *Kenji Higashi*[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1, Gakuen-cho, Sakai, Osaka 599-8531 Japan

Superplasticity is generally associated with fine grains, grain boundary sliding, accommodation, high tensile ductility and high strain-rate-sensitivity at elevated temperatures. The attention is paid to the high-strain-rate superplasticity and/or low-temperature superplasticity of the fine-grained materials in order to shed light on the mechanism of superplastic flow. The recently reported results after the project entitled "Towards Innovation in Superplasticity (1997~1999)" will be summarized with focusing to (a) novel processing procedures to achieve ultra-fine structures, (b) pertinent deformation models and (c) new phenomena of low-temperature superplasticity and nanocrystalline superplasticity. The new research area of grain boundary plasticity, furthermore, will be discussed.

10:55 AM
Deformation of Superplastic Al2O3/Y-TZP Particulate and Particulate Laminate Composites: Jue Wang[2]; Desiderio Kovar[1]; *Eric M. Taleff*[1]; [1]University of Texas, Dept. of Mech. Engrg., 1 Univ. Sta., C2200, Austin, TX 78712-0292 USA; [2]University of Texas, Matls. Sci. & Engrg. Prog., 1 Univ. Sta., C2200, Austin, TX 78712-0292 USA

Particulate composites of Al_2O_3 and Y-TZP were some of the earliest and are still among the most successful superplastic ceramic composites. Superplastic Y-TZP is required in Al_2O_3/Y-TZP particulate composites to retain fine grain sizes in the Al_2O_3 phase, which normally coarsens at elevated temperatures. However, the high cost of Y-TZP relative to Al_2O_3 provides incentive to understand the effect volume fraction has on deformation behavior of this composite system in order to engineer composites which minimize the expensive Y-TZP phase while achieving desired behaviors. To this end, a number of particulate and particulate laminate Al_2O_3/Y-TZP composites have been investigated over a range of compositions. These composites were mechanically tested over a range of strain rates and a limited range of temperatures near 1350°C. Particulate laminate composites were tested in both the isostress and isostrain orientations. A constrained isostrain model is found to best describe, among current models, the behaviors of both particulate composites and particulate laminate composites tested in either the isostress or isostrain orientations. The similarity in behaviors between particulate laminates in the isostress and isostrain orientations is attributed to layer constraint, which enables the strongest layer to dominate deformation behavior.

11:15 AM
Texture and Microstructure of Superplastic 7475 Aluminum: *Alexandre J. Blander*[1]; Jerzy A. Szpunar[1]; [1]McGill University, Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada

Orientation imaging microscopy (OIM) and x-ray diffraction were used to determine the effect of the superplastic response with respect to texture, grain boundary character distribution (GBCD) and microstructure in a 7475 statically recrystallized aluminum alloy. Results indicate the initial normal behavior of a microstructure deforming mainly by accommodated grain boundary sliding which induces rapid texture and grain boundary randomization. At latter stages of deformation, the accommodation mechanism is disrupted at a threshold strain, where the deformation mechanism is altered and dislocations are observed be concentrated at triple junctions. After this threshold strain is reached, crystallographic slip is observed as well as unaccommodated grain boundary sliding which ultimately leads to cavitation and fracture. Cavitation occurs at grain boundaries and triple junction where dislocations are concentrated, and is a result of difference in deformation behavior of adjacent grains having different Taylor factors. The kernel average misorientation function of OIM was used to indicate the level of strain within the grains and how the accommodation mechanism, or lack of it, affects the internal grain strain.

11:35 AM
Studies of Void Growth and Interaction in a Eutectic Sn-Pb Alloy: *Matthew Mulholland*[1]; Victor Caraveo[1]; Tariq Khraishi[1]; Yu-Lin Shen[1]; Mark Horstemeyer[2]; [1]University of New Mexico, Mech. Engrg. Dept., MSC01-1150, Albuquerque, NM 87131 USA; [2]Mississippi State University, Mech. Engrg. Dept., 206 Carpenter Bldg., PO Box ME, Mississippi State, MS 39762 USA

In this work, an extensive set of parametric experimental studies have been performed on the plastic deformation of voids and their interaction in a superplastic solder alloy (eutectic Sn-Pb). To begin with, the strain-rate sensitivity, i.e. the m-curve, of the alloy has been fully characterized. The effect of the number of voids and their initial size on the rate of void growth has been investigated. Finite-element computations have also been performed to shed some light on the micromechanics of void deformation. Explanation of the ensuing ductility is attempted based on the m-curve.

Aluminum Reduction Technology: Emerging Technologies

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Alton T. Tabereaux, Alcoa Inc., Process Technology, Muscle Shoals, AL 35661 USA

Wednesday AM, March 17, 2004 — Room: 213D — Location: Charlotte Convention Center

Session Chair: Halvor Kvande, Hydo Aluminium, Metal Prod. Div., Oslo N-0240 Norway

8:30 AM
Integrated Multiphysics & Computational Fluid Dynamics Modeling of a Carbothermic Aluminium Reactor: *Dimitrios I. Gerogiorgis*[1]; B. Erik Ydstie[1]; [1]Carnegie Mellon University, Dept. of Chem. Engrg., Doherty Hall DH 3112, 5000 Forbes Ave., Pittsburgh, PA 15213 USA

The present simulation study elaborates on a FE CFD model (Gerogiorgis et al., 2003) developed for the core stage of a carbothermic reduction reactor (Johansen and Aune, 2002), which is aimed at the industrial implementation of carbothermic aluminium production. Carbothermic reduction is an alternative to the conventional Hall-Héroult electrolysis process and is characterized by cost and environmental advantages and by its challenging complexity. The quadruple PDE problem (electric charge, heat, momentum and molar species balances) for the slag flow in the core reactor stage is solved using a commercial solver (FEMLAB® v. 2.3), to obtain potential, temperature, velocity and species concentration distributions in a 2D domain representing the complete core second stage of a proposed carbothermic reactor design. The interaction among Joule heating, endothermic reaction, natural Boussinesq convection and turbulent flow phenomena is of paramount importance for understanding the performance of the core stage; conducting CFD simulations in order to advance with the latter goal is very important, since reliable high-temperature mea-

surements are remarkably costly and laborious. The main objective of this CFD study is to extract reactor design guidelines and conclusions about the reactive slag flow, under the instantaneous thermodynamic equilibrium assumption. Explicitly addressing the effect of design parameters on state variable distributions is vital: the sensitivity analyses conducted with respect to crucial process design variables reveal the controllability margin of the ARP reactor, exposing nontrivial heating optimization problems.

8:55 AM
Alumina Solubility Study in Ionic Liquids with PF_6 Anion: *Mingming Zhang*[1]; Ramana G. Reddy[2]; [1]University of Alabama, Dept. of Metallurgl. & Matls. Engrg., 254 Bevill Bldg., 126 Seventh Ave., Tuscaloosa, AL 35487 USA; [2]University of Alabama, ACIPCO, Ctr. for Green Mfg., Dept. of Metallurgl. & Matls. Engrg., PO Box 870202, A-129 Bevill Bldg., 126 Seventh Ave., Tuscaloosa, AL 35487-0202 USA

Traditional cryolite electrolysis process in the production of aluminum has severe environmental problems and are extremely energy intensive. High temperature electrolysis(900-1000°C) inevitably has high production costs and pollutants emission. While using ionic liquids as electrolyte near room temperature can easily avoid these problems. In this paper, the solubility of alumina in several kinds of ionic liquids with PF_6 anion was studied. Two different methods(emf, mechanic stirring) were used to determine the solubility. The solubility of alumina in ionic liquids was found to be relatively low as comparing to that in molten cryolite. The measured solubility also depended on method used to determine the dissolved alumina. From 100°C or higher temperature to their decomposition points, C_6mimPF_6 and C_8mimPF_6 exhibited measurable ability to dissolve alumina, which reach about 1 wt% and 0.5 wt% respectively at 200°C. Possible methods were also given in order to enhance the solubility of alumina in ionic liquids.

9:20 AM
New Opportunities for Aluminum Electrolysis with Metal Anodes in a Low Temperature Electrolyte System: *Jianhong Yang*[1]; John N. Hryn[1]; Greg K. Krumdick[1]; Joseph A. Pomykala[1]; Boyd R. Davis[2]; Alain Roy[2]; [1]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA; [2]Kingston Process Metallurgy, Inc., Kingston, Ontario Canada

A new electrolyte system for low-temperature aluminum electrolysis was investigated. Analysis indicated that the solubility of alumina was about 4 wt% at 700°C. Baseline electrolysis tests at 700°C (graphite materials as both anode and cathode) showed the process works well with automatic alumina feeding. Metal anodes have been tested at 700°C in 10A, 20A and 100A cells for up to 100 hours at an anode current density of 0.5 A/cm2. With TiB2-C composite as the cathode, the current efficiency was around 85% based on recovered aluminum. Aluminum product purity was typically better than 99.5 wt%, with the major impurity being copper, typically less than 0.2 wt%. The promising results suggest that aluminum electrolysis with inert metal anodes in the new low temperature electrolyte system can be realized. This work is supported by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy under contract W-31-109-Eng-38.

9:45 AM
Laboratory Test and Industrial Application of a Ambient Temperature Cured TiB2 Cathode Coating for Aluminum Electrolysis: Qingyu Li[1]; *Jie Li*[2]; *Yonggang Liu*[3]; Yanqing Lai[2]; Jianhong Yang[2]; Yexiang Liu[2]; [1]Guangxi Normal University, Chmst. Dept., Gueilin, Guangxi 5410043 China; [2]Central South University, Sch. of Metallurgl. Sci. & Engrg., Changsha, Hunan 410083 China; [3]Aluminum Corporation of China Limited Guangxi Branch, Pingguo, Guangxi 531400 China

A concept of ambient temperature curable TiB2 cathode coating is put forward for aluminum electrolysis, and the ambient temperature curable TiB2 cathode coating has been prepared successfully. Differing from previous TiB2 cathode coating solidified approximately at 200?•, the ambient temperature curable TiB2 cathode coating can be solidified at room temperature, so the heating equipment is not necessary, which simplifies the preparation process and facilitates the industrial application of TiB2 cathode coating. Laboratory test results show that the properties of the cathode coating are excellent, the electrical resistivity is 23.8?Ê?¶.m, the compressive strength is 33.6MPa, and sodium expansion of cathode in aluminium electrolysis is reduced about 50%. Results from commercial prebaked anode aluminium electrolysis cells show that the coated cells have much better current distribution, lower cathode voltage drop, higher current efficiency, and less sodium related damage compared to the contrastive cells.

10:10 AM Break

10:20 AM
Iron-Nickel Alloy Slow-Consumable Anode for Aluminum Electrolysis: *Zhongning Shi*[1]; Junli Xu[1]; Zhuxian Qiu[1]; [1]Northeastern University, Light Metals Metall. Inst., Sch. of Matls. & Metall., PO Box 117#, Shenyang, Liaoning 110004 China

An alloy Fe-43Ni (wt.%) made by powder metallurgy as an anode for aluminum electrolysis is described in the present paper. Electrolysis by the anode was conducted for ten hours at 850°C in molten salts which consisted of NaF-43.7%AlF3-8%NaCl-5%CaF2-4%Al2O3 by mass. The anodic current density was set at 0.75A/cm2. Electrolysis was conducted smoothly with a fluctuation of cell voltage within 3.8-4.1V. The results by Electron Probe Micro-analyzer and X-ray Diffraction show that the generation film on the anodic substrate consisted of iron oxide major phase and nickel oxide, nickel ferrite minor phase. The alloy anode shows good performance in terms of anti-oxidation and resistant corrosion. Average annual wear rate is 20.77 millimeter, and purity of primary aluminum is 97-98%.

10:45 AM
An Improved Pyroconductivity Test of Spinel-Containing Cermet Inert Anodes in Aluminum Electrolysis: *Yanqing Lai*[1]; *Jie Li*[1]; Zhongliang Tian[1]; Gang Zhang[1]; Qingwei Qin[1]; Qingyu Li[1]; Yexiang Liu[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., 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 maintain good 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 behavior were studied, which included the particle size of raw materials, manufacture process, phase composition and morphology et al.

Beyond Nickel-Base Superalloys: Niobium Silicides

Sponsored by: Structural Materials Division, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), SMD-High Temperature Alloys Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Refractory Metals Committee
Program Organizers: Joachim H. Schneibel, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA; David A. Alven, Lockheed Martin - KAPL, Inc., Schenectady, NY 12301-1072 USA; David U. Furrer, Ladish Company, Cudahy, WI 53110 USA; Dallis A. Hardwick, Air Force Research Laboratory, AFTL/MLLM, Wright-Patterson AFB, OH 45433 USA; Martin Janousek, Plansee AG Technology Center, Reutte, Tyrol A-6600 France; Yoshinao Mishima, Tokyo Institute of Technology, Precision and Intelligence Laboratory, Yokohama, Kanagawa 226 Japan; John A. Shields, HC Stark, Cleveland, OH 44117 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA

Wednesday AM Room: 211B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Kazuhiro Ito, Kyoto University, Matls. Sci. & Engrg., Kyoto 606-8501 Japan; Hongbin Bei, University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996 USA

8:30 AM Invited
A Comparative Overview of Mo- and Nb-Base Metal/Silicide Systems for High Temperature Structural Applications: *Madan G. Mendiratta*[1]; Sarath K. Menon[1]; Triplicane A. Parthasarathy[1]; Dennis M. Dimiduk[2]; Dallis A. Hardwick[2]; Patrick L. Martin[2]; [1]UES Inc., Matls. & Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA; [2]AFRL/MLLMD, Metals, Ceram. & NDE Div., 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA

Research and development efforts are presently underway to explore alloys within Mo-Si-B and Nb-Ti-Cr-Si-Hf-Al-Sn systems with a goal to significantly exceed the temperature capability of the current

Ni-base superalloy for jet engines. Both these alloys systems consist of a refractory metal phase for fracture resistance and silicides and other intermetallic phases for high temperature oxidation and creep resistance. This presentation is a comparative overview of the progress made on the systems through continuing research at AFRL/UES and other organizations. Physical properties (density, melting point, thermal conductivity, and thermal expansion coefficients) will be discussed in the context of design. Presentation will also include compositions, processing, microstructural evaluation, oxidation behavior and mechanical properties. Cyclic oxidation experiments have been carried out from 600-1350°C and tensile, compressive, fatigue, toughness and creep properties have been determined from RT-1400°C. T he oxidation mechanisms as well as fracture and damage mechanisms will be compared for the two alloy systems. Results of on-going research as well as venues for further critical work will be presented. Worked performed on AFRL/MLLM Contract #F33615-01-C-5214.

9:00 AM

Phase Stability and Conceptual Microstructures for the Design of Nb-Base Superalloys: *Gautam Ghosh*[1]; Axel van de Walle[1]; Mark D. Asta[1]; Greg B. Olson[1]; [1]Northwestern University, Dept. of Matls. Sci. & Engrg., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

As a part multi-institutional and multi-disciplinary research under AFOSR-MEANS program, we present phase stability modeling and conceptual microstructures for the design Nb-base superalloys, with emphasis on conceptual design principles and tools employed. In the absence of sufficient experimental information, successful integration of ab initio phase stability data with CALPHAD fomalism constitutes one of the key components of this research. To demonstrate the efficacy of this approach, we present the results phase stability modeling of Nb-Al-Pd-Hf system. To design microstructures analogous to Ni-Base superalloys, bcc-based ordered aluminides (B2 and Heusler) are chosen as strengthening phases for Nb-base alloys. Prototype ternary and quarternary alloys heat treated at 1000 and 1200C over extended time show the evidence for high microstructural stability.

9:15 AM

Microstructure Evaluation of LENS™ Deposited Nb-Ti-Cr-Si Alloys: *Ryan Richard Dehoff*[1]; Peter C. Collins[1]; Hamish L. Fraser[1]; Michael J. Mills[1]; [1]Ohio State University, Matls. Sci. & Engrg. Dept., 2041 College Rd., Columbus, OH 43210 USA

Nb-Si "in-situ" metal ceramic composites consist of Nb_3Si and Nb_5Si_3 intermetallic phases in a body centered cubic Nb solid solution, and show promising potential for elevated temperature structural applications. The drawback to the binary Nb-Si system is oxidation and high metal loss rate at elevated temperatures however, the addition of Cr and Ti have been shown to increase the oxidation resistance at high temperatures. In this study, the LENS™ (Laser Engineered Net Shaping) process is being implemented to construct the Nb-Ti-Cr-Si alloy system from elemental powder blends due to availability of material and low relative cost. Advantages of the LENS™ process include the ability to produce near net shaped components with graded compositions as well as a more uniform microstructure resulting from the negative enthalpy of mixing associated with the silicide phases. The optimum LENS™ processing parameters were determined to produce a graded composition with a Nb:Ti ratio at 2:1, a Cr composition of 10 at %, and a range of Si contents from 5 to 15 at.%. The various microstructures were examined using SEM and TEM techniques. Hardness measurements and microtensile testing are being performed to evaluate the mechanical behavior of these alloys.

9:30 AM

Microstructural Properties of Nb-Si-B Alloys: *Stefan Drawin*[1]; Pierre Petit[1]; [1]ONERA, DMMP, BP72, Châtillon 92322 France

Refractory metal silicide based alloys are currently studied as candidate materials for future high performance turbine engines submitted to high airfoil temperatures (up to 1300°C). Addition of boron may improve their oxidation resistance, which remains a crucial issue for these materials. This has been applied to both the Mo-Si system and, more recently, to the Nb-Si system. Alloys with compositions in the Nb-rich corner of the Nb-Si-B system have been prepared by multiple cold crucible arc-melting and homogenised at high temperature. The microstructural properties of theses alloys have been investigated using scanning electron microscopy, electron probe micro-analysis and X-ray diffraction and will be presented. Rietveld analysis has been used to investigate the structure of each phase.

9:45 AM

Phase Relation and Mechanical Property of Nb-Si Based Refractory Intermetallic Alloys with Ternary Elements: *Won-Yong Kim*[1]; In-Dong Yeo[1]; Sung-Hwan Lim[2]; [1]Korea Institute of Industrial Technology, Advd. Matls. R&D Div., 472 Kajwa-dong, Suh-ku, Inchon 404-254 Korea; [2]Kangwon National University, Dept. of Advd. Matls. Sci. & Engrg., Chunchon, Kangwon 200-701 Korea

Ternary phase diagrams, microstructure and mechanical properties of Nb-Si based refractory intermetallic alloys containing ternary alloying element were investigated. Molybdenum and Vanadium were chosen as ternary alloying elements because of their high melting point and smaller atomic size than Nb in order to expect a high temperature strength and room temperature fracture toughness. It was found that both ternary alloying elements have a significant effect to modify the microstructure from dispersed structure to maze-like structure in Nb solid solution/Nb_5Si_3 intermetallic composites. 0.2% offset yield strength at room temperature increased with increasing content of ternary elements in Nb solid solution and volume fraction of Nb_5Si_3 for both ternary alloy systems. At 1773 K, Mo addition has a positive role to increase yield strength, on the contrary V addition has a role to decrease yield strength. Fracture toughness of ternary alloys was superior to the binary alloys.

10:00 AM Break

10:30 AM

Oxidation Resistance and Mechanical Properties of a Powder Metallurgical (PM) Nb-Silicide Alloy: *P. Jéhanno*[1]; M. Boening[1]; A. Venskutonis[1]; B. Bewlay[2]; M. R. Jackson[2]; [1]Plansee AG, Tech. Ctr., Reutte in Tirol 6600 Austria; [2]General Electric, Global Research, Schenectady, NY USA

A niobium-silicide alloy with additions of Ti, Hf, Cr and Al was manufactured using a powder metallurgical processing route comprising melting and atomization of a pre-sintered ingot. After compaction of the powder via HIP, the material was extruded using a reduction in area of 6:1. The microstructural characterization of both HIPed and extruded materials occurred via SEM, EDS and XRD analysis. The microstructure consisted of a Nb solid solution surrounding intermetallic particles with Nb5Si3- and Nb3Si-type structure. Tensile tests were performed at temperatures ranging from 800°C to 1300°C. A strength level equivalent or in some cases superior to directionally solidified alloys was observed. Furthermore, the excellent oxidation resistance of the base material was confirmed by isothermal heat treatments in air at temperatures ranging from 800 to 1200°C. The microstructures and mechanical properties will be compared with those of cast alloys.

10:45 AM

Atomic Diffusion and Phase Equilibria at the Interfaces of CoAl/Ir Multi-Layer on Co_3AlC- and Nb_5Si_3-Base Alloys: *Yoshisato Kimura*[1]; Sachiyori Shiina[2]; Tatsuya Shimizu[2]; Yoshinao Mishima[1]; [1]Tokyo Institute of Technology, Matls. Sci. & Engrg., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa 226-8502 Japan; [2]Tokyo Institute of Technology, Matls. Sci. & Engrg., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa 226-8502 Japan

Protection against severe oxidation damages is an inevitable issue of heat resistant alloys and it becomes much more important at significantly high temperatures beyond 1273 K. Multi-layered functional coating is one of attractive solutions without sacrificing mechanical properties. Phase equilibria information plays a quite important role in designing heat resistant alloys since atomic diffusion is extremely active at such high temperatures. In the present work, we selected B2-CoAl as Al-reservoir for Al_2O_3 formation and Ir solid-solution as diffusion barrier especially against Al inward-diffusion. Base materials are $E2_1$- Co_3AlC based and Nb_5Si_3 based heat resistant alloys. Atomic diffusion and phase equilibria have been investigated in detail using several diffusion couples. To evaluate the potential of Ir as diffusion barrier, temperature dependence of the diffusion coefficient of Al in Ir solid-solution (Ir-8at%Al) has been quantitatively determined, for instance 8.9×10^{-19} m^2/s at 1573 K, using Boltzmann-Matano model with electron probe micro-analysis.

11:00 AM

Processing, Microstructure and Mechanical Properties of (Nb)/Nb_5Si_3 Two-Phase Alloys: *Yoshinao Mishima*[1]; Yoshisato Kimura[1]; Nobuaki Sekido[2]; Hiroaki Yamaoka[1]; [1]Tokyo Institute of Technology, Dept. of Matls. Sci. & Engrg., 4259 Nagatsuta, Midoriku, Yokohama, Kanagawa 226-8502 Japan; [2]University of Wisconsin, Dept. of Matls. Sci. & Engrg., 1509 Univ. Ave., Madison, WI 53706 USA

It has been shown that a fine lamellar structure composed of Nb solid solution, (Nb), and Nb_5Si_3 is formed through eutectoid decomposition in the Nb-Si binary system and its ternary derivatives. Alloys with such microstructure would exhibit a high strength at over 1400 K yet showing room temperature toughness of over 20MPa $m^{1/2}$ if a proper lamellar spacing is chosen In the present work, effects of processing on the microstructure evolution and mechanical properties are investigated on the Nb-18at%Si alloys prepared by hot pressing

and spark plasma sintering(SPS). The powders used in the present work are of pure Nb and Nb_5Si_3 in order for the fabrication to become possible at temperatures higher than the melting point of Si and to reduce the formation of SiO_2. The results show that the SPS yields more uniform two-phase microstructure but the alloys fabricated by hot pressing tends to provide higher elevated temperature strength.

11:15 AM
Effect of Ternary Elements on the Eutectoid Decomposition Behavior of Nb_3Si into (Nb)/Nb_5Si_3 in the Nb-Si-X Alloys: *Seiji Miura*[1]; Miki Aoki[1]; Yasuhiko Saeki[1]; Kenji Ohkubo[1]; Yoshinao Mishima[2]; Tetsuo Mohri[1]; [1]Hokkaido University, Div. of Matls. Sci. & Engrg., Kita-13, Nishi-8, Kita-ku, Sapporo, Hokkaido 060-8628 Japan; [2]Tokyo Institute of Technology, Dept. of Matls. Sci. & Engrg., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa 226-8502 Japan

Alloys based on Nb-aluminides and silicides have been shown to exhibit superior high temperature strength than the commercial nickel base superalloys, however lack of room temperature ductility and high temperature oxidation resistance is the major drawback for further development. In order to improve the toughness of these alloys, microstructure control is a key method by which ductile phase toughening could effectively play a role in the alloys based on brittle intermetallic compounds. In the present work, effect of various ternary additions on the microstructure evolution of (Nb)/Nb_5Si_3 lamellar structure is investigated in the Nb-Si-X alloys. It has been shown that the kinetics of the eutectoid decomposition of high temperature Nb_3Si into (Nb) and Nb_5Si_3 was sluggish in the binary Nb-Si system and that it is enhanced by Ti additions. The TTT diagrams for the decomposition experimentally determined and the microstructure of the products formed under various conditions is observed in the ternary system with an element such as Zr. Then the role of the ternary element on the decomposition kinetics is discussed.

11:30 AM
Study of the Effects of Ti, Mo and B Additions on the Microstructure of Nb-Silicide Based *In Situ* Composites: *Jie Geng*[1]; Panayiotis Tsakiropoulos[1]; Guosheng Shao[1]; [1]University of Surrey, Sch. of Engrg., Mech. & Aeros. Engrg., Metall. Rsch. Grp., Guildford, Surrey GU2 7XH England

The effects of Ti, Mo and B additions on phase selection and microstructure development in as cast and heat-treated Nb-Si-Cr-Al in situ composites have been studied. In the alloys the Cr + Al and Si contents and the Si/(Cr+Al) ratio have been selected for optimum oxidation resistance and creep behaviour. The alloys were prepared using clean melting and casting in water-cooled copper crucibles/moulds. Molybdenum offers solid solution strengthening and Ti and B play important roles in phase selection and microstructure control in both the as cast and the heat treated alloys. In our study particular attention has been paid on the dependence of phase selection on cooling rate in ingots and on phase equilibria involving the Nbss, the MxSiy (M=Mo, Nb, Ti), T2 and Laves phases. The results of microstructural characterization will be presented and discussed.

Bulk Metallic Glasses: Bio, Corrosion, and Fracture Behavior

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

Wednesday AM Room: 209A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Joe A. Horton, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37830 USA; John J. Lewandowski, Case Western Reserve University, Matls. Sci. & Engrg., Cleveland, OH 44106 USA

8:30 AM Invited
Biomedical Applications of Bulk Metallic Glasses: *Joe A. Horton*[1]; Doug E. Parsell[2]; Mark L. Morrison[3]; Don M. Nicholson[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S-MS6115, PO Box 2008, Oak Ridge, TN 37830 USA; [2]University of Mississippi, Dept. of Biomatls., Med. Ctr., Jackson, MS 39216 USA; [3]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37966-2200 USA

Bulk metallic glasses (BMG) have a number of unique mechanical properties including a high elastic limit, low modulus, high tensile strength and reasonable hardness and toughness that make them attractive for some biomedical applications. For future implant materials, magnetic properties are also critical due to possible need for subsequent MRIs. Even with 14.6% Ni, MRI images of our reference material are excellent. First applications of BMG materials in the biomedical arena may be as tools used in interventional MRI procedures where imaging characteristics of the tools are critical. Preliminary biocompatability, corrosion, and fatigue results will be presented. This research was sponsored by the ORNL seed money program and by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC05-00OR22725 with ORNL operated by UT-Battelle, LLC.

8:55 AM Cancelled
Zirconium-Based Bulk Metallic Glasses for Biomedical Applications

9:20 AM
Comparisons of Aqueous Corrosion Behaviors of Zr-Based Bulk Metallic Glasses and Nanocomposites: *Brandice Green*[1]; William Peter[1]; Raymond Buchanan[1]; Yoshihiko Yokoyama[2]; Peter Liaw[1]; Mark Morrison[1]; [1]University of Tennessee, Matls. Sci. & Engrg., 434 Dougherty Engrg. Bldg., Knoxville, TN 37996-2200 USA; [2]Himeji Institute of Technology, Matls. Sci. & Engrg., Shosha 2167, Himeji City Japan

To date, the research on the corrosion properties of Zr-based bulk metallic glass (BMG) nanocomposites has been limited. Most reported research on corrosion properties has been on fully amorphous Zr-based BMGs and has focused on one chemical composition. Furthermore, few studies have investigated the possible similarities and contrasts among nano-structured BMGs and completely amorphous BMGs. In the present work, the aqueous electrochemical corrosion behaviors of $Zr_{50}Cu_{40}Al_{10}$, $Zr_{50}Cu_{30}Al_{10}Ni_{10}$, and nano-structured $Zr_{50}Cu_{37}Al_{10}Pd_3$ were studied. Cyclic-anodic-polarization, immersion, and dynamic-anodic-polarization experiments were conducted on each composition in a 0.6 M NaCl electrolyte (simulated seawater) at room temperature. The cyclic-anodic-polarization results showed that $Zr_{50}Cu_{30}Ni_{10}Al_{10}$ exhibited passive behavior at the open-circuit corrosion potential with a low corrosion rate. However, $Zr_{50}Cu_{30}Ni_{10}Al_{10}$ was found to be susceptible to pitting corrosion at elevated potentials. On the other hand, $Zr_{50}Cu_{40}Al_{10}$ was found to be highly susceptible to pitting corrosion, with pit initiation occurring at, or slightly above, the corrosion potential and accelerating at higher potentials. To confirm the polarization results, BMG samples were immersed in the 0.6 M NaCl electrolyte and observed to determine incubation-time periods for pit initiation. Pit initiation did not occur over a 3-hour time period for $Zr_{50}Cu_{30}Ni_{10}Al_{10}$. However, pit initiation occurred within 15 minutes for $Zr_{50}Cu_{40}Al_{10}$. Preliminary observations indicated that the nano-structured $Zr_{50}Cu_{37}Al_{10}Pd_3$ will have significantly different corrosion properties than the amorphous BMG compositions. This research effort was made possible by the funding of the National Science Foundation Integrative Graduate Education and Research Training (IGERT) Program on "Materials Lifetime Science and Engineering" (DGE-9987548), with Drs. W. Jennings and L. Goldberg as contract monitors and by Tennessee Advanced Materials Laboratory (TAML).

9:45 AM
Electrochemical Studies of a Zr41.2 Ti13.8 Ni10 Cu12.5 Be22.5 Bulk Amorphous Alloy in Physiologically-Relevant Environments: *Mark L. Morrison*[1]; Raymond A. Buchanan[1]; Atakan Peker[2]; William H. Peter[1]; Joe A. Horton[3]; Peter K. Liaw[1]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 434 Dougherty, Knoxville, TN 37996-2200 USA; [2]Liquidmetals Technologies, 25800 Commercentre Dr., Ste. 100, Lake Forest, CA 92630 USA; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS6115, Oak Ridge, TN 37831-6115 USA

Cyclic-anodic-polarization tests were conducted on a Zr-based bulk amorphous alloy with a chemical composition of $Zr_{41.2}Ti_{13.8}Ni_{10}Cu_{12.5}Be_{22.5}$ (at.%). Samples were compared in two different electrolytes. A series of tests were conducted in an aerated 0.6 M NaCl electrolyte at room temperature and in a phosphate-buffered saline (PBS) electrolyte with a physiologically-relevant dissolved oxygen content at 37°C. For both electrolytes, the alloy demonstrated passive behavior up to the mean pitting potentials. The mean corrosion penetration rates were low but a susceptibility to localized pitting corrosion was observed in both electrolytes. Furthermore, the differences between the protection potentials and the open-circuit corrosion potentials in each electrolyte

were relatively low. Thus, the material may undergo pitting corrosion at surface flaws or after incubation time periods, depending upon the local and bulk environments.

10:10 AM
H Charging of a Zr-Based Bulk Metallic Glass: Ping Wang[1]; *Sharvan Kumar*[1]; [1]Brown University, Div. of Engrg., 182 Hope St., Box D, Providence, RI 02912 USA

The potential to use a Zr-based bulk metallic glass as a hydrogen storage medium is explored. Hydrogen was cathodically charged into the glass using an electrochemical cell with acid and base media, and to a lesser extent by gas charging at temperature and pressure. Charging parameters were systematically varied including current density, solution concentration and solution temperature; the effects of pre-anneal and intermediate anneal on charging kinetics were also examined. A significant amount of hydrogen can be introduced in the glass before it exhibits cracks (the maximum in this study being H/M = 1.46). In all cases, the material remains amorphous. Attempts to extract the hydrogen from the glass were however futile; the residual hydrogen interferes with the devitrification process and also affects the glass transition temperature. These results will be discussed and the potential for bulk metallic glasses to store and release hydrogen will be examined.

10:35 AM Invited
Effects of Changes in Loading Mode on Bulk Metallic Glasses and Bulk Metallic Glass Composites: *Alex K. Thurston*[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Matls. Sci. & Engrg., 10900 Euclid Ave., White Bldg., Cleveland, OH 44106 USA

Bulk Metallic Glass composites are of interest because of the unique properties of the amorphous structure, while utilizing some additional deformation capabilities provided by the composite additions. Tests are being conducted on the Bulk Metallic Glass as well as the composites in both notched bending and in fatigue precracked conditions. The effects of changes in the test temperature on mechanical properties and fractography will be presented. In addition, the effects of changes in loading mode on the fracture behavior are being determined.

11:00 AM Cancelled
Glass Formation Ability and Mechanical Properties of Cu-Zr-Ti-Sn Bulk Metallic Glasses

11:25 AM
Internal Strain Measurements in Bulk Metallic Glasses (BMG) and BMG Composites Using Pair Distribution Function (PDF) Analysis: *Bjorn Clausen*[1]; Thomas E. Proffen[1]; Seung-Yub Lee[2]; Ersan Ustundag[2]; [1]Los Alamos National Laboratory, LANSCE-12, PO Box 1663, MS H805, Los Alamos, NM 87545 USA; [2]California Institute of Technology, Matls. Sci. Dept., Keck Laboratory, M/C 138-78, 1200 E. California Blvd., Pasadena, CA 91125 USA

The development of bulk metallic glass (BMG) matrix composites is driven by the need to improve the ductility and failure behavior over the monolithic BMG. Similar developments have taken place for metal and ceramic matrix composites (MMCs and CMCs). Neutron powder diffraction have been used extensively in this research to in-situ determine the load sharing between the phases in MMC and CMC composites with crystalline phases. However, the powder diffraction technique is not capable of yielding information about the BMGs due to their amorphous structure. In the present work we have used pair distribution function (PDF) analysis - also called the "total scattering technique" - to obtain information about the changes in inter atomic distances in the BMG as a function of applied load. In-situ neutron PDF measurements were made during compression tests of Vitreloy 106 ($Zr_{57}Nb_5Al_{10}Cu_{15.4}Ni_{12.6}$) monolith and composite samples using the SMARTS and NPDF instruments at LANSCE.

Carbon Technology: Anode Quality and Performance

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Markus Meier, R&D Carbon, Sierre CH 3960 Switzerland; Amir A. Mirchi, Alcan Inc., Arvida Research and Development Centre, Jonquiere, QC G7S 4K8 Canada; Alton T. Tabereaux, Alcoa Inc., Process Technology, Muscle Shoals, AL 35661 USA

Wednesday AM Room: 213A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Jim Kissane, Mozal Aluminium Smelter, Maputo Mozambique

8:30 AM
Optimization of Rodding Room Operation to Enhance Productivity: *Masood Talib Al Ali*[1]; Raja Javed Akhtar[1]; Saleh Ahmad Rabba[1]; [1]Dubal Aluminium Company Limited, PO Box 3627, Dubai United Arab Emirate

In 1989 DUBAL retrofitted its Rodding Room to support production of over 536,000 tonnes of aluminium per year. The retrofitted plant was designed to produce 750 anodes in two-shifts operation of 8 hours each. The third shift was dedicated entirely to carryout maintenance activities and a built in spare capacity for the security of the plant operation. During 2001/2002 a decision was made to increase DUBAL metal out put to 710,000 tonnes per annum under a project code named "Kestrel". Therefore, Anode Plant Management was given another challenging target of completing the current requirement of 750 anodes in 12 hours shift operation. Achieving this target was essential to maintain the status quo in the Roddng Operation. This paper describes step-by-step approach to identify bottlenecks, fine-tuning of plant and process followed by tests and trails along with other improvement to confirm the practicality of the proposal. The confidence achieved during the trials lead to a decision to carryout permanent modification as identified during the trials to enhance Rodding Room Operation to produce 920 anodes in two shifts of 8 hours each to support post Kestrel metal prduction without significant increase in its resources.

8:55 AM
Going Beyond SPC - Why We Need Statistical Thinking in Operations Such as Carbon Plants: *Keith Sinclair*[1]; Barry Alexander Sadler[2]; [1]Sinclair Associates Inc., 2006 Northwood Dr., Maryville, TN 37803 USA; [2]Net Carbon Consulting Pty Ltd., Unit 1, 21 Luck St., Eltham, Victoria 3095 Australia

Statistical thinking is based on the principles that all work occurs in interconnected processes, variation exists in all processes, and reducing this variation is the key to process improvement. The effective use of statistical methods such as Statistical Process Control requires that an implementation framework be established through statistical thinking. Several examples relevant to Carbon Plant operations are provided where the failure to apply statistical thinking to process monitoring and improvement has resulted in waste and lost opportunities. Some appropriate actions for Managers in applying statistical thinking are then outlined.

9:20 AM
Survey on Worldwide Prebaked Anode Quality: *Raymond C. Perruchoud*[1]; Markus W. Meier[1]; Werner K. Fischer[1]; [1]R&D Carbon Ltd., PO Box 362, Sierre 3960 Switzerland

Anode quality data of 60 prebaked carbon plants were reviewed. The ranges and mode values of the means and of the variability of anode properties were determined and discussed. Some examples of distribution of properties are examined. Causes of inferior or excellent anode quality figures were considered and their effects on the anode behavior and on pot performance are addressed. Interrelationships of thermal shock relevant properties are reported. The bench mark anode quality that can be achieved in a modern plant using typical raw materials is also given.

9:45 AM
Outlook of the Anode Requirement for the World Aluminium Industry in 2015: Werner K. Fischer[1]; *Ulrich Mannweiler*[2]; Age J. de Vries[3]; [1]R&D Carbon Ltd., PO Box 362, Sierre 3960 Switzerland; [2]Mannweiler Consulting, Hadlaubstrasse 71, Zurich 8006 Swtzerland; [3]Research and Development Services W.L.L., Manama Bahrain

Aluminium is a metal with unbroken growth - with 25 Mio. tons produced in 2002 and a growth rate between 2 to 3%, an additional smelter capacity of more than 500'000 tpa is required each year. A shift of classical production locations can be observed from Europe and USA to new locations with sea port access and low energy cost. A shift can also be observed from many small to few large producers with increased current intensities. Soederberg cells are replaced by prebaked anode technology in particular for brownfield expansions. Accordingly an adapted trend for the anode production sites can be observed. Thereby main focus is given to optimize the specific production cost. This document gives an assumption about the worldwide anode requirement up to 2015 by taking into consideration today's situation and the changing needs.

Cast Shop Technology: Alloying and Furnace Processing

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; Alton T. Tabereaux, Alcoa Inc., Process Technology, Muscle Shoals, AL 35661 USA

Wednesday AM Room: 213B/C
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Denis Bernard, Alcan Primary Metal Group, Laterriere Works, Laterriere, QC G7N 1A2 Canada; David H. DeYoung, Alcoa Inc., Alcoa Tech. Ctr., Alcoa Ctr., PA 15069 USA

8:30 AM
A Method for Prediction of Compacting Behaviour and Mechanical Resistance of Mn Compacts for Aluminium Alloying: Ricardo Fernández-Serrano[2]; Gaspar González-Doncel[2]; Raquel Antolín[1]; Tomás Posada[1]; *Gregorio Borge*[1]; [1]Bostlan SA, Techl., Polig Ind Trobika, Mungia 48100 Spain; [2]Centro Nacional de Investigaciones Metalúrgicas (CENIM-CSIC), Phys. Metall., Gregorio del Amo, 8, Madrid 28040 Spain

Mn is usually added to aluminium furnaces using compacted mixtures of Mn and Al powders. Many Al producers are highly concerned with breakage of the compacts during transportation and handling, since Mn fine powders release due to compacts weakness can lead to low recoveries and safety concerns during alloying. Mechanical resistance of compacted powders depend on different factors, such as shape and size of the compact, kind of pressing machine, applied pressure, and quality of the raw materials. This work presents an experimental, systematic, and reproducible method for establishing a useful mechanical resistance concept for Mn-Al compacts. Different Mn-Al minitablets have been studied using this method, and an empirical model for predicting the mechanical behaviour of Mn compacts has been developed. The method allows to predict the mechanical resistance of Mn80, Mn85, Mn90, and Mn95 compact powders considering characteristics of the raw materials and the pressing process.

8:55 AM
Aluminum Weighing Measurement in Tilting Furnaces: *Daniel Audet*[1]; Luc Parent[2]; Marlene Deveaux[2]; John Courtenay[3]; [1]Universite du Quebec a Chicoutimi, 555 boul. de l'Universite, Chicoutimi, Quebec G7H 2B1 Canada; [2]BDHTech, 200 Clement-Gilbert, Chicoutimi, Quebec G7H 5B1 Canada; [3]MQP LTD, 6 Hallcroft, Knowle Soli Hull B93 9EW GB UK

The best way to prepare aluminum alloys is to know the heel weight at the end of the previous cast and the weight of pure aluminum added. Many systems have been tried to measure the aluminum weight in tilting furnaces, including load cells, lasers and radars. None of these systems are satisfactory. A measuring system based on the pressure in the piston used to tilt the furnace has been developed. It is easily retrofitted to the existing furnaces and measure weight with an accuracy of ± 200 kg. The system can be used to monitor the weight either it is a heel or a completely full furnace. It can also measure weight in real time during the transfer from the holding furnace. Results of the system in plant operation will be presented.

9:20 AM
Combined Metal Skimming and Melt Treatment System for Metal Transport Crucibles: *Jon Hjaltalin Magnusson*[1]; [1]ALTECH JHM hf, Lynghals 10, Reykjavik IS 110 Iceland

In metal transported in large crucibles from potrooms to casthouse are impurities and on the metal surface is bath remains and dross. Aluminium smelters therefore install an automatic melt treatment and skimming system for cleaning the metal in these crucibles. The main aim is to improve metal quality (ISO 9001) and improve the skimming efficiency and at the same time the health, safety and environmental conditions for the employees (ISO 14001) by avoiding manual skimming. For melt treatment AlF_3 flux is recommended. Controlled by a PLC program, the flux will be injected into the melt and distributed by the double rotor of the Melt Treatment System. Before ending the treatment cycle the introduced treatment gas will support the purification of the melt by floating inclusions (e.g. aluminium carbides) to the surface and removing hydrogen for degassing. Before the melt purification cycle a Skimming System will remove dross and bath remains from the metal surface in the crucible and after the melt treatment it will repeat the surface cleaning before the metal is transported to the holding furnaces in the casthouse. The effective removal of the bath material, dross and impurities from the surface of the metal in the transport crucibles, ensures the cleanliness of the metal.

9:45 AM
In-Furnace Refining Using Pyrotek's HD-2000 and FIF-50 Rotary Injector Systems: *Robert A. Frank*[1]; Peter J. Flisakowski[1]; [1]Pyrotek, Inc., SNIF Systems, 100 Clearbrook Rd., Ste. 325, Elmsford, NY 10523-1116 USA

Demand for high quality aluminum, environmental restrictions, and economic pressures create the need for improvements in the melt treatment process. Pyrotek's HD-2000 is designed to replace conventional furnace treatments, such as steel flux wands, graphite flux tubes, and porous plugs. In addition, the combination of the HD-2000 with Pyrotek's FIF-50 can be used for injecting solid fluxes and refining agents. This paper describes the improvements in hydrogen, alkali metal, and inclusion removal of the HD-2000 system versus previous processing at a specific plant. Improvements in emissions, dross formation, production rate, and downstream metal quality are also documented.

10:10 AM Break

10:45 AM
Improved Molten Metal Quality at the Outlet of the Furnace Through the IRMA Treatment: *Pierre Le Brun*[1]; Alain Mathis[1]; [1]Pechiney, Ctr. de Recherches de Voreppe, 725, rue Aristide Berges, BP 27, Voreppe Cedex 38341 France

In the '80, Pechiney developed a furnace treatment technology aiming at a significant quality increase. The technology is based on rotary gas treatment of the molten metal. Since its first implementation, this technology has widely developed inside Pechiney casthouses. The paper reviews the basics of the technology. The improvements of the molten metal quality that can be obtained through this technology are presented in detail: hydrogen, inclusion, and alkaline or alkaline-earth elements removal. Environmental and economic issues are also discussed.

11:10 AM
Mathematical Modeling of Aluminum Refining by Rotary Injection: *Pierre Proulx*[1]; Fouzi Kerdouss[1]; Jean-François Bilodeau[2]; Sébastien Vaudreuil[2]; [1]Université de Sherbrooke, Chem. Engrg., 2500 Blvd. Université, Sherbrooke, Québec J1K 2R1 Canada; [2]Alcan International Ltd., Arvida R&D Ctr., PO Box 1250, 1955 Blvd. Mellon, Jonquière, Québec G7S 4K8 Canada

The successful implementation of rotary injection technologies for molten metal treatment depends on the understanding and control of the complex fluid dynamics involved. Computational fluid dynamics offer a powerful tool in order to understand and optimize the rotary flux injection process in holding furnaces (RFI). In the proposed work, the impeller is explicitly described in three dimensions using a Multiple Reference Frames model. Dispersed gas bubbles and molten salt droplets dynamics in the turbulent molten metal are modeled using an Eulerian-Eulerian approach with a dispersed k-epsilon turbulent model. Bubble and droplet size distribution dynamics account for coalescence and break-up mechanisms. The alkali removal reaction is assumed to be controlled by mass transfer. The reaction rate is then determined by the interfacial area calculated from the bubble and droplet dynamics. The modeling results are compared with experimental data for fluid dynamics and chemical reaction.

11:35 AM
A Kinetic Study on the Magnesium Removal from Molten Aluminum Using SF_6 Gaseous Mixtures: *Alfredo Flores*[1]; David Villegas[1]; Marco Antonio González[1]; Jose Escobedo; [1]CINVESTAV-IPN, Unidad Saltillo, PO Box 663, 25000 Saltillo, Coahuila México

Magnesium removal from molten aluminum scrap has been currently performed at cast shop level in industrial facilities. However, chlorine is still in use in many foundries, with the ecological and technical problems concerning its usage. Instead of using chlorine, gaseous mixtures containing small amounts of SF_6, O_2, in argon are proposed, as strong oxidizing conditions for selective magnesium removal are attained. In this paper, a kinetic study is presented, using pneumatic injection through porous lances. The magnesium removal rates were measured as a function of temperature, gas flow rate, composition of the gaseous mixtures, and design of the injection lance. High removal efficiencies, in the order of 97%, were obtained for a combination of operating conditions. Analysis of solidified samples, slags and fumes produced permitted to determine the chemical reactions taking place. Finally, low losses of molten metal were obtained, in the order of 10% in weight.

CFD Modeling and Simulation of Engineering Processes: Process Modeling I

Sponsored by: Materials Processing & Manufacturing Division, ASM/MSCTS-Materials & Processing, MPMD/EPD-Process Modeling Analysis & Control Committee, MPMD-Solidification Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)

Program Organizers: Laurentiu Nastac, Concurrent Technologies Corporation, Pittsburgh, PA 15219-1819 USA; Shekhar Bhansali, University of South Florida, Electrical Engineering, Tampa, FL 33620 USA; Adrian Vasile Catalina, BAE Systems, SD46 NASA-MSFC, Huntsville, AL 35812 USA

Wednesday AM Room: 206A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Adrian S. Sabau, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37836-6083 USA; Kanchan Kelkar, Innovative Research, Plymouth, MN 55447 USA

8:30 AM Opening Remarks - Adrian Sabau

8:35 AM

Computational Fluid Dynamics: A Tool for the Materials Technology: *Aniruddha Mukhopadhyay*[1]; [1]Fluent Inc., Matls. Procg., 10 Cavendish Ct., Lebanon, NH 03766 USA

Starting with Chorin (1959) and Harlow (1965), over the past several decades Computational Fluid Dynamics (CFD) has come up as a matured analysis tool for conducting virtual experiments with significant time and cost savings. It complements and reduces physical testing. CFD has penetrated diverse materials industry much the same way as Computer-Aided-Design (CAD) did more than a decade ago. As the toolkits for background mathematical algorithms improved, reliability, consistency, and user-friendliness aspects improved significantly too. Recent trends have contributed to the rapid growth and widespread adoption of CFD. In this presentation, brief historical perspective of CFD applications in several materials processing industry will be presented using a few case studies. Emphasis will be on metals, glass and polymer applications.

9:10 AM

A Study of the Weld Pool Dimensions and Temperature Profiles Generated for GTA Welds of Gamma Titanium Aluminide (TiAl): Kirtikumar B. Bisen[1]; *Viola L. Acoff*[1]; Mario Arenas[1]; Nagy El-Kaddah[1]; [1]University of Alabama, Dept. of Metallurgl. & Matls. Engrg., 126 7th Ave., Tuscaloosa, AL 35401 USA

Stiff competition in industries requiring high strength, lightweight materials has forced manufacturers to seek ways to implement materials with more attractive properties. Gamma titanium aluminides are a class of materials that maintain their strength at high temperatures and are light in weight which make them attractive for automobile and aerospace applications. In both of these industries, fusion welding is used extensively to join the various components. Several results have been published on the structural characterization of gas tungsten arc welding (GTA) of gamma titanium aluminide however, only a few of these studies concentrate on the physics of arc welding. This study investigates the physics of arc welding as a function of welding parameters for GTA welding of gamma titanium aluminides. Experiment results were used to provide a comprehensive validation of the model. The experimental observations were in close agreement with the model predictions.

9:35 AM

Analysis of Heating Pattern With the Kind of Steel in Induction Heating Furnace: *K. H. Cho*[1]; Young-jin Jung[2]; [1]RIST, Energy Team, #32, Hyoja-Dong, Namku, Pohang, Kyung-buk 790-600 Korea; [2]Pusan National University, Mech. Engrg., 30, JangJunDong, KumJungKu, Pusan 609-735 Korea

Induction heating is a very common process for melting metals and alloys. If an alternating magnetic field is applied to the workpiece, it penetrates the surface of it and delivers heat within the material, heating it more rapidly and evenly than any other diffusion-dependent processes. However, some operating control condition with the variation of operating condition and the kind of steel is necessary to increase the quality and the productivity. A precise knowledge about the electromagnetic field, the eddy currents and the temperature distribution is necessary to optimize continuous induction heating process. Studies were conducted on the numerical model to predict the temperature of workpiece effectively and correctly. The validity of numerical model is proved by the comparison of calculated and measured temperature distributions through the workpiece. In the future, this could be extended to include more complex model and melting of any material including alloy and heat treating processes.

10:00 AM

Numerical Simulation of Vacuum Dezincing of Lead Alloy: *George Stefanov Djambazov*[1]; Chris Bailey[1]; Mayur K. Patel[1]; Jennifer Shrimpton[1]; [1]University of Greenwich, Computing & Math. Scis., Old Royal Naval College, Park Row, Greenwich, London, England SE10 9LS UK

Removing zinc by distillation can leave the lead bullion virtually free of zinc and also produces pure zinc crystals. Batch distillation is considered in a hemispherical kettle with water-cooled lid, under high vacuum (50 Pa or less). Sufficient zinc concentration at the evaporating surface is achieved by means of a mechanical stirrer. The numerical model is based on the multiphysics simulation package PHYSICA. The fluid flow module of the code is used to simulate the action of the stirring impeller and to determine the temperature and concentration fields throughout the liquid volume including the evaporating surface. The rate of zinc evaporation and condensation is then modelled using Langmuir's equations. Diffusion of the zinc vapour through the residual air in the vacuum gap is also taken into account. Computed results show that the mixing is sufficient and the rate-limiting step of the process is the surface evaporation driven by the difference of the equilibrium vapour pressure and the actual partial pressure of zinc vapour. However, at higher zinc concentrations, the heat transfer through the growing zinc crystal crust towards the cold steel lid may become the limiting factor because the crystallization front may reach the melting point. The computational model can be very useful in optimising the process within its safe limits.

10:25 AM Break

10:45 AM

The Application of CFD to the Design of Electric Furnaces: Lowy Gunnewiek[1]; *Lanre Oshinowo*[1]; Tom Plikas[1]; Ross Haywood[2]; [1]Hatch Associates Ltd., 2800 Speakman Dr., Mississauga, Ontario L5K 2R7 Canada; [2]Hatch, 152 Wharf St., Brisbane, Queensland 4000 Australia

Electric furnace smelting is one of the principal unit operations for ferroalloy production, and increased process intensity, improved availability, minimal maintenance and a longer campaign life are common objectives for electric furnace operation in order to obtain favourable economics of production. Furnace designers have continued to develop innovative solutions that have allowed these objectives to be realised, e.g., furnace cooling systems, and numerical modelling plays a key role in the design process. Enhanced understanding of the energy transfer process in the furnace and the ability to develop, evaluate and optimise components of the crucible design to match process requirements are the key motivators for modelling. This paper provides an overview of recent applications of numerical modelling to the design of electric furnaces. Several examples are presented, including furnace bath modelling, crucible cooling system design, taphole design, baking of Soderberg electrodes, off-gas systems, and fume control and building ventilation.

11:10 AM

Simulation of Combustion and Metal Heating in a Mobile Heat Treatment Furnace: *Yongxiang Yang*[1]; Reinier A. de Jong[1]; Markus A. Reuter[1]; [1]Delft University of Technology, Applied Earth Scis., Mijnbouwstraat 120, Delft 2628 RX The Netherlands

In this paper, a mobile heat treatment furnace from Akkermans Gloeitechniek, a Dutch company, was simulated with Computational Fluid Dynamics (CFD). The furnace is cylindrical and has four oil

burners, and large metal products such as dredging pumps and fans can be flexibly heat treated to obtain required microstructure and mechanical properties through stress relief, annealing, hardening and tempering. The furnaces are exposed to the open air, and thus the heat loss becomes significant and varies from day to day and from season to season. Since the temperature of the metal products to be heat treated can only be measured on the surface, the temperature evolution inside the metal could not be tracked in practice. CFD simulation provides a useful tool to predict the temperature evolution within the metal pieces and within the combustion space of the furnace. Energy distribution and heat loss can be subsequently estimated. An overall energy balance through temperature measurement indicted relatively low energy efficiency. Especially during the soaking period, the high energy supplied is barely used to compensate the heat loss of the furnace to maintain a constant temperature of the metal piece. The current CFD modelling aims to give advice on how to modify the design and optimize the operation to reach higher energy efficiency and low heat loss. The CFD model consists of turbulent combustion model, radiative model in participating media, and conjugate heat transfer between the combustion space and solid metal. Temperature measurement was carried out to provide thermal boundary conditions and overall energy balance of the operation. After the model validation with temperature measurement within the combustion space and metal surfaces, furnace optimization will be conducted.

Computational Thermodynamics and Phase Transformations: Phase Equilibria and Thermodynamic Assessments

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizer: Jeffrey J. Hoyt, Sandia National Laboratories, Materials & Process Modeling, Albuquerque, NM 87122 USA

Wednesday AM Room: 202A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

8:30 AM
On the Ab Initio and CALPAHD Approaches to Lattice Stability: *Zi-Kui Liu*[1]; Yi Wang[1]; Lonq-Qing Chen[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., Univ. Park, PA 16802 USA

The bct, bcc, and fcc structures of a pure element can be related through a continuous crystal axis expansion and contraction of the same crystal lattice. In the present work, ab initio calculations are carried out for various pure elements and binary systems with either fcc or bcc as their stable structures. The total energies are plotted as a function of the c/a ratio. It is observed that if the total energy of the fcc structure for a pure element is a minimum, then that of the bcc structure is a maximum, and vice versa. The relative magnitude of the total energies of the fcc and bcc structures in binary systems changes with composition. These results are discussed in connection with the lattice stability used in the CALPHAD community.

8:50 AM
Thermodynamic Modelling of Multicomponent Systems - Design of Critical Experiments: *Karin Frisk*[1]; [1]Swedish Institute for Metals Research, Drottning Kristinas väg 48, SE_11428 Stockholm Sweden

Thermodynamic modelling is a powerful tool in alloy development, and the CALPHAD (CALculation of PHAse Diagrams) method is today widely spread. The method involves coupling of the phase diagram and thermochemistry of all the possible stable or metastable phases in a system. The CALPHAD method has proved to be successful when applied to complex materials with many components and phases, where limited experimental information is available and experimental determinations are time consuming. However, when extending to composition or temperature ranges that have not previously been studied, validation by experiments is needed. A few examples of experimental validation of thermodynamic databases for multicomponent alloys performed for steel and cemented carbides are given. The choice of experiments and the interpretation of the results are discussed. The discrepancies between calculations and experiments that were found could be explained by inconsistencies in binary and ternary systems due to lack of experimental information, or a need for better descriptions of metastable phases. It was found that the accuracy of the calculations was improved by performing new analysis of metastable phases. To increase the ranges where reliable extrapolations can be made, critical experiments were designed to determine interactions in ternary systems. The methods to select critical experiments, and the resulting improvement of the thermodynamic description is discussed.

9:10 AM
Thermodynamic Assessment of the Ag-Cu-Ti System: *Raymundo Arroyave*[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 4-047, Cambridge, MA 02139 USA

A thermodynamic description for the Ag-Ti binary system was developed using all the existing experimental phase diagram data available and the resulting parameters were discussed in light of very recent thermochemical measurements of Ag-Ti melts. Using the description of the Ag-Ti binary and with the accepted descriptions for the Ag-Cu and Cu-Ti sub-systems, the Ag-Cu-Ti ternary system is critically assessed and a thermodynamic model is developed so the available experimental data are represented as accurately as possible. The phases CuTi2 and AgTi2 are modeled using a single two-sublattice model, (Ag,Cu)1(Ti)2, assuming that these phases form a continuous solid solution. The CuTi and AgTi phases are also modeled using a single sublattice model, (Ag,Cu,Ti)1(Ag,Cu,Ti)1, and their penetration into the ternary compositional triangle is correctly predicted. Ternary interactions for the liquid phase were incorporated. Experimental and calculated results are compared and the reliability of the model is confirmed.

9:30 AM
Phase Equilibria and Thermodynamic Modeling of Al-Based Metallic Glasses: *Michael C. Gao*[1]; Necip Unlu[1]; Gary J. Shiflet[1]; [1]University of Virginia, Matls. Sci. & Engrg., 116 Engineer's Way, PO Box 400745, Charlottesville, VA 22904-4745 USA

Thermodynamic and phase equilibria studies were performed for two aluminum-based glass-forming systems, namely, Al-Co-Ce and Al-Ni-Nd over a compositional range of 60-100 at% Al. First the glass formation range was determined using melt-spinning techniques and its crystallization behavior was studied using XRD, DSC and TEM. Then a number of alloys in each system were chosen for solid-state phase equilibria study using XRD, SEM and TEM, while DTA was used to characterize the solidus and liquidus temperatures. Using these results combined with other published data, these systems were thermodynamically optimized in their Al-rich corners using the CALPHAD approach. The relationship among thermodynamics, glass forming ability and crystallization behavior will be discussed.

9:50 AM Break

10:00 AM
The Calculated Energetics of Ni-Mo and Ni-Ta Alloy: *Yi Wang*[1]; Shihuai Zhou[1]; Zikui Liu[1]; Longqing Chen[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 106 Steidle Bldg., State College, PA 16802-5006 USA

The energetics of the binary compounds, Ni8Mo, Ni4Mo, Ni3Mo, Ni2Mo, delta-Ni24[Ni4xMo4(5-x)]Mo12 (x = 0 - 5), Ni8Ta, Ni3Ta, Ni2Ta, Ni6Ta7, and NiTa2, are studied using the first-principles calculations. It is shown that delta-Ni24(Ni4Mo16)Mo12, has the lowest energy of formation among the range of x examined. The calculated formation energies demonstrates that the delta-NiMo phase is metastable at 0 K and that Ni8Mo and Ni2Mo are stable at 0 K whereas the experimental high temperature phase diagram shows no Ni8Mo and Ni2Mo. For the Ni-Ta system, our results show that Ni8Ta, Ni2Ta, and NiTa2 are stable at 0 K while Ni6Ta7 is not. Experimentally, there are three types of Ni3Ta, i.e. Ni3Ta(2)S, Ni3Ta(3)S, and Ni3Ta(12)S, existed with Ni3Ta(12)S being the most stable one at temperature above 1128 K. However, our calculation shows that Ni3Ta(3)S is the most stable one at 0 K with Ni3Ta(2)S is the next and then Ni3Ta(12)S.

10:20 AM
First-Principles Calculations and Modeling on Lattice Parameters of the Ni-Al-Mo-Ta System: *Tao Wang*[1]; Yi Wang[1]; Long-Qing Chen[1]; Zi-Kui Liu[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16802 USA

The lattice parameter is an important material property. In the present work, the temperature and composition dependences of lattice parameters of the gamma and gamma prime phases in the Ni-Al-Mo-Ta system are modeled phenomenologically using the CALPHAD approach. In this approach, phases are described by sublattice models. To model the composition dependence, the lattice parameters of all end-

members in the sublattice models are needed, and some of these end-members are not stable. First-principles calculations are thus carried out to compute the lattice parameters of those end-members using the full-potential linearized augmented plane wave (FP-LAPW) method implemented in the WIEN2k package. Lattice parameters in the Ni-Al-Mo-Ta are thus calculated and compared with available experimental data.

10:40 AM
Investigation of Enthalpy of Formation for All the Possible Binary Laves Phases of Group IIA: *Yu Zhong*[1]; Jorge O. Sofo[2]; Zi-Kui Liu[3]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 107 Steidle Bldg., Univ. Park, PA 16802 USA; [2]Pennsylvania State University, Matls. Simulation Ctr., Univ. Park, PA 16802 USA; [3]Pennsylvania State University, Matls. Sci. & Engrg., 209 Steidle Bldg., Univ. Park, PA 16802 USA

First-principles calculations are performed for all combinations of elements in Group IIA (Be, Mg, Ca, Sr, Ba, Ra) for the possible binary A2B laves phases in C14, C15 and C36 structures. According to the work of Zhu et al.,[1] the atomic ratio of the two elements is the most important factor in determining the stability of the laves phases. A lave phase can be considered to have two sublattices with preferable occupancies of individual atoms. In the current thermodynamic modeling, they are modeled with a two-sublattice model, i.e. (A,B)2(A,B). This model has 4 end-members: A2B, A2A, B2B, B2A. Their enthalpies of formation are calculated to examine its relationship with the stability and solubility range of laves phases. [1]J. H. Zhu, C. T. Liu, L. M. Pike and P. K. Liaw,"Enthalpies of formation of binary Laves phases" Intermetallics, 10 (2002) 579-595.

11:00 AM
Evaluation of the Thermodynamic Properties of the Ni-Mo-Ta System Incorporating First-Principles Calculations: *Shihuai Zhou*[1]; Yi Wang[1]; Tao Wang[1]; JingZhi Zhu[1]; Rebecca A. MacKay[2]; Long-Qing Chen[1]; Zi-Kui Liu[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., 107 Steidle Bldg., State College, PA 16802 USA; [2]NASA Glenn Research Center, Matls. Div., 21000 Brookpark Rd., Cleveland, OH 44135 USA

The phase equilibria and thermodynamic properties of the ternary Ni-Mo-Ta system were analyzed. The enthalpies of formation of the stable or metastable d-NiMo, Ni2Mo, Ni3Mo, Ni4Mo, Ni8Mo, NiTa2, NiTa, Ni2Ta, Ni3Ta and Ni8Ta phases were calculated by the first-principles method using Vienna Ab-initio Simulation Package (VASP) at 0 K. The results indicate that the compounds, Ni2Mo, Ni3Mo with the Cu3Ti structure, Ni4Mo, Ni8Mo, NiTa2, Ni2Ta, Ni3Ta with the Al3Ti structure and Ni8Ta are stable phases at 0K, while the compounds, d-NiMo, Ni3Mo with the Al3Ti structure, NiTa, Ni3Ta with the Cu3Ti and Pt3Ta structures are metastable phase at 0K. In the thermodynamic description, the stable Ni3Mo and metastable Ni3Ta phases with the Cu3Ti structure were treated as same phase, while the metastable Ni3Mo and stable Ni3Ta phases with the Al3Ti structure were described as same phase. The non-stoichiometric stable Ni3Ta phase at high temperature with the Pt3Ta structure and Ni3Mo phase with the Cu3Ti structure were described with a two sublattice model. With the first-principles and experimental phase equilibrium data, the Gibbs energy function of individual phase was determined. The calculated results of the ternary Ni-Mo-Ta system were compared with the first-principles data and the experimental data in the literature.

11:20 AM
First-Principles Study of the Order-Disorder Transition of the Al4Cu9(Gamma) Phase: *Chao Jiang*[1]; Long-Qing Chen[1]; Zi-Kui Liu[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., Univ. Park, PA 16802 USA

Based on recent experimental observations, the gamma phase in Al-Cu system undergoes a D83->D82 order-disorder transition, which is of second-order type instead of the previously believed first-order. In the present work, this transition was investigated using the first-principles/CALPHAD hybrid computational approach. The gamma phase was modeled using a (Al,Cu)3(Al,Cu)3(Al,Cu)1(Al,Cu)1(Cu)5 five-sublattice model. The formation enthalpies of all the end-members were obtained from first-principles total energy calculations. All structures are fully relaxed with respect to cell-internal and -external degrees of freedom. The entropies of formation of the end-members and the interaction parameters within each sublattice were adjusted to fit the order-disorder transition temperature and the phase diagram. The model calculations are compared with experimental observations.

Cost-Affordable Titanium Symposium Dedicated to Prof. Harvey Flower: Creative Fabrication

Sponsored by: Structural Materials Division, SMD-Titanium Committee
Program Organizers: M. Ashraf Imam, Naval Research Laboratory, Washington, DC 20375-5000 USA; Derek J. Fray, University of Cambridge, Department of Materials Science and Metallurgy, Cambridge CB2 3Q2 UK; F. H. (Sam) Froes, University of Idaho, Institute of Materials and Advanced Processes, Moscow, ID 83844-3026 USA

Wednesday AM Room: 206B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Vladimir Moxson, ADMA Products Inc., Twinsburg, OH 44087 USA

8:30 AM
Commercialization of the Armstrong Process for Producing Titanium Alloy Powder: *Richard P. Anderson*[1]; William Ernst[1]; Lance Jacobsen[1]; Dariusz Kogut[1]; [1]ITP, 20634 W. Gaskin Dr., Lockport, IL 60441 USA

The Armstrong Process produces high purity titanium and titanium alloy powders by injecting a jet of vaporized TiCl4 (or mixed metal chlorides for alloys) into a stream of flowing sodium. The powder characteristics (size distribution, morphology, flow and packing characteristics) may be tailored for various applications by varying the system operating parameters. Development of the Armstrong process has gone through the traditional pre-commercialization stages. A pilot plant became operational in early '03. The small size and benign operating conditions (low temperature and pressure) in the reactor, combined with the continuous nature of the overall process, allows for production of high purity powder at a fraction of the current production cost of titanium powder and ingot. The overall economics of the process will be discussed and compared with other production technologies. Powder samples and parts made from the powder will be available for inspection.

9:00 AM
The Utilization of Recycled Titanium Ores and Innovative Plasma Arc Technology for the Production of Low Cost Titanium Metal Powders: *Lowell V. Sieck*[1]; Mark S. Shuey[1]; Daniel I. Kaplan[1]; [1]Industrial Technologies of New York, LLC, Yonges Island, SC 29449 USA

Titanium metal alloys remain the metals of choice for advance materials use in the aerospace, automotive, and medical implant industries. As is widely recognized, the use of titanium metal alloys in these industries is presently limited by the availability of low cost titanium metal alloy powders and available fabrication processes. While recent advances in fabrication processes, including use of titanium powder alloys for improved near-net shapes and laser and electron beam methods for joining, have improved opportunities for use of titanium alloys; the availability of low cost, high quality, titanium metal alloy powders has not significantly improved. However, recent advances that have been made in a combination of technologies involving: 1) recovering titanium ore from industrial waste streams, 2) smelting low cost, recycled, titanium ore directly into titanium metal stock using innovative plasma arc technology, and 3) an innovative design for gas atomization of titanium metal stock may result in significant reductions in the cost of high quality titanium powders. These innovations could allow the development and marketing of low cost titanium metal powders that can augment the use of titanium powders in many existing industries as well as contribute to the development of new commercial applications for titanium metals.

9:30 AM
Powder Metallurgy Ti-6Al-4V Components Produced from Low Cost Blended Elemental Powders by Hot Pressing: *V. A. Duz*[1]; V. S. Moxson[1]; F. H. (Sam) Froes[2]; F. Sun[2]; J. S. Montgomery[3]; [1]ADMA Products, Inc., 8180 Boyle Pkwy., Twinsburg, OH 44087 USA; [2]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), McClure Hall, Rm. 437, Moscow, ID 83844-3026 USA; [3]Army Research Laboratory, Aberdeen Proving Ground, MA 21005-5066 USA

Cost effective method for manufacturing Ti-6Al-4V chunky components from Blended Elemental (BE) powder using a hot pressing consolidation technique will be discussed. Large size preforms with various sintered densities have been used for hot consolidation to near

net densities (over 99% of theoretical). Various microstructures developed in sintering and high temperature deformation will be presented.

10:00 AM
Effect of Oxygen Content on Properties of Cast Ti-6Al-4V Alloy: Mustafa Guclu[1]; Ibrahim Ucok[1]; Joseph R. Pickens[1]; [1]Concurrent Technologies Corporation, 100 CTC Dr., Johnstown, PA 15904 USA

An objective of the Combat Vehicle Research (CVR) Program at Concurrent Technologies Corporation is to effect greater use of low-cost titanium alloys such as Ti-6-4 in combat vehicle and other defense applications. It is clear from various defense programs that in the near term, use of high-oxygen (up to 0.25 wt%) titanium alloys such as those made by single-melt processing, is leading the way in reducing costs and expanding the use of titanium alloys in defense applications. Ti-6-4 components, including the gun pod and elevation arms for the Mobile Gun System, were fabricated with different oxygen levels, by investment casting and the rammed graphite casting processes. The effect of oxygen content on mechanical properties was examined. Tensile properties were determined, as required by typical casting specifications, and found to exceed the minimum requirements for all oxygen levels investigated. Extensive mechanical testing and evaluation will be needed to further expand application areas of low-cost titanium castings.

10:30 AM
Cost Effective Powder Metallurgy Approach to Produce Titanium Alloy Plates: *V. S. Moxson*[1]; V. A. Duz[1]; J. S. Montgomery[2]; F. H. (Sam) Froes[3]; [1]ADMA Products, Inc., 8180 Boyle Pkwy., Twinsburg, OH 44087 USA; [2]Army Research Laboratory, Aberdeen Proving Ground, MA 21005-5066 USA; [3]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), McClure Hall, Rm. 437, Moscow, ID 83844-3026 USA

Cost effective method for manufacturing Ti-6Al-4V plates using a Blended Elemental (BE) powder metallurgy (P/M) approach will be discussed. 48" wide plates with various thickness produced by this method has been metallographic evaluated at various stages of production. Detailed studies of the microstructures developed in sintering and high temperature rolling will be presented.

11:00 AM
Titanium Eddy Current Measurement: *John Paul Wallace*[1]; Robert M. Siegfried[2]; Jerome Dunn[3]; [1]Casting Analysis Corp., 8379 Ursa Ln., Weyers Cave, VA 24486 USA; [2]Adelphi University, Dept. Math. & Comp. Sci., South Ave., Garden City, NY 11530 USA; [3]Casting Analysis Corp, 48 Montague Rd., Sunderland, MA 01375 USA

In process eddy current measurements for oxygen variation, weld detection and defect detection are compromised by two physical properties of alpha-titanium. These are the hexagonal crystal symmetry which complicates the conductivity measurement and the BCC-HCP phase transition alters texture and microstructure of the metal upon cooling from solidification. Multifrequency application of inverse analysis using probes allows the removal of geometric effects of shape and liftoff. What remains is the analysis of the normal sources of conductivity variations such as impurity alloying, cold work, recovery and recrystallization structures. Modifying sensors to detect the symmetry contributions of a textured structure and extending the inverse analysis to probe depth variations of the local electrical conductivity tensor allows a separation of texture verses alloying contributions to the conductivity. In the simplest measurement application where one wants to separate oxygen alloying effects by measuring the bulk electrical conductivity, a local understanding of the microstructure is required in order to separate the contributions of the oxygen from the microstructure. Base line data will be presented from high purity single crystals through cold worked structures showing the range of responses of the key effects influencing conductivity variation. Then the procedure of automating this analysis in real time will presented for application to in process monitoring and inspection.

11:30 AM
Titanium in the Family Automobile: The Cost Challenge: *F. H. (Sam) Froes*[1]; H. Friedrich[2]; J. Kiese[3]; D. Bergoint[4]; [1]University of Idaho, Inst. for Matls. & Advd. Processes, McClure Bldg., Rm. 437, Moscow, ID 83844-3026 USA; [2]Volkswagen AG, Letter Box 1777, Wolfsburg D-38436 Germany; [3]Volkswagen AG, Wolfsburg D-38436 Germany; [4]Bergoint Engineering GmbH, Akazienweg 11, Alsbach 64665 Germany

With advances in extraction/fabrication techniques and ever increasing gasoline prices the advantage of using lightweight materials such as titanium in automobiles continues to increase. The major drawback - high cost - is omnipresent. However innovative extraction and fabrication approaches are leading to a deceased cost. The present status and future potential for titanium use in the family automobile will be presented.

Dislocations: Plasticity, Voids, and Fracture

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Elizabeth A. Holm, Sandia National Laboratories, Albuquerque, NM 87185-1411 USA; Richard A. LeSar, Los Alamos National Laboratory, Theoretical Division, Los Alamos, NM 87545 USA; Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Wednesday AM Room: 201A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

8:30 AM Invited
A Multi-Phase Field Theory of Dislocation Dynamics: M. P. Ariza[1]; A. M. Cuitino[2]; M. Koslowski[3]; *M. Ortiz*[1]; [1]California Institute of Technology, Div. of Engrg. & Applied Sci., Pasadena, CA 91125 USA; [2]Rutgers University, Mech. & Aeros. Engrg. Bldg., Rm. D158, Piscataway, NJ 08854 USA; [3]Los Alamos National Laboratory, Theoretical Div., MS B268, Los Alamos, NM 87545 USA

A multi-phase field theory of dislocation dynamics and strain Hardening in ductile single crystals is developed. The theory accounts for: an arbitrary number and arrangement of dislocation lines over multiple slip systems; the long-range elastic interactions between dislocation lines; the core structure of the dislocations; the interaction between the dislocations and an applied stress field; and the (possibly irreversible) interactions with short-range obstacles and other dislocations, resulting in hardening. A chief advantage of the present theory is that it is analytically tractable with the aid of standard tools of analysis such as the discrete Fourier transform and Gamma-convergence. The multi-phase field representation enables complex geometrical and topological transitions in the dislocation ensemble, including dislocation loop nucleation, bow-out, pinching, and the formation of Orowan loops. The theory predicts a range of behaviors which are in qualitative agreement with observation, including: hardening and dislocation multiplication in single slip under monotonic loading; the Bauschinger effect under reverse loading; the fading memory effect, whereby reverse yielding gradually eliminates the influence of previous loading; the evolution of the dislocation density under cycling loading, leading to characteristic 'butterfly' curves; the formation of dislocation networks andcellular structures; and others.

9:05 AM
The Effects of Grain Size and Dislocation Source Density on the Strengthening Behavior of Polycrystals a Two Dimensional Discrete Dislocation Simulation: *S. B. Biner*[1]; J. R. Morris[1]; [1]Iowa State University, Ames Lab., Metal & Ceram. Scis., Ames, IA 50011 USA

The evolution of the flow stress for grain sizes ranging from about 11 to 0.5 μm under shear deformation was examined. The grain boundaries were assumed to be both the only sources for dislocation nucleation and also the only obstacles to the dislocation motion. The simulations were carried out for two sets of system sizes and grain morphologies. For the grain size ranges considered, an inverse relationship between the grain size and 0.2% offset flow stress in the form of Hall-Petch relationship $[d]^{-1/2}$ was observed, although there is some uncertainty in the exponent. The evolution of flow stress follows a narrow band when expressed as a function of dislocation density divided by the dislocation source density and hence suggests a scaling with the grain size as seen in our earlier study. This work was performed for the USDOE by Iowa State University under contract W-7405-Eng-82. This research was supported by the Director of Energy Research, Office of Basic Sciences.

9:25 AM
Relationships of Fracture Toughness and Dislocation Mobility in Intermetallics: *Kwai S. Chan*[1]; [1]Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA

An analytical method has been developed and utilized to compute the Peierls-Nabarro (P-N) barrier energy for relevant slip systems in several intermetallics. The P-N barrier energy and generalized fault energy are combined and used as a measure of dislocation mobility. Furthermore, a fracture model has been developed to describe the process of thermally activated dislocations moving away from the

crack tip and to predict the corresponding fracture resistance. The correlation indicates that fracture toughness increases with decreasing values of the P-N barrier energy and the generalized stacking fault energy, in accordance with the fracture model formulated based on thermally activated slip. The use of the fracture model for predicting the effects of slip behavior, temperature, and alloy additions on fracture resistance is demonstrated for selected intermetallics including NiAl, TiAl, Laves phase, and Nb-based silicides. Work supported by AFOSR through Contract No. F4962001-C-0016, Dr. Craig S. Hartley, Program Manager.

9:45 AM
Dislocation Analysis of Fatigue Crack Growth: *Kuntimaddi Sadananda*[1]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, Washington, DC 22152 USA

Fatigue crack growth behavior is analyzed using discrete dislocations. Equilibrium configuration of both crack-dislocations and crystal-lattice dislocations are determined by minimizing the total elastic energy of the system. The effect of plastic zone on the crack tip stress intensity factor is calculated using Lin-Thomson equations. Crack is then extended elastically, while the plastic zone moves behind the crack tip forming crack wake plasticity. The effect on the crack tip stress intensity factor is determined to evaluate the role of crack tip plasticity on crack growth rate. The role of internal stresses, effects of overloads and underloads are estimated.

10:05 AM
Laser Shock Compression of Copper Monocrystals: Mechanisms for Dislocation and Void Generation: *Matthew S. Schneider*[1]; Bimal Kad[1]; Fabienne Gregori[2]; Daniel H. Kalantar[3]; Bruce A. Remington[3]; Marc A. Meyers[1]; Vlado A. Lubarda[1]; [1]University of California, Matls. Sci. & Engrg., MC 0418, La Jolla, CA 92093 USA; [2]University of Paris 13 France; [3]Lawrence Livermore National Laboratory, Livermore, CA 94450 USA

Copper and copper aluminum with two orientations ([001] and [134]) were subjected to high intensity laser (energy levels of 40-300 J; energy densities of 15-70 MJ/m2 and durations below 10 ns). The defects created are characterized by transmission electron microscopy. An orientation-dependent threshold stress for twinning is observed. The results are rationalized in terms of a criterion in which slip and twinning are considered as competing mechanisms. A constitutive description is applied to the two orientations, incorporating both slip and twinning. The predictions are in agreement with experiments. The threshold stress for twinning in the [001] orientation is 20-40 GPa, whereas the one for the [134] orientation is 40-60 GPa. The threshold stress is calculated, considering the effect of shock heating. The constitutive description provides a rationale for the experimental results; the calculated thresholds are 18 GPa for [001] and 25 GPa for [134]. A mechanism for void generation and growth based on the emission of geometrically necessary dislocations is proposed and analytically formulated.

Electrochemical Measurements and Processing of Materials: Electrochemical Metal Production

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Aqueous Processing Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Uday B. Pal, Boston University, Department of Manufacturing Engineering, Brookline, MA 02446 USA; Akram M. Alfantazi, University of British Columbia, Department of Metel & Materials Engineering, Vancouver, BC V6T 1Z4 Canada; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Wednesday AM Room: 212A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Adam C. Powell, Massachusetts Institute of Technology, Matls. Sci. & Engrg., Cambridge, MA 02139-4307 USA; James W. Evans, University of California, Matls. Sci. & Mineral Engrg., Berkeley, CA 94720 USA

8:30 AM Invited
Zirconia-Based Inert Anodes for Green Synthesis of Metals and Alloys: *Uday B. Pal*[1]; Ajay Krishnan[1]; Christopher Manning[1]; Sourav Sen[1]; [1]Boston University, Mfg. Engrg., 15 St. Mary's St., Boston, MA 02215 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. A cathode is placed in the solvent and an appropriate electric potential is applied between the electrodes to synthesize the metals from their oxides. The full-benefit of the process can be realized if it is conducted at temperatures between 1100-1400°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. This paper reports the recent progress of a continuing laboratory-scale investigation involving different types of zirconia-based inert anodes employed at temperatures between 1100-1400°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.

9:00 AM Invited
Towards Carbon-Free Metals Production by Molten Oxide Electrolysis: *Donald R. Sadoway*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 8-203, Cambridge, MA 02139-4307 USA

Molten oxide electrolysis (MOE) is an extreme form of molten salt electrolysis, a technology that has been producing tonnage metal for over 100 years: aluminum, magnesium, lithium, sodium, and the rare-earth metals are all produced in this manner. MOE is distinguished by the avoidance of halide electrolytes and carbon anodes which enables the production of oxygen gas instead of halogens or CO_2. Accurate knowledge of the electrical properties of candidate electrolytes is needed for the design of industrial electrolytic cells because joule heating establishes the thermal balance. In the absence of a fully satisfactory technique for making high-accuracy measurements, the coaxial cylinders technique was invented. The variation of electrical conductivity and transference number with temperature and composition has been measured for melts in the FeO - MgO - CaO - SiO_2 system. Electrolysis at 1450ºC produced iron at the cathode and oxygen gas at the anode.

9:30 AM Invited
Production of Titanium Powder Through an Electronically Mediated Reaction: *Toru H. Okabe*[1]; Takahito Kakihira[2]; Takashi Abiko[2]; [1]University of Tokyo, Inst. of Industrial Sci., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8505 Japan; [2]University of Tokyo, Grad. Sch. of Engrg., 4-6-1 Komaba, Meguro-ku, Tokyo 153-8505 Japan

In the recent years, a new titanium reduction process directly from titanium oxide (TiO2) by the electrochemical reduction of TiO2 in molten calcium chloride (CaCl2) was investigated as an alternative process of the Kroll process [Chen et al.: Nature, 407 (2000) 361], and there is a large variety of titanium reduction processes currently under investigation [Ono & Suzuki: Journal of Metals, 54 Feb., (2002) 59]. In this study we explored the production of titanium powder by an electronically mediated reaction (EMR) [Okabe & Sadoway: J. Materials Research, 13 (1998) 3372]. Feed material, TiO2, and reductant calcium alloy containing nickel, etc. were charged into electronically isolated locations in a CaCl2 molten salt at 1173 K. The flow of current through an external path between the feed and reductant locations, and electrochemical potentials of the feed electrode were monitored during the reduction experiment [Okabe et al.: J. Alloys and Compounds, 288(1999) 200]. After the experiment, pure titanium powder with low nickel content was obtained although liquid Ca-Ni alloy was used as the reductant. This clearly demonstrates that titanium metal powder can be produced by electrochemical reactions, without direct physical contact between the feed (TiO2) and reductant (calcium alloy). In some experiments, pure titanium in sponge form with 99.5 mass% purity (3500mass ppmO) was obtained. The method has the potential for preventing accumulation of impurities into titanium deposits because impurities in the molten salt can be trapped by the reductant alloy placed in the different location from the titanium reduction site. Energy efficiency of the reduction process can also be improved when combined with conventional molten salt electrolysis (MSE) of CaCl2 for reductant production. For producing calcium alloy reductant, MSE of CaCl2-CaO molten salt is investigated. Difference and features of various reduction processes are discussed.

10:00 AM Break

10:30 AM
A Phase-Field Model of the Cathode Interface in Transport-Limited Metal Reduction and Refining Processes: *Wanida Pongsaksawad*[1]; Adam C. Powell[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 4-043, Cambridge, MA 02139 USA

In the transport-limited electrochemical reactions coupled with fluid flow such as metal reduction and refining processes, the cathode exhibits a Mullins-Sekerka instability, resulting in the growth of dendrite of solid metal into the electrolyte. To simulate the cathode interface dynamics, a two-dimension model is developed using the Cahn-Hillard phase field method. In this work, the system of interest includes a solid cathode in molten salt electrolyte. A detailed multi-component two-phase flux-metal model is presented, which describes vorticity flow in the electrolyte and multicomponent diffusion driven by the chemical and electrical potentials at the diffuse interface. As a result of these simulations, the effect of shear flow rate on the growth of dendrites under the electrochemical reactions can be studied.

11:00 AM
Reaction Mechanism of Electrochemical Reduction of Titanium Dioxide: *Wang Shulan*[1]; Li Yingjun[1]; [1]Northeastern University, Dept. of Chmst., No. 3-11, Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China

The electrochemical reduction mechanism of TiO2 in molten CaCl2 was studied systematically by using cyclic voltammetry, chronoamperometry and A.C impedance techniques in temperature range from 1073K to 1133K. The reaction mechanism of electrochemical reduction of TiO2 was found to be the two-step reactions: TiO2 is reduced to the intermediate production TiO, and then reduced to pure Ti.

Failure of Structural Materials: Fundamentals

Sponsored by: Structural Materials Division, SMD-Structural Materials Committee
Program Organizers: Michael E. Stevenson, Metals and Materials Engineering, Suwanee, GA 30024 USA; Mark L. Weaver, University of Alabama, Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202 USA

Wednesday AM Room: 211A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Mark L. Weaver, University of Alabama, Metallurgl. & Matls. Engrg., Tuscaloosa, AL 35487-0202 USA

8:30 AM Keynote
Failure of Structural Materials: *Michael E. Stevenson*[1]; [1]Metals & Materials Engineers, 1039 Industrial Ct., Suwanee, GA 30093 USA

Failures of engineered structures and components pose substantial threats in terms of economic cost, and often, public safety. The analysis of such failures, therefore, takes on an important role in safegaurding against said losses. This paper will discuss the analysis of structural failures from the perspective of metallurgical/mechanical analysts. Topics such as investigation methodology, emerging analytical tools, and computational modeling will be discussed.

9:00 AM
Fracture Mechanics Applications in Aircraft Structural Failure Analysis: *Jeffrey L. McDougall*[1]; [1]Metals & Materials Engineers, 1039 Industrial Court, Suwanee, GA 30024 USA

Cracked aluminum alloy structural components observed during routine post-flight inspection of corporate-type aircraft were subjected to a full-scale failure investigation. The failures were fatigue related. Inadequate mechanical properties were also involved. Through the use of fatigue striation spacing and fracture mechanics, it was able to be determined if operative stress levels in combination with other factors such as residual stresses and recent material changes contributed to the failures.

9:20 AM Invited
Material Degradation and Structural Collapse from the Fire Investigator's Perspective: *Eric Stauffer*[1]; [1]ATS, Fire Investigation Unit, 1190 Atlanta Industrial Dr., Marietta, GA 30066 USA

The purpose of fire investigation is to determine the origin and the cause of a fire. The first step in doing so is recognizing, observing, and analyzing burn patterns. The second step is identifying and evaluating fuel availability, ignition source, and the interaction between them. While the examination of artifacts for failure analysis is beyond the competence of the fire investigator and is not pertinent in most fire scenes, observations of material degradations and structural collapses are essential to proper investigation. The goal of this presentation is to introduce the patterns and characteristics of a fire scene that interest fire investigators and to interpret these from a fire investigator's perspective. These include burn patterns, depth of char, melted metals, such as steel, copper, and aluminum, and displacement of walls and floors, etc. Scientifically incorrect concepts such as glass crazing and concrete spalling will be presented and demystified.

9:40 AM
Characterization of Ship Hull Steel Plates After Explosive Loading and Conventional Mechanical Testing: Christian Klinger[1]; Joachim Kinder[1]; Dietmar Klingbeil[1]; Werner Österle[1]; *Pedro Dolabella Portella*[1]; [1]Federal Institute for Materials Research and Testing (BAM), Matls. Engrg., Unter den Eichen 87, Berlin D-12205 Germany

The capsizing of the MV Estonia in the Baltic Sea in September 1994 lead to the loss of 852 lives. The ship wreck lies on the ground at a depth of about 70 meters in an international area. An intergovernmental commission concluded that the accident was caused by the loss of the bow visor due to the failure of its hinges and locks. However, independent groups sustain that the loss of the bow visor was primarily due to explosions on board of the Estonia. Some of these groups organized an expedition in August 2000, allowing divers to cut two pieces out of the forward bulk head of the Estonia. These pieces were located at the border of holes in the hull, whose aspect could be compatible with an explosion. In order to confirm the hypothesis of an explosion, steel plates of the same quality used in the building of the ship were submitted to different mechanical tests in a very broad interval of strain rates. Further specimens of the steel plates were submitted to explosions using different types of explosives as employed either for civil or military applications. The microstructure of the steel plates deformed either in mechanical tests or in explosions was characterized using light and electron microscopy. The only microstructural feature which could be unambiguously traced back to an explosive loading was the presence of deformation twins across the whole thickness of the plates in the vicinity of the fracture surface. Since deformation twins are not present in the specimens recovered from the Estonia, it was concluded that no explosion occurred in the vicinity of these specimens.

10:00 AM
Microstructural Characteristics of Shear Localization in a Cold Rolled 316L Stainless Steel: *Qing Xue*[1]; George T. Gray[1]; Shuh Rong Chen[1]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech., MST-8, G755, Los Alamos, NM 87545 USA

The microstructural characteristics of adiabatic shear localization in a cold-rolled 316L stainless steel (316L SS) was studied. A forced shear technique on a split-Hopkinson pressure bar was utilized to generate localized deformation on "hat-shaped" specimens. The prestraining effect of the cold rolling was examined by using transmission electron microscopy. Comparison of the initial microstructure between the as-received and the cold-rolled 316L SS characterized the defect effects on the formation of shear localization. The as-received steel was seen to possess a low density of defects while the cold rolled steel included a high density of slip bands, microbands and deformation twins. The existence of profuse defects and their substructure in the cold-rolled steel led to a higher susceptibility to shear localization. The microstructure near and inside the shear bands was carefully investigated. Fine subgrains with an average size of 100 nm were found inside shear bands.

10:20 AM Break

10:35 AM
Microstructural Influences on the Failure of Large Gray Iron Castings: *Jeffrey L. McDougall*[1]; [1]Metals & Materials Engineers, 1039 Industrial Ct., Suwanee, GA 30024 USA

Hydrostatic pressure testing of two large gray iron castings resulted in brittle overload failures. Thorough failure investigations revealed casting defects associated with both origins. Both incidences were related to the mold. The first incident involved a defect that resulted in a stress concentration that reduced the normally large critical flaw size to the microstructural level. Material selection was also a concern of the manufacturer. Through the use of fracture mechanics, the viability of faribcating from gray iron was also determined. A microstructural gradient was observed at the fracture origin of another casting. Inadequate mold desgin resulted in rapid cooling at a thin-walled region of the casting. The development of the microstructural gradient was investigated.

10:55 AM
Failure Analysis of a Steel Gear: *Itamar Ferreira*[1]; Ângela Cristina Mazzei[1]; Ruís Camargo Tokimatsu[2]; [1]UNICAMP, Fac. of Mech. Engrg., Matls. Engrg., PB 6122, Campinas, São Paulo 13083-970 Brazil; [2]UNESP, Fac. of Engrg., Mech. Engrg., Av. Brasil Centro, 56, Ilha Solteira, São Paulo 15385-000 Brazil

The hydrogen embrittlement is a very common problem. In spite of a large number of researches that has been made, some steel components still failure by this phenomenon. The gear steels are usually very susceptible to hydrogen embrittlement. The hydrogen contamination of the steel can occur in the initial steps of the steel fabrication process or in the heat treatment or carburizing process of the gear. The objective of this paper is the identification of the cause of a cylindrical gear failure (875 mm of external diameter and 241 mm of width) made of a forged 17CrNiMo6 steel, that was designed for using in an Alcohol Plant, in São Paulo state, Brazil. The gear failure was observed during the start up of the gearbox. The gearbox assemblage was finished in March 2002 and the failure of the gear was detected in April 2002, six weeks after assembling. The gear was assembled on the shaft by interference fit. Chemical analysis, metallography, hardness and fracture toughness tests have been conducted. A visual inspection indicated that the gear fractured from the keyway. It was observed that the chemical composition is in accordance with the steel specification. The microstructures at the core (bainite) and at the carburizing case (tempered martensite) are also as expected from the gear fabrication process. The Rockwell Hardness results, 30 to 35 HRC at the core and near the keyway, 40 to 42 HRC some millimeters from the carburizing case, and 52 HRC at the carburizing case, are also as expected. The median value of fracture toughness is 71.2 MPm1/2, in terms of KQ, and 0.045 mm, in terms of CTODc, obtained from three-point bend tests, by using specimens of 10 mm thick machined from the steel near the keyway, indicate that the gear steel is not embrittled. Quasi-cleavage has been identified as the predominant micromechanism of fracture on the fracture surface of the gear, intergranular fracture in same regions on the carburizing case, and micro voids coalescence near the carburizing case and a decreasing quantity of micro voids when going to the core of the gear. Considering that (a) the gear fracture was of the delayed type, in presence of normal stress due to the assembling the gear on the shaft by using interference fit; (b) the chemical composition, the microstructure, and hardness of the steel are in accordance with the specifications and as expected from the fabrication process; (c) the fracture toughness levels of the steel near the keyway are reasonable; (d) the predominant fracture micromechanism is quasi-cleavage; (e) the stress levels from the inference fit are as expected; it is possible to conclude that the failure of the gear was due to hydrogen embrittlement.

11:15 AM
Study on the Cold Formability of Drawn Non-Heat Treated Steels: *K. S. Park*[1]; D. L. Lee[2]; C. S. Lee[1]; [1]Postech, Matls. Sci. & Engrg., San 31, Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 Korea; [2]POSCO, Tech. Rsch. Labs., 1, Koedong-dong, Nam-gu, Pohang, Kyungbuk 790-600 Korea

Non-heat treated steels are attractive in the steel-wire industry since the spheroidization and quenching-tempering treatment are not involved during the processing. Therefore, it is important to investigate optimum materials showing a good combination of strength and formability after the drawing process. In this study, three different steels such as dual phase steels, low-Si steel, and ultra low carbon bainitic steel were used to study their mechanical properties and the cold formability. The cold formability was investigated by estimating the deformation resistance and the forming limit. The deformation resistance was estimated by calculating the deformation energy and the forming limit was evaluated by measuring the critical strain revealing crack initiation at the notch tip of the specimens. The results showed that deformation resistance was the lowest in the low-Si steel, and the forming limit strains of ultra low carbon bainitic steel, low-Si were higher than that of commercial SWECH45F steel.

General Abstracts: Session VI

Sponsored by: TMS
Program Organizers: Adrian C. Deneys, Praxair, Inc., Tarrytown, NY 10591-6717 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Wednesday AM — Room: 208B
March 17, 2004 — Location: Charlotte Convention Center

Session Chair: Thomas Battle, DuPont Titanium Technologies, Wilmington, DE 19880 USA

8:30 AM
The Effects of Hysteresis on Solder Wetting: *F. Michael Hosking*[1]; Frederick G. Yost[2]; Cynthia L. Schwartz[3]; [1]Sandia National Laboratories, PO Box 5800, MS 0889, Albuquerque, NM 87185-0889 USA; [2]Trapezium Technology, 2549 Elfego Rd. NW, Albuquerque, NM 87107 USA; [3]MO-SCI Corporation, 4000 Enterprise Dr., Rolla, MO 65401 USA

The phenomenon of solder hysteresis is investigated. During typical solder wetting, the advancing and receding contact angle and associated wetting force, which corresponds to the solder meniscus weight, can exhibit a wide range of measured values. Wilhelmy plate wetting experiments were conducted to determine the effects of temperature, cycling rate, dwell time and immersion depth on hysteresis. The test materials were 60Sn-40Pb (wt. %) solder and oxygen free high conductivity (OFHC) copper. Solder hysteresis was confirmed with no evidence of a constant equilibrium wetting force at the advancing and receding steady-state conditions. It is concluded that wetting forces and dynamic contact angles are dependent upon dynamic test conditions rather than inherent solder and substrate properties. Hysteresis also appears to decrease with increasing test cycle, strongly suggesting that solder/substrate and substrate/flux reactions, which take place early in the wetting process, play a key role in determining the extent of hysteresis.

8:55 AM
Dissolution of Copper Oxide in Molten Fluxes: *Peng Fan*[1]; W. D. Cho[1]; [1]University of Utah, Dept. of Metallurgl. Engrg., Salt Lake City, UT 84112 USA

The dissolution rate and the solubility of solid copper oxides (CuO and Cu2O) in various molten fluxes based on CaO-Al2O3 have been studied at high temperatures. The effects of temperature and flux composition on the dissolution rate and the solubility have been determined for the two copper oxides. Based on the physical and chemical properties of the molten fluxes and the interaction between solid copper oxides and the molten fluxes, the mechanism of the dissolution is discussed.

9:20 AM
The Precipitate Selectivity of Perovskite in FCSTMA Slag: *Liaosha Li*[1]; Dong Yuanchi[1]; *Sui Zhitong*[2]; [1]Anhui University of Technology, Key Lab. of Metallurgl. Engrg. & Resources Recycling, Hudong Rd., Maanshan, Anhui Province 243002 China; [2]Northeastern University, Sch. of Matl. & Metall., HePing Div., ShenYang, Liaoning Province 110006 China

The slag studied is a deoxidization system of FeOx-CaO-SiO2-TiOy-MgO-Al2O3. The behavior of TiO2 selectively enriched in Perovskite was investigated and discussed at given conditions as following: Oxidization of molten slag could change multivalent Ti-bearing oxides into TiO2 and increase the activity of TiO2 within slag, which made TiO2 combine with CaO in slag to form Perovskite. The oxidation not only changed compositions and structures of slag phases but also promoted majority of TiO2 migrating into Perovskite. The increment of Perovskite precipitated by oxidization could go up to 30% ~ 40% of the precipitation in its original slag. Besides, raising the slag basicity could increase the activity of CaO in slag that facilitated the selective precipitation of Perovskite as well. If slag basicity was adjusted up to 1.3, about 80% amount of TiO2 in slag could be collected in Perovskite.

9:45 AM
Adsorption Study on Rejected Muds of Bayers Process Using Different Flocculants: *Benjamin Toppo*[1]; *Balakrushna Padhi*[1]; *S. P. Mahapatra*[1]; [1]National Aluminium Company Limited, Damanjodi, Orissa 763008 India

High molecular weight water soluble polymers (flocculants) are commonly added to mineral slurries formed during hydro-mineralogical processing to improve solid-liquid separation. Choice of flocculants for gravity settling devices such as thickeners has historically based on settling tests performed in the laboratory. A mechanistic information about settling of resudial muds (hematite, alumino-goethite) of bayers process for extraction of alumina by alkali digestion of bauxite has been emphasized in the present study. In addition to monitoring the adsorption of polyacrylate in situ as a function of time, ionic strength and temperature has provided information related to adsorption (surface area) and absorption study. A FTIR and XRD technique has shown to be applicable to investigating the adsorption of flocs using synthetic and natural flocculating agents which can provide the adsorption segments onto mud (hematite, alumino-goethite) and (loop & tails) of the polymer at different caustic concentration and slurry concentration in bayer's process.

10:10 AM Break

10:20 AM
Reduction of Zinc-Containing Metallurgical Residues in a Fluidized Bed Reactor: *S. M. Taghavi*[1]; M. Halali[1]; [1]Sharif University of Technology, Matl. Sci. & Engrg. Dept., Tehran Iran

In this report, the possibility of recovering zinc oxide from zinc oxide containing wastes has been studied. The ore was reduced in a fluidized bed furnace using a CO/CO2 mixture as reducing agent. It has been shown that with increasing in temperature and time of reaction, recovery percent will increase, also when depth of bed increases, recovery percent will decrease. The best result was shown that zinc oxide content in product was more than %93.5.

10:45 AM
Removal of Toxic Ions from Dilute Waste Streams by Novel Electrodeposition Techniques: *Michael L. Free*[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 416, Salt Lake City, UT 84109 USA

Removal of toxic ions from dilute waste streams by novel electrodeposition techniques will be discussed. The effects of electrode types, potentials, and electrodeposition parameters will be presented.

11:10 AM
New Generation Special Cements and the Using Theirs in Industry: *N. Ilyoukha*[1]; V. Timofeeva[1]; [1]Academic Ceramic Center, 8 Frunze Str., 61002, Kharkov Ukraine

New cements produced by the solid state sintering, contents compounds having double oxides was used as the raw stuff. Its are efficient bonds for castables, rammed mixes, covering and guniting. Refractory composite materials based on them are meant to protect units from influence of temperature more than 18000 C and aggressive media and used for manufacturing lining in petrochemistry reactors, energy head-treatment and high-temperature gas-dynamic channels (more 20000 C), in refractory lining of quarts glass tanks, for production crucibles used in the melting of pure metals, including alloys on rare-earth elements, high-temperature oxides, in burial of wastes nuclear reactors. Composites from oxides zirconium, hafnium are intended for use as solid electrolytes in the high temperatures electrolyzes, fuel elements, gas analyzers sensors and oxidability steel sensors, oxygen pumps as well as for the service at the temperatures up to 23000 C as construction products (pipes, sheaths, crucibles). The using of composites based on special cements ensures to increase in the heating units up to more 23000 C, to production of monocrystal high purity degree at the expense of theirs contamination exclusions by the harmful admixtures. The production of special cements and composites based on them can be organized at a joint venture.

High Risk Technologies in Metallurgy with Commercial Potential: Session I

Sponsored by: TMS, PGA-Public and Governmental Affairs Committee
Program Organizers: Jean-Louis Staudenmann, NIST, Advanced Technology Program, Division 473, Gaithersburg, MD 20899-4730 USA; Diran Apelian, Worcester Polytechnic Institute, Metal Processing Institute, Worcester, MA 01609-2280 USA

Wednesday AM Room: 210A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Jean-Louis Staudenmann, NIST, Advd. Tech. Prog., Gaithersburg, MD 20899-4730 USA; Diran Apelian, Worcester Polytechnic Institute, Metal Procg. Inst., Worcester, MA 01609-2280 USA

8:30 AM Introductory Remarks

8:40 AM
Accelerated Materials Development by Computational Design: *Greg B. Olson*[1]; [1]Northwestern University/QuesTek Innovations LLC, 225 Sheridan Rd., Evanston, IL 60208 USA

Responding to the excessive time and cost of traditional empirical materials development, a multi-institutional multidisciplinary research initiative over the past two decades has delivered a revolutionary Materials by Design(TM) technology based in computational systems design. First demonstrated in nanodispersion-strengthened high-performance steels, the technology is now being applied to Ni-base, Nb-base and Al-base superalloys, advanced shape-memory alloys and bulk metallic glasses. In a collaboration with the Center for Heat Treating Excellence (CHTE) at WPI, mechanistic simulations accelerate the process optimization of a new class of High-Temperature Carburizing steels for advanced gear and bearing applications. Under the DARPA-AIM initiative, simulation and probabilistic modelling are integrated with efficient experimentation to accelerate the full development and qualification cycle. An early demonstration of the AIM methodology is underway in the ESTCP certification testing of the computationally-designed QuesTek Ferrium S53(TM) stainless landing gear steel.

9:05 AM
High Viscosity Liquid and Semi-Solid Metal Casting: Processes and Products: Merton C. Flemings[1]; *William L. Johnson*[2]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 4-415, Cambridge, MA 02139 USA; [2]California Institute of Technology, Dept. of Matls. Sci., 1200 E. Calif. Blvd., MC 138-78, 339 Keck Lab., Pasadena, CA 91125 USA

Semi-solid forming has now taken its place among advanced industrial processing methods. It is becoming a process of choice for cast aluminum automotive parts requiring high strength and integrity. The main benefits of semi-solid forming being exploited today arise from the high viscosity of the semi-solid metal and from its reduced solidification shrinkage. In spite of the fact that the process in now over thirty years old, we continue to learn much about the fundamentals of formation of the desired metallurgical structure, the rheological properties of this structure, and about new economic ways of practicing the technology. Another, much newer, casting process that also benefits from high viscosity and low solidification shrinkage is that of forming bulk metallic glasses. These glasses have properties that make them attractive for a number of applications ranging from cell phone cases to sports applications. Our understanding of the rheological behavior of these materials is now developing quickly, as are concepts for new forming processes that will take advantage of the unique rheology.

9:30 AM
The Rapid Plasma Quench Production of Ultrafine Metal and Ceramic Powders: *Alan Donaldson*[1]; Ronald Cordes[1]; [1]Idaho Titanium Technologies, Idaho Falls, ID USA

The plasma quench process uses plasma to heat reactants to a temperature where desired reactions take place, then expanding the product gases through a Delaval supersonic expansion nozzle into a cold gas to prevent back reactions. Cooling rates are in excess of a billion Kelvins per second. We have produced high yields of titanium, magnesium, and aluminum by this process, in several reactor configurations. The initial product is always a submicron powder that has condensed from the rapidly cooling gas, but we are developing methods for increasing the particle size into the low micron range by recycling the gas-entrained powder back through the condensation zone. An in-

line classifier removes particles when they reach the desired size. Supersonic gas exiting the nozzle drives the recirculation and rapidly and efficiently mixes the product gas with the cold quench gas by the eductor effect. We are currently working on problems associated with scaling up.

9:55 AM
The Development of the Isothermal Melting Process for Aluminum - A High Risk/High Value Technology: *C. Edward Eckert*[1]; [1]Apogee Technology, Inc., 1600 Hulton Rd., Verona, PA 15147 USA

The thermal efficiency of conventional fuel fired reverberatory melting furnaces used in the aluminum industry ranges from 15 to 45%. Melt loss averages 5 to 8%. The industry wide implementation of a melting process capable of a gross thermal efficiency of 70% and 3% melt loss would result in a U.S. energy savings of 46 trillion BTU/year, if such a process were available. This paper describes a joint program between Apogee Technology, the U.S. Department of Energy, and Commonwealth Aluminum to develop a melting process that is capable of eclipsing the performance of 70% efficiency/3% melt loss. The key technology enabler for the process is a high heat flux direct immersion resistance heater. Such heaters do not commercially exist and therefore had to be developed under this program. The development of the heater is described in this paper, along with methods of risk management. Novel methods for heat production thorough electrical energy conversion, internal heater heat transfer, and overall protection of the system from molten aluminum had to be developed. Such a heater was developed with versions currently in b - site evaluation and, is discussed at length, in this paper.

10:20 AM Break

10:30 AM
UltraCem™: A Coating With High Wear Resistance and With a Low Coefficient of Friction: *E. McComas*[1]; Richard H. Bourret[1]; T. Dyer[1]; [1]Universal Chemical Technologies, Inc., 7825 SW Ellipse Way, Stuart, FL 34997 USA

UltraCem™ is a multifunctional tribological coating comprised of approximately 95% nickel and 6% boron that creates a hard, wear resistant surface with a low coefficient of friction, while achieving a metallurgical bond to ferrous alloys and a greater than 10,000 PSI bond on non-ferrous alloys. The topographic surface of UltraCem™ is nodular, resulting in the reduction of surface contact with mating surfaces and excellent locations for entrapment of lubrication. UltraCem™ is uniform as deposited, eliminating the requirement for secondary machining. Thickness can range from 2-500 microns and is deposited by autocatalytic reduction. UltraCem™ does not induce hydrogen embrittlement. As deposited, UltraCem™ is not completely amorphous and is in a transitional state between an amorphous mixture of nickel-boron and very fine crystalline nickel. As plated, the hardness is approximately equal to that of hard chrome or approximately 1000 KpH. After heat-treatment at between 400°F and 725°F, the coating crystallizes and clusters of nickel boride form to further increase hardness to approximately 1,400 KpH. The coating is unique in that it works extremely well against itself to reduce friction and eliminate the requirement for wet lubrication and mating surfaces. The result is a single coating with a high hardness, low friction, uniform deposit, achieving a metallurgical bond to ferrous substrates and a superior mechanical bond to even titanium without the induction of hydrogen embrittlement. The technology of UltraCem™ will be presented, its development will be reviewed, and the commercial impact it is making in both defense and commercial sectors will be discussed.

10:55 AM
Issues in the Commercialization of the Laser Engineered Net Shaping (LENS®) Process: *William Hofmeister*[1]; Michelle Griffith[2]; [1]Vanderbilt University, Chem. Engrg., Olin Hall Room 107, 24th & Garland Ave., Nashville, TN 37212 USA; [2]Sandia National Laboratory USA

The promise of "on-demand" part fabrication from computer-aided design (CAD) to direct metal deposition led Sandia National Laboratories to form a Cooperative Research and Development Activity (CRADA) to support the LENS® process. Twelve industrial partners joined the CRADA team with interests spanning from injection mold to aerospace parts fabrication. Over the four years, the group covered a wide range of issues; including development of process parameters, mechanical testing of LENS® deposited parts, refinement of tool path generation codes, sensors for process control, process modeling, machine development, cost analysis, and various intellectual property issues. Despite this large body of development work, the full commercial potential of the LENS® process has not been realized. We will outline the CRADA path to the development of the LENS® process and discuss the issues involved in acceptance of the process by the fabrication community.

11:20 AM
High Speed Identification and Sorting of Nonferrous Metal Scrap: *David B. Spencer*[1]; [1]Spectramet, LLC, 7 Alfred Cir., Bedford, MA 01730 USA

Each year, U.S. industry discards tens of billions of pounds of nonferrous metals as waste because it is either impractical or uneconomical to recover this material using current technology. In addition, billions of pounds of nonferrous metals are shipped overseas to China and elsewhere, for separation into higher value scrap grades using low cost labor for visual identification and hand sortation. Methods of scrap metal sortation using spark testing, chemical analysis, and optical emission or spectrographic analyzers are typically slow and cumbersome. Heavy media techniques are rapid, but can only sort by density. Color sortation techniques are effective for grouping alloys but cannot effectively sort by alloy type. An opportunity exists to recover low-grade nonferrous scrap by sorting them into various alloys with high accuracy by applying spectrographic analysis techniques. A new company dedicated to commercializing this technology, named Spectramet® LLC, is in the midst of developing a platform of new, high-speed identification and sorting technologies using optoelectronic methods. The Spectramet Technology can sort a wide range of metals and alloys both quickly and accurately. This paper will describe the progress of work funded by the National Science Foundation and the NIST Advanced Technology Program aimed at high-speed identification and sorting of mixed nonferrous metal scrap.

11:45 AM
Fluidized Beds - The Quantum Leap in Thermal Processing: *James Van Wert*[1]; [1]Amcast Industrial Corporation, Dayton, OH 45459 USA

As companies move to implement lean manufacturing concepts to achieve the ultimate goal of "one piece flow", the first step in achieving the goal is to map the "value stream". A value stream is all the actions or process steps, both value added and non-value added, currently required to produce the product. In the foundry industry and in many other metal-processing industries, one of the main process steps in the value stream is the thermal treatment process, commonly called "heat treatment". As the industrial engineers working on the future state value stream map soon find out, what they have discovered is not only the most significant bottleneck, but also a plant monument. Many of the thermal treatment practices and equipment were developed decades ago and have seen little improvement until now. This paper will attempt to describe the next generation in thermal processing equipment, to achieve a reduction in solution heat treatment and artificial aging times by as much as 125%. The primary aluminum alloy families currently being studied are the Aluminum-Silicon-Magnesium alloys and the Aluminum-Silicon-Copper-Magnesium alloys. These alloys have demonstrated significant microstructural changes along with dramatic cycle time reductions.

Hume Rothery Symposium: Structure and Diffusional Growth Mechanisms of Irrational Interphase Boundaries: Session V

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS)
Program Organizer: H. I. Aaronson, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

Wednesday AM
March 17, 2004
Room: 208A
Location: Charlotte Convention Center

Session Chair: W.-Z. Zhang, Tsinghua University, Beijing 100084 China

8:30 AM Invited
Edge-to-Edge Matching in Thin Films: *Christophe Detavernier*[1]; Christian Lavoie[1]; Ahmet Ozcan[2]; [1]IBM T.J. Watson Research, 1101 Kitchawan Rd., Rte. 134, PO Box 218, Yorktown Heights, NY 10598 USA; [2]Boston University, Dept. of Physics, 590 Commonwealth Ave., Boston, MA 02215 USA

We demonstrate the presence of a new type of texture in thin films of cubic $CoSi_2$, tetragonal α-$FeSi_2$ and orthorhombic NiSi formed on Si(100) substrates. Pole figures for these films consist of complex patterns of narrow lines. For NiSi, the patterns are created by grains for which the NiSi(202) or (211) planes are parallel to Si(110)-type planes in the substrate. Because of the constraint that a set of planes in the film is preferentially parallel to a set of planes in the substrate, the texture manifests itself as an off-normal fiber texture, with the fiber axis perpendicular to Si(110)-type planes (i.e. at 45° and 90° from the surface normal). Since the spacing between NiSi(202) or (211) planes is nearly identical to the spacing of 0.1920nm between Si(220) planes, an alignment of these planes will result in an interface that is periodic along one direction in the plane of the interface.

9:10 AM Invited
The Orientation Relationships and Interfacial Structure of S Phase Precipitates in Al-Cu-Mg Alloys: *Barry Muddle*[1]; Jian-Feng Nie[1]; Graham Winkelman[1]; [1]Monash University, Sch. of Physics & Matls. Engrg., Victoria 3800 Australia

One of the major precipitate phases in Al-Cu-Mg alloys is S phase that has an orthorhombic structure. It is a common view that this S phase forms in the form of lath, with its habit plane and long axis parallel to {210} and <001>, respectively, of the matrix phase. It was reported recently that this S phase also form in other orientation relationships. However, the interfacial structure of the S phase in these newly observed orientation relationships and its role in the growth are yet to be established. This paper reports results of detailed characterisation of the orientation and interfacial structure of S phase precipitates using high-resolution transmission electron microscopy and electron microdiffraction. The existence of a discrete set of orientation relationships between S and α-Al matrix will be demonstrated, each related by a small angular rotation about a common axis parallel to the long axis of the laths. The correlation between interfacial structure and orientation of precipitates, and the applicability of a Moire plane approach to the morphology of the S phase will be described and discussed.

9:50 AM Invited
Atomic Structure of Incoherent Interfaces in Zr-N Alloys: Peng Li[2]; *James M. Howe*[1]; William T. Reynolds[3]; [1]University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA 22904-4745 USA; [2]Arizona State University, Ctr. for Solid State Sci., Tempe, AZ 85287-1704 USA; [3]Virginia Polytechnic Institute, Dept. of Matls. Sci. & Engrg., Blacksburg, VA 24060 USA

The atomic structures of incoherent interfaces in metal alloys are not well understood. In this investigation, conventional and high-resolution transmission electron microscopy (TEM), crystallographic analyses and near-coincident-site (NCS) atom modeling were used to investigate the atomic structures of planar {111} and {110} ZrN interfaces between the Zr and ZrN phases in a Zr-34at.%N alloy. In all cases, the Zr and ZrN phases have high-index orientation relationships and display poor atomic matching in the high-resolution TEM images and NCS atom analyses, indicating that the interfaces are atomically incoherent. The {111} ZrN interfaces are faceted over hundreds of nanometers and contain atomic steps, suggesting that such features cannot be used to define partial coherency at interfaces. The {110} ZrN interface is rougher than the {111} ZrN interfaces, indicating that there may be both atomically faceted and rough incoherent interfaces. This research was supported by NSF under Grant DMR-9908855.

10:30 AM Break

10:45 AM Invited
On Relationships between Coherency, Structure and Energetics of Irrational Interphase Boundaries: *Thaddeus (Ted) B. Massalski*[1]; William A. Soffa[2]; David E. Laughlin[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]University of Pittsburgh, Matls. Sci. & Engrg., Benedum 842, Pittsburgh, Pittsburgh, PA 15260 USA

In this paper we will put the above topic in perspective, first by reviewing the classical definition(s) of coherency and then by reviewing the reasons for the perceived need to extend this concept to include one dimensional commensuration. We shall consider critically both past and current ideas in this area (including the present symposium). The experimental observations will be reviewed and discussed, as will be the work which is theoretical and/or simulated. The importance of energetics will be explored for both the nucleation and the growth processes of irrational boundaries.

Internal Stresses and Thermo-Mechanical Behavior in Multi-Component Materials Systems: Anisotropy and Residual Stresses

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee, EMPMD-Thin Films & Interfaces Committee, SMD-Composite Materials Committee-Jt. ASM-MSCTS

Program Organizers: Indranath Dutta, Naval Postgraduate School, Department of Mechanical Engineering, Monterey, CA 93943 USA; Bhaskar S. Majumdar, New Mexico Tech, Department of Materials Science and Engineering, Socorro, NM 87801 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Darrel R. Frear, Motorola, Tempe, AZ 85284 USA; John E. Sanchez, Advanced Micro Devices, Sunnyvale, CA 94088 USA

Wednesday AM — Room: 209B
March 17, 2004 — Location: Charlotte Convention Center

Session Chairs: John J. Lewandowski, Case Western Reserve University, Cleveland, OH 44106 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Sci. Ctr., Los Alamos, NM 87545 USA

8:30 AM Invited
In-Situ Deformation and Fracture Response in Ti-Al Based Alloys: *Bimal Kad*[1]; Bjorn Clausen[2]; Mark A.M. Bourke[2]; [1]University of California, Structural Engrg., 409 Univ. Ctr., La Jolla, CA 92093-0085 USA; [2]Los Alamos National Laboratory, LANSCE-12, Los Alamos, NM 84505 USA

TiAl-based low-symmetry alloys with single or two-phase microstructures are probed via neutrons during in-situ compression loading at ambient temperatures. Strain measurements are recorded for the predominant γ-TiAl phase and the minority α_2 phase both parallel and perpendicular to the loading axis, in the loaded and unloaded states, in the elastic and plastic regimes. Single phase α_2-Ti_3Al and γ-TiAl behavior is extracted using sample compositions to the left and right respectively of the $\alpha_2+\gamma$ phase field. The composite response is probed using a variety of alloy compositions and processing conditions within the $\alpha_2+\gamma$ phase field. The deformation anisotropy of the constituent phases, particularly in the <a> and <c> directions, following load excursions into the plastic regime is elucidated. This anisotropy is correlated with the underlying crystallographic deformation systems (slip and/or twinning) in the constituent phases. Such anisotropic response is modified in the two-phase alloys with subtle evidence of the particular contribution of the α_2-phase in improving the bulk response in commercially viable alloys.

8:55 AM
Co-Deformation and Residual Stress Accumulation in Alpha-Beta Brass: *Kelly T. Conlon*[1]; David Dye[2]; [1]National Research Council of Canada, Chalk River Labs., Sta. 18, Chalk River, Ontario K0J 1J0 Canada; [2]Imperial College London, Dept. of Matls., S. Kensington Campus SW7 2AZ UK

We have investigated the accumulation of microstrains during the deformation of alpha-beta Cu-Zn alloys using in-situ neutron diffraction. Dual phase Cu-Zn alloys were fabricated by melting and casting elemental Cu and Zn granules of nominal 99.9% purity. Subsequent hot extusion of the ingots results in a modest rod-like texture in both phases, with alpha (111) and beta (110) fibres aligned along the extrusion axis of the rod. In-situ neutron diffraction experiments were carried out on specimens machined from the rods using a tesile testing rig mounted on a neutron diffractometer. The sign and magnitude of the residual strains in the minority beta phase following the application of tensile plastic strains demonstrate an unusual grain orientation dependence. Beta phase grains with a (002) orientation aligned along the tensile axis accumulate enormous residual tensile elastic strains of 1% following a deformation of 10%. On the other hand, the sign and magnitude of the strains in beta grains with a (220) orientation are modest and compressive, indicating the onset of yield and subsequent co-deformation with the alpha matrix. These unusual results will be discussed in light of older results obtained by Izumi and co-workers on dual phase alpha-beta bi-crystals.

9:20 AM Invited
Residual Stress Analysis in Graphite/Pmr-15 Composites Using X-Ray Diffraction: B. Benedikt[1]; M. Lewis[1]; *P. Rangaswamy*[1]; M.

Kumosa[2]; P. Predecki[2]; M. Gentz[2]; [1]Los Alamos National Laboratory, Engrg. Scis. & Applications Div., Los Alamos, NM 87545 USA; [2]University of Denver, Div. for Advd. Matls. & Struct., Dept. of Engrg., Denver, CO 80208 USA

This work addresses the problem of determining thermally induced residual stresses in graphite/PMR-15 composites from embedding aluminum inclusions between the first and second plies of 6-ply unidirectional and 4-ply woven laminates. The stresses in the inclusions were measured using X-ray diffraction (XRD) from the (422) aluminum diffracting planes. These stresses were than used to calculate the average thermal residual stresses in the composites using the visco-elastic Eshelby method. Furthermore a comparison were made between the measured stresses from Xrd experiments and calculated stresses from the Eshelby method with predictions obtained from the visco-elastic CLPT (inter-laminar model) and the Eshelby method (intra-laminar model). The accuracy of these results was further validated using a four-point bend test for the same material using different bending moments. The talk will highlight the merits of the proposed methodology of using metallic filler particles as sensors to measure internal and applied stresses in polymeric composite systems. The application of this methodology towards investigating the effect of matrix degradation on thermally induced residual stresses by aging in air and nitrogen for 1064 hours and 1170 hour, respectively, at 315°C will also be presented.

9:45 AM Invited
A Study of Residual Stresses and Mechanical Properties of Thermal Spray Coating Systems: *Thomas Gnäupel-Herold*[1]; Frank S. Biancaniello[2]; Henry J. Prask[1]; [1]National Institute of Standards and Technology, Matls. Sci. & Engrg. Lab., 100 Bureau Dr., Stop 8562, Bldg. 235, Rm. B105, Gaithersburg, MD 20899 USA; [2]National Institute of Standards and Technology, Metall. Div., 100 Bureau Dr., Stop 8556, Gaithersburg, MD 20899-8556 USA

The thermal spray process produces a deposit characterized by residual stress (RS) and a microstructure with high defect density. The first is caused by the differences of temperatures and coefficients of thermal expansion (CTE) between deposit and substrate. The imperfect microstructure is caused by the deposition of individual droplets which creates incomplete contact between splats as well as porosity and inclusions. For several coating systems we have conducted an investigation of residual stresses, elastic constants, defect structure and adhesive properties. We found that for medium to high porosities the RS are controlled by the drop in the effective coating elastic modulus. For low defect densities the coating properties approach the bulk values, and both the CTEs and the deposition temperature determine the RS. The elastic modulus depends on the defect density which is controlled by, among other parameters, the particle/droplet size.

10:10 AM Invited
Effects of Residual Stresses on Fatigue of Composite Systems: *John J. Lewandowski*[1]; H. Hassan[1]; C. Liu[1]; [1]Case Western Reserve University, Dept. of Matls. Sci. & Engrg., Cleveland, OH 44106 USA

Discontinuously reinforced aluminum based composites have been prepared to contain different levels of residual stress. The levels of residual stress were characterized by X-ray diffraction as well as via measurement of the Bauschinger effect in materials heat treated to different conditions. The low cycle fatigue behaivor was characterized in these cases. In addition, the effects of residual stresses on fatigue crack growth in laminated aluminum composites were also evaluated after heat treatment as well as after different thermal exposures.

10:35 AM
In-Situ Neutron Diffraction Studies of Directionally Solidified NiAl-Mo Fibrous Composites During Tensile Loading: *H. Bei*[1]; E. P. George[2]; Hahn Choo[1]; Donald W. Brown[3]; G. M. Pharr[1]; [1]University of Tennessee, Matls. Sci. & Engrg., Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; [3]Los Alamos National Laboratory, Lujan Ctr., Los Alamos, NM 87545 USA

NiAl-Mo pseudo-binary eutectic alloys were directionally solidified in a high temperature optical floating zone furnace to produce well-aligned fibrous composites in which NiAl is the continuous matrix phase and Mo the discontinuous rod-like phase. Specimens machined parallel to the long axes of the fibers were loaded in tension at temperatures in the range 700-1000°C and stresses of 300-500 MPa while in-situ neutron diffraction experiments were performed to investigate the detailed deformation mechanisms in the composite. In particular, changes in load partitioning in the two phases were studied in various stress and temperature regimes where the matrix and fiber are elastic/elastic, plastic/elastic, and plastic/plastic. In addition, attempts were made to estimate dislocation densities in each phase by observing line broadening.

11:00 AM
Thermal Residual Stress Characterization in TiC-Ni3Al Intermetallic Matrix Composties Via In-Situ Neutron Diffraction: *James J. Wall*[1]; Hahn Choo[2]; James W. Richardson[3]; Terry N. Teigs[4]; Peter K. Liaw[2]; [1]University of Missouri, Dept. of Mech. & Aeros. Engrg., Columbia, MO 65211 USA; [2]University of Tennessee, Matls. Sci. & Engrg. Dept., Knoxville, TN 37996 USA; [3]Argonne National Laboratory, Intense Pulsed Neutron Source, Argonne, IL 60439 USA; [4]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

TiC-Ni3Al composites were produced by reactive vacuum sintering of TiC/Ni/NiAl compacts, followed by low-pressure hot-isostatic pressing. Upon cooling from the processing temperature, thermal residual stress (TRS) was developed in the composite due to the thermal expansion coefficient mismatch of the two constituents. The residual stress was measured using in-situ neutron diffraction over a temperature range of approximately 50-1250K. A 3-dimensional elasto-viscoplastic (EVP) finite-element model (FEM) was used to predict the TRS evolution. The measured TRS behavior deviated from that predicted by the EVP model. The Ni3Al matrix demonstrated a high resistance to both creep relaxation and low-temperature plasticity under the temperature regime studied. Possible reasons for the unanticipated TRS evolution are discussed. This work is supported by the NSF International Materials Institutes (IMI) Program DMR-02331320.

11:25 AM
Multiscale Internal Stress Investigation of BaTiO3: *Robert Rogan*[1]; Ersan Ustundag[1]; Nobumichi Tamura[2]; Lawrence Margulies[3]; Henning Poulsen[3]; Ulrich Lienert[4]; Mark R. Daymond[5]; [1]California Institute of Technology, Dept. of Matls. Sci., Pasadena, CA 91125 USA; [2]Lawrence Berkeley National Laboratory, Advd. Light Source, Berkeley, CA 94720 USA; [3]Risø National Laboratory, Matls. Rsch. Dept., Roskilde Denmark; [4]Argonne National Laboratory, Advd. Photon Source, Argonne, IL 60439 USA; [5]Rutherford-Appleton Laboratory, ISIS Neutron Scattering Facility, Chilton, Didcot OX11 0QX UK

For the first time, internal and residual stresses in BaTiO3 were investigated at multiple length scales using X-ray microdiffraction. Tetragonal BaTiO3 single and polycrystals and thin films were scanned with X-ray beams ranging in size from 1 mm to 200 mm. Laue patterns were collected from regions around cracks and highly stressed areas. These patterns yielded information about orientation relationships caused by domain boundaries as well as the three-dimensional strain tensor associated with each feature. In one particular study, the evolution of elastic strain and texture (domain switching) in a single embedded grain of a polycrystalline BaTiO3 under electrical loading was investigated. The results suggest the presence of significant internal stress/strain fields and complicated domain formations inside BaTiO3.

International Laterite Nickel Symposium - 2004: Process and Operational Lessons Learned - Part I

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Process Mineralogy Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizer: William P. Imrie, Bechtel Corporation, Mining and Metals, Englewood, CO 80111 USA

Wednesday AM Room: 217B/C
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Ian G. Skepper, BHP-Billiton, Ravensthorpe Nickel Project, Perth, WA 6850 Australia; John G. Reid, Director, Reid Resource Consulting Pty. Ltd., Indooroopilly, Brisbane 4068 Australia

8:30 AM
Sixty Years of Caron: Current Assessment: Faustino G. Prado[1]; *Faustino L. Prado*[1]; [1]Prado Technology Corporation, Mining & Minerals, PO Box 274206, Tampa, FL 33688 USA

For more than a century, the AAC (ammonia/ammonium carbonate) process has been applied to the recovery of various metals. A variation of it, the Caron Process was first used in the early 1940's in Cuba for the extraction and recovery of nickel from laterites. The original Nicaro plant has been in continuous operation since then.

Years later the Caron process was used again in Australia at the Greenvale (Yabulu) Nickel/Cobalt plant, commissioned in 1974. Other projects (Sered, Niquelandia, Nonoc, Punta Gorda, Albania) as well as several studies have been carried out. The Caron Process is alive and still promising. Members of our organization have been working with AAC and the Caron Process since 1952. This paper will review current approaches and potential improvements (enhancement of recoveries of Ni and Co, reduced energy consumption and maintenance simplification).

9:00 AM
Yabulu 25 Years On: *John G. Reid*[1]; John E. Fittock[2]; [1]Reid Resource Consulting Pty Ltd., 1 Carnoustie Ct., Indooroopilly, Brisbane, Qld 4068 Australia; [2]BHP Billiton, QNI Pty Ltd., PMB 5, Mail Ctr., Townsville, Qld 4818 Australia

When the paper "Operations at the Greenvale Nickel Project Mine and Refinery" was delivered at the 1st Laterite Symposium in 1979, the Yabulu plant had been operating for only 4 years. The design was based on the Caron plant at Nicaro, Cuba, built in 1944, also by Freeport. Yabulu had 10 Herreshoff ore roasters and production in 1978 was 18,680 tonne of nickel in impure oxide sinter and 763 tonne cobalt in an impure mixed sulphide. In the past 25 years two more roasters have been added, but ore processing rate increased only marginally, to 2.4M t/y, to allow a lower feed rate per roaster, with associated higher recovery. Imported ore has replaced the closed Greenvale mine. Nickel production in the past 25 years has risen by 67% and cobalt by 146% and the products have changed completely. The plant changes that have contributed to the increased recovery, production and product value are discussed.

9:30 AM
The Evolution of the Greek Ferronickel Production Process: *Emmanuel N. Zevgolis*[1]; [1]National Technical University of Athens, Sch. of Mining & Metallurgl. Engrg., 9 Heroon Polytechniou Str., Athens, Zografou 15780 Greece

After the Krupp-Renn process was shown to be unsuitable for treatment of the Greek nickeliferous laterites, the LM process was developed. It involved roasting reduction up to metallic iron in a Rotary Kiln (R/K) and smelting in an Electric Furnace (E/F). Ferronickel (with 15% Ni) produced in the E/F, was upgraded to 90-95% Ni in an O2 converter. Raw Ni was then electrolyzed to pure Ni. The LM process was not applied for very long however, due to various problems. So, the Larco process was developed, i.e., pre-reduction in R/Ks, smelting of the calcine in E/Fs for ferronickel production (15% Ni) and enrichment to 20% Ni, in an O2 converter. Until now, many technological improvements have been invented and applied or adjusted to the process, which have made it a fully reliable and competitive one. Some of the improvements were the invention and application of a new granulation process for liquid ferronickel, the modification of the existing R/Ks, E/Fs and converters and the design of new ones, and others that are described in the paper.

10:00 AM Break

10:10 AM
The Optimisation and Control of Process Chemistry at Bulong Nickel Operations: *John Frederick O'Callaghan*[1]; [1]BHP Billiton, Ravensthorpe Nickel Ops., Mt Newman House, Level 12, 200 St. Georges Tce, Perth, Western Australia 6000 Australia

Bulong remains the only hydrometallurgical process plant in the world that recovers nickel and cobalt directly from high pressure acid leach (HPAL) laterite leach liquors without need for precipitation of an intermediate product (e.g. sulphide, hydroxide or carbonate). Direct solvent extraction (DSX) of both nickel and cobalt in combination with electrowinning of nickel has been operation at Bulong since early 1999. Focus in the first years has been on ramp-up and resolution of commissioning problems. More recently the focus has been on debottlenecking and process optimisation. A number of significant process problems have been resolved, the most difficult being the formation of gypsum in the nickel solvent extraction circuit. Sound control of process chemistry is essential to ensure the production of high quality nickel and cobalt products at the lowest possible cost. This paper describes the numerous changes and enhancements to the process chemistry and plant equipment at Bulong and discusses the importance of ore leach chemistry on downstream impurity metal removal. Potential additional enhancements to further reduce unit costs are also presented.

10:40 AM
Two Steps Forward and One Step Back: A Case in Arrested Development in Laterite Processing: *Larry M. Southwick*[1]; Stanley Duda[2]; [1]L.M. Southwick & Associates, 992 Marion Ave., Ste. 306, Cincinnati, OH 45229 USA; [2]Consulting Engineer, PO Box 1014, La Canada, CA 91011 USA

Considerable process and equipment development and analysis were conducted during design of Freeport Sulfur's laterite nickel facilities in the late 1950' s. These covered ore concentration at Moa Bay, Cuba, and concentrate refining at Port Nickel, Louisiana. Both facilities utilized acid pressure leaching and product refining steps that were similar to those later used elsewhere in the laterite processing industry. Unfortunately, developments in Cuba removed the Moa Bay facilities from Freeport control. This deprived the project of a start-up that would have allowed thorough resolution of difficulties and a quicker attainment of production capacity. This meant lost opportunity to positively prove and complete the technology when optimum resources and staff were available. This paper reviews the original rationale of how design was developed via laboratory, bench scale and pilot studies by Freeport Sulfur. Some later operating problems will also be discussed. This case is unusual, but the lessons learned may be instructive in the application of difficult and complicated technologies, as well as the need to understand and retain access to the original development and design activities.

11:10 AM
Reduction in Energy Costs in Cuban Caron Process Plants: *Raul Rodriguez Gan*[1]; [1]Principle Specialist for Nickel, Ministry of Basic Industries Cuba

Since the 1973 energy crisis, high fuel oil costs have been eroding the economy of the Nicaro and Punta Gorda Plants that employ the Caron Process for laterites processing. Since 1995, cuban crude oil production has growth dramatically, with progressive utilization in power generation and nickel production plants. It is explained how Punta Gorda and Nicaro have been able to burn high density cuban crude oil with high sulphur content, to replace the expensive Bunker C fuel oil that previously was purchased at low preferential price from USSR and achieving a profitable operation. Problems that arouse due to the high sulphur content of the cuban crude oil, have demanded extensive investigations in the process chemistry and novelty solutions, with corresponding changes in process conditions. Innovative options are shown that will allow further cost reductions in the future.

Lead-Free Solders and Processing Issues Relevant to Microelectronic Packaging: Interfacial Interactions, Intermetallics and Substrates

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee
Program Organizers: Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/ Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Michael R. Notis, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18015 USA; Jin Yu, Korea Advanced Institute of Science and Technology, Center for Electronic Packaging Materials, Department of Materials Science & Engineering, Daejeon 305-701 Korea

Wednesday AM Room: 219B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: C. Robert Kao, National Central University, Dept. of Chem. & Matls. Engrg., Chungli City 320 Taiwan; K. N. Subramanian, Michigan State University, Dept. of Chem. Engrg. & Matls. Sci., E. Lansing, MI 48824-1226 USA

8:30 AM Invited
Morphology and Kinetics of Interfacial Reaction Between SnAg3.5 Solder and Electroless Ni-P Metallization: *Zhong Chen*[1]; Min He[1]; Guojun Qi[2]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Nanyang Ave., Singapore 639798 Singapore; [2]Singapore Institute of Manufacturing Technology, Surface Engrg., 71 Nanyang Dr., Singapore 638075 Singapore

This work summarizes the interfacial reaction between lead-free solder SnAg3.5 and electrolessly plated Ni-P metallization in terms of morphology of the intermetallic compound (IMC) and its growth kinetics. Comparison with pure Ni as metallization is made whenever

possible so that the role of P in electroless nickel can be clarified. Three major aspects will be highlighted in this paper: 1. The effect of post-reflow cooling rate: It is found that the cooling rate significantly affects the morphology of IMC. For the first time we report the formation of a secondary phase nucleating on Ni3Sn4 IMC surface when the cooling rate exceeds certain limit. 2. Liquid-solid reactions: The formation and growth of Ni-Sn IMC are examined at different reflow parameters. IMC spallation and Kirkendall voids have been observed and discussed. 3. Solid-state reactions: The growth kinetics at different annealing temperatures has been studied. Kirkendall voids are found at the interface between Ni3P layer and Ni-P metallization after prolonged thermal aging.

8:55 AM

Effect of Cu Concentration on the Solid-State Reactions Between SnAgCu and Ni: *W. C. Luo*[1]; J. Y. Tsai[1]; C. W. Chang[1]; Y. C. Shieh[1]; C. Robert Kao[1]; [1]National Central University, Dept. of Chem. & Matls. Engrg., Chungli City 320 Taiwan

The SnAgCu solder is considered a very promising lead-free replacement for the Sn-37Pb solder. For industrial uses, a 0.2% uncertainty in composition is generally accepted. However, our recent study revealed that, during soldering, the Cu concentration had a very strong effect on the reactions between SnAgCu solders and Ni. When the Cu concentration was low (x=0.3 and lower), the reaction product was $(Ni_{1-x}Cu_x)_3Sn_4$ at the interface of a solder joint. At high Cu concentrations (x=0.6 and higher), the reaction product was $(Cu_{1-y}Ni_y)_6Sn_5$. When the Cu concentration was in-between (x=0.4 and 0.5), both $(Ni_{1-x}Cu_x)_3Sn_4$and $(Cu_{1-y}Ni_y)_6Sn_5$, formed. In other words, during soldering, the Cu concentration must be strictly controlled in order to obtain consistent results. In this study, we would like to extend our earlier study to investigate whether this strong concentration dependency also occurs during the solid-state aging of the solder joints. We aged solder joints at the solid-state at several different temperatures for time as long as 4000 hours. The solder compositions studied include SnAg0.2Cu, SnAg0.3Cu, SnAg-0.4Cu, SnAg-0.5Cu, SnAg-0.6Cu, SnAg-0.7Cu, and SnAg-1.0Cu. Analysis techniques used include optical microscope, SEM, EPMA, and XRD.

9:15 AM

Interfacial Reaction Study of Solder Joint with Sn-Ag-Cu Solder Ball and Various Solder Pastes in Lead Free WLCSP: *Huann-Wu Chiang*[1]; Morris Chen[1]; *Jeffrey C.B. Lee*[2]; Johnson Liu[2]; Simon Li[2]; [1]I-SHOU University, Matls. Sci. & Engrg., #1, Sec. 1, Hsueh-Cheng Rd., Ta-Hsu Hsiang, Kaohsiung County, Taiwan 84008 Taiwan; [2]Advanced Semiconductor Engineering, Inc., Engrg. Ctr., 26, Chin 3rd Rd., Nantze Export Procg. Zone, Kaohsiung 811 Taiwan

The interfacial reactions of solder joint between Sn-Ag-Cu solder ball and various solder pastes such as, Sn-3.0Ag-0.5Cu, 63Sn-37Pb and Sn-8Zn-Al, will be investigated in wafer level CSP package. After appropriate SMT reflow process on PCB with Cu ¨COSP and Cu-NiAu surface finish, samples will be subject to TCT (-40~125¡æ) 1000 cycle and HTS (150¡æ) 1000hrs reliability test. Sequentially, the cross-section analysis is also scrutinized by SEM/EDX to observe metallurgical evolution in the interface and solder bulk itself. The relationship among morphology, shear strength, and solder paste composition will be explored in the study. A mature Sn-37Pb solder ball wafer level CSP will be used for comparison as well.

9:35 AM

An Investigation of Intermetallic Phase Morphology in Lead-Free Solders: *Mohammad Faizan*[1]; *Robert A. McCoy*[2]; [1]Youngstown State University, Civil Engrg. Dept., Youngstown, OH 44555 USA; [2]Youngstown State University, Mech. Engrg. Dept., Youngstown, OH 44555 USA

During soldering of a copper substrate using Sn-rich, lead-free solders, a Sn-Cu interaction takes place at the copper/solder interface. Copper is dissolved into the molten solder and subsequently intermetallic compounds (IMC's) are formed at the interface. Understanding of the growth rate and morphology of IMC's at the copper/solder interface is critical in achieving reliable soldered joints. This paper presents an analysis of an experimental investigation of soldering of a copper substrate using two solder compostions: pure Sn and Sn-3.5% Ag. Experimental results indicated that the solder composition, the temperature of the molten solder, and the dwell time (time the molten solder remains in contact with the substrate) controlled the growth of the IMC's. The IMC's grew in a scalloped shape for both solders but the IMC's for the Sn-Ag solder were detected as more irregular with needle-like scallops.

9:55 AM

The Effect of Isothermal Aging on the Thickness of Intermetallic Compound Layer Growth Between Low Melting Point Solder and Ni Plated Cu Substrate: *Dae-Gon Kim*[1]; Seung-Boo Jung[1]; Chang-Youl Lee[1]; [1]Sungkyunkwan University, Dept. of Advd. Matls. Engrg., 300 chunchun-dong, jangan-gu, Suwon, Gyeonggi-do 440-746 Korea

The growth kinetics of intermetallic compound layer formed between low melting point solder (pure In, In-48Sn solder) and Ni/Cu substrate by solid state isothermal aging were examined for 0 to 100 days. A guantitative analysis of the intermetallic compound layer thickness as a function of time and temperature was performed. Experimental results showed that the intermetallic compounds, such as In27Ni10 and In11Sn23Ni16 were observed for different solders, respectively. The layer growth of intermetallic compound in the couple of low melting point solders/electrolytic Ni system satisfied the parabolic law at given temperature range. As a whole, because the values of time exponent(n) have approximately 0.5, the layer growth of the intermetallic compound was mainly controlled by diffusion mechanism over the temperature range studied. The apparent activation energies for intermetallic compound growth were 60.03 kJ/mol for In27Ni10 and 72.83 kJ/mol for In11Sn23Ni16, respectively.

10:15 AM Break

10:25 AM Invited

Interfacial Reaction of Lead-Free Sn-3.8Ag-0.7Cu, Sn-0.7Cu, and Sn-1.7Cu Solders with Pt: Tae hyun Kim[1]; *Young Ho Kim*[1]; [1]Hanyang University, Matls. Engrg., Haengdang-dong, seongdong-gu, Seoul 133-791 S. Korea

The interfacial reaction and the intermetallic formation in the interface betweem Sn solder containing small amount of Cu with Pt were investigated. Sn-0.7wt%Cu and Sn-1.7wt%Cu solders were reacted with Pt by dipping the Pt/Ti/Si specimens into the molten solder at 260°. Sn-3.8wt%Ag-0.7wt%Cu solder was reacted with Pt by reflowing the solder paste on the Pt/Ti/Si substrae at 250°. The intermetallic formation was characterized by using scanning electron microscopy, energy dispersive x-ray spectroscopy, x-ray diffractometry, and transmission electron microscopy. Pt-Sn interemtallic formed in the solder/Pt interface and Cu6Sn5 type interfacial intermetallic was not observed in all specimens. A parabolic relationship exist, between the thickness of the Pt-Sn intermetallic layer with reaction time, which indicates the intermetallic formation in the solder/Pt interface is diffusion-control.

10:50 AM

Intermetallic Compounds and Adhesion Strength Between the Sn-9Zn-1.5Ag-0.5Bi Lead-Free Solder and Unfluxed Cu Substrate: *Tzih-Yao Liu*[1]; Moo-Chin Wang[2]; Min-Hsiung Hon[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., 1 Ta-Hsueh Rd., Tainan 70101 Taiwan; [2]National Kaohsiung University of Applied Science, Dept. of Mech. Engrg., 415 Chien-Kung Rd., Kaohsiung 80872 Taiwan

The intermetallic compounds (IMCs) formed at the interface between the Sn-9Zn-1.5Ag-0.5Bi lead-free solder alloy and unfluxed Cu substrate have been investigated by X-ray diffraction (XRD), optical microscopy (OM), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and energy dispersive spectrometry (EDS). The melting point and melting range of the Sn-9Zn-1.5Ag-0.5Bi solder alloy are determined as 196°C and 10°C, respectively, by differential scanning carolimeter (DSC). The Cu6Sn5 and Cu5Zn8 IMCs were identified at the Sn-9Zn-1.5Ag-0.5Bi/unfluxed Cu interface as wetted at 250°C for 10 s. The interfacial adhesion strength increases from 7.36 ± 0.72 to 10.85 ± 0.56MPa with increasing the soldering time from 10 to 30 s.

11:10 AM

Coupling Effect on Interfacial Reactions of Flip Chip Solder Joints with Ti/Cr-Cu/Cu Under Bump Metallization and Ni-P/Cu/Au Pad Metallization During Reflow in Microelectronic Packaging: *Tung Liang Shao*[1]; Yuan Ming Huang[1]; Te Sheng Chen[1]; Chih Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Engrg., Jsin Shu 300 Taiwan

The soldering technology is still important and widely applied in the advanced flip chip product. The purpose of this study was to identify and prove the existence of coupling effect on interfacial reactions between flip chip solder bumps and the under bump metallization (UBM) systems. Eutectic Sn/Pb solders were applied for the samples. Three types of sample (including the dumped die, the bumped substrate and the flip chip package) were examined. The binary Cu-Sn compounds and the Ni-Sn compounds were formed for the bumped die and the bumped substrate after the first reflow. The tenary Ni-Cu-Sn

compounds were formed for the flip chip package during the second reflow. By comparing the composition of intermetallic compounds (IMC) for these three types of sample, the coupling effect on interfacial reactions of solders and the flip chip systems was proved. Not only Ni and Cu coupling were observed, but also Au coupling effect was found, which Au-Sn IMC was found on the surface of Ni-Cu-Sn IMC on the chip side.

11:30 AM
Inhibiting Growth of Ni3P Crystalline Layer in Ni(P) Substrate by Reacting with Cu-Doped SnAg Solders: *S. J. Wang*[1]; H. R. Kao[1]; C. Y. Liu[1]; [1]National Central University, Dept. of Chem. Engrg. & Matls. Engrg., No. 300 Jung-Da Rd., Chung-Li Taiwan

Interfacial reactions between Sn(Cu) alloys and Ni(P) substrates have been studied. Comparing to Sn/Ni(P), the formation of Ni3P layers in Sn(Cu)/Ni(P) reaction couples were very limited. The sluggish growth of Ni3P layer attributed to a layer of Cu-Sn compound layer formed on the Ni(P) substrate, which effectively isolated the Ni(P) substrate from reacting with solder. The eventual formation of Ni3P compound layer depended on the Ni diffusion in the Cu-Sn compound. Also, we found that the higher Cu-content Sn(Cu) alloys had less Ni3P formation. In this talk, we will present the kinetics of the interfacial reactions between Sn(Cu) alloys and Ni(P) substrates under solid-state aging and liquid-state reflowing. The correlation between the mechanical strength of solder joints and the interfacial reactions will be also reported.

11:50 AM
Effects of Substrate Metallization and Pad Structure on the UBM Degradation of Flip Chip Packages Under Thermal Aging: *Jenq-Dah Wu*[1]; [1]Advanced Semiconductor Engineering, RD Lab, 26, Chin 3rd Rd. Nantze Export Processing Zone, Kaohsiung 811 Taiwan

Thermal aging of flip chip packages is performed to investigate reliability issues associated with structural evolution of UBM and solder interconnects. UBM employed is a thin film Al/Ni(V)/Cu metal stack; while bump material is eutectic Sn/Pb solder. Structural integrity of UBM/bump system is characterized by die pull/shear tests of FCBGAS without underfill. Fracture strength degradation and transtion of failure patterns from ductile solder cleavage to brittle UBM fracture at Ni(V) layer provides evidence of UBM degradation under prolonged thermal aging. Two substrate surface finishes are considered in this paper, i.e. electroless Ni/immersion Au and OSP. It is observed that solder bumps of FCBGAs with OSP substrates remain intact after 3,000 hrs of HTST, while UBM failure is found for FCBGAs with Ni/Au substrates. This implies that Ni/Au substrate metallization plays critical role on the interfacial reactions of Al/Ni(V)/Cu UBM and solder bumps.

Magnesium Technology 2004: Casting Processes and Properties

Sponsored by: Light Metals Division, LMD-Magnesium Committee
Program Organizer: Alan A. Luo, General Motors, Materials and Processes Laboratory, Warren, MI 48090-9055 USA

Wednesday AM Room: 203B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Darryl Albright, Hydro Magnesium, Livonia, MI 48152 USA; Eli Aghion, Dead Sea Magnesium, Beer-Sheva 84111 Israel

8:30 AM
Effect of Inhibitor Gas on Mould - Magnesium Reactions in Investment Casting: *Zhan Zhang*[1]; Guy Morin[2]; [1]Intermag-Modelex Inc., Saint-Nicolas, PQ Canada; [2]Centre Intégré de Fonderie et de Métallurgie, Trois-Rivières, PQ Canada

In order to assess the behaviour of mould-magnesium reaction, the ceramic shell moulds with different binder and refractory particles were prepared for pouring AZ91 magnesium alloy. To restrict mould-magnesium reactions, inhibitor gas was guided into shell moulds for removal of oxygen in shell moulds and formation of barrage between mould and magnesium. The results of experiments show that a mixture of CO2 and proper concentration of SF6 used as inhibitor gas can effectively limit mould-magnesium reactions even if the temperature of ceramic shell moulds is higher than 450°C. A surface analysis with AES (auger electron spectroscopy) and ESCA (electron spectroscopy for chemical analysis) has been performed on the surface of magnesium parts cast under the inhibitor gas. It was discovered that a special layer appeared on the surface, in which the elements such as magnesium, oxygen, fluorine, aluminium, carbon, silicon, etc. were detected. The thickness of fluorine-containing layer varies with the variation of the concentration of fluorine in the gas mixture. The models of mould-magnesium reactions and their preventions are described in this paper.

8:50 AM
The Role of Microstructure and Porosity in Ductility of Die Cast AM50 and AM60 Magnesium Alloys: *Gurjeev Chadha*[1]; John E. Allison[2]; J. Wayne Jones[1]; [1]University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48105 USA; [2]Ford Motor Company, Matls. Sci. Dept., Dearborn, MI 48124 USA

The deformation and fracture behavior of die-cast AM50 and AM60 magnesium alloys have been examined to determine the processes leading to fracture in bending and tension and to elucidate the influences of microstructure and porosity on ductility. Damage accumulation in terms of crack initiation, growth, linking and eventual failure was quantified as a function of strain for as-cast plates with section thicknesses of 2, 6 and 10mm. Microstructure and porosity distribution are dependent on section thickness, with bands of high porosity resulting from the nature of die filling. In both tensile and three-point bend studies, damage accumulation, and subsequent ductility at fracture, are strongly influenced by the heterogeneity in porosity distribution, with early crack formation occurring in regions of highest porosity. The role of microstructure on the growth of cracks from porosity during straining has been examined in situ by scanning electron microscopy and the critical strains to fracture have been studied by a surface strain mapping technique to determine the nature of strain localization around pores in these alloys. Using this information, a model is constructed that relates the observed ductility of these alloys with critical microstructural features.

9:10 AM
First Magnesium V-Process Casting: *Sayavur I. Bakhtiyarov*[1]; *Ruel A. Overfelt*[1]; [1]Auburn University, Mech. Engrg., 202 Ross Hall, Auburn, AL 36849-5341 USA

The Solidification Design Center (Auburn University, AL, a NASA Research Partnership Center) and Jones Engineering, Inc. (Lawrenceville, GA) conducted the first successful magnesium V-process (vacuum sealed molding process) casting. Since the invention of the V-process casting technique in Japan in 1971, it has been believed that the unique casting process is suitable for any metal (gray, ductile, malleable iron, various grades of steel or aluminum and copper base alloys) except magnesium. Currently die casting is the primary technique for production of magnesium parts for high volume automotive applications. However, the need for magnesium structural components spurs the need for alternative processes. Application of this vacuum-sealed manufacturing process for magnesium casting directly addresses the high priority needs identified in the Metalcasting Industry Technology Roadmap and the primary mission of the AFS Magnesium Division - the development of alternative casting processes for magnesium. An exploration of the possibility of combining the benefits of V-process casting with the advantages of magnesium was really challenging mainly due to low thermal heat content and high chemical reactivity of magnesium. In this paper we present casting procedures of the first magnesium V-process casting. The major mechanical properties and metallography of the casting are studies and compared to those obtained for magnesium sand casting.

9:30 AM
Evaluation Between Mechanical Properties and Die Casting Process Control by Toguchi Method for Magnesium Alloy AM60B: *Chi Ming Hung*[1]; Mao Sheng Chang[1]; [1]Metal Industries Research & Development Center, Casting Tech. Sect., 1001 Kaonan Hwy., Kaohsiung, Taiwan 811 Taiwan

On demanding of environmental protection, light metals are used more and more in automobiles in recent years. It is a good choice to apply more magnesium castings to an automobile to reduce its weight so that the emission amount of CO2 will decrease. When it comes to automobile parts, the mechanical properties are very important. However, the mechanical properties of die casting are significantly influenced by the process parameters. The study is to examine the effects of cold chamber die casting process parameters on mechanical properties of AM60B magnesium alloy. We choose four process parameters-die temperature, second phase speed, pressure and cooling time- as the control factors, and use Toguchi method to analyze which of them has important influence on mechanical properties of AM60B.

9:50 AM Break

10:20 AM
Characterization of Mg Alloy Solidification and As-Cast Microstructures: *Y. W. Riddle*[1]; M. M. Makhlouf[1]; [1]Worcester Polytechnic Institute, Advd. Casting Rsch. Ctr., Worcester, MA USA

In previous research presented at TMS 2003 we established a non-equilibrium thermal analysis method and apparatus for the study of Mg alloy solidification. In the present research a specialized dendrite coherency cell has been developed and used to enhance understanding of solid network formation during Mg alloy solidification. Combining thermal analysis and dendrite coherency measurements from the liquid-to-solid state, and microscopy on the as-cast microstructure provides a thorough description of salient solidification features and forms a basis for understanding liquid-to-solid processing behavior in Mg alloys. Alloys from all families of Mg cast alloys including several of the newly developed alloys have been studied using thermal analysis, dendrite coherency, and microscopy with the results presented here. The aim of this project is to form a coherent and complete encyclopedia of solidification features and pursuant as-cast microstructures from the full range of Mg casting alloys as a practical tool for foundry metallurgists and future researchers interested in alloy development. When practical, comparison of conventionally cast and high-pressure die cast microstructures is included.

10:40 AM
Industrial Sludge Investigations in AZ91D, AM60B and AM50A Die-Casting Operations: *Chris Patrick Corby*[1]; Ma Qian[1]; Nigel Jeffery Ricketts[2]; Rob Bailey[3]; [1]University of Queensland, CRC for Cast Metals Mfg. (CAST), Div. of Matls., Sch. of Engrg., Brisbane, QLD 4072 Australia; [2]CSIRO, Mfg. & Infrastructure Tech., 2643 Moggill Rd., Pinjarra Hills, Brisbane, QLD 4069 Australia; [3]Australian Magnesium Corporation, Covington, KY USA

Excess sludge generation is a costly problem facing the magnesium casting industry. With a view to address this economic concern, an investigation into the constituents of several sludge samples found in AM50A, AM60B and AZ91D die-casting operations was conducted. Chemical analysis from the literature suggests that the major proportion of sludge is entrapped Mg metal and oxide. Electron microscopy was used to confirm that the major chemical species in industrial die casting sludge samples were magnesium oxide, Mn-Al-Fe intermetallic particles and a high proportion of entrained metal. The iron content of the intermetallic phases present in the sludge was found to be very low, suggesting that the intermetallic particles in the sludge are mainly Mn-Al phases. Process parameters are discussed with a view to minimising sludge formation in magnesium die casting furnaces.

11:00 AM
Filling Characteristic of AZ91D Magnesium Alloys During EPC: *Shae K. Kim*[1]; Gue-Serb Cho[1]; Kyong-Whoan Lee[1]; Hyung-Ho Jo[1]; [1]Korea Institute of Industrial Technology, Advd. Matl. R&D Ctr., 472 KaJwa-Dong, Seo-Ku, Incheon 404-254 Korea

Magnesium alloys are gaining increased importance for automotive applications due to their low density and high strength-to-weight ratio. Diecasting is currently used for magnesium components for the excellent castability of magnesium alloys allowing very thin and complicated sections to be cast. With increasing the number of potential applicastions, however, other forming processes, especially, other casting processes should be considered and developed. The aim of this study is to develop a complete EPC (Expendable Pattern Casting) for magnesium alloys. The special attention will be given to metal-mold reaction and fluidity of AZ91D magnesium alloys during the process.

11:20 AM
Numerical Understanding of Forced Convection in Melt of Squeeze Casting Magnesium Alloy: *Alfred Yu*[1]; Naiyi Li[2]; Henry Hu[1]; [1]University of Windsor, Mech., Auto. & Matls. Engrg., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ford Motor Company, Mfg. Sys. Dept., Ford Rsch. Lab., Dearborn, MI 48121 USA

As a continuous effort on fundamental study of squeeze casting of magnesium alloys, a 3-D mathematical model has been developed to simulate the melt flow, heat transfer and solidification phenomena. In this paper, the developed model simulates the events of metal flow with various initial filling velocities during squeeze casting of magnesium alloys. The influence of forced convection resulting from initial filling velocities on heat transfer between the melt and the mold is examined. The temperature distribution in the squeeze casting during and after filling is also predicted. The significance of taking forced convection into consideration for precision of computing solidification patterns following the filling stage is discussed and highlighted.

Materials by Design: Atoms to Applications: Design for Mechanical Functionality I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA; Krishnan K. Sankaran, The Boeing Company, Phantom Works, St. Louis, MO 63166-0516 USA

Wednesday AM Room: 210B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Krishnan K. Sankaran, The Boeing Company, Phantom Works, St. Louis, MO 63166-0516 USA

8:30 AM Invited
The Design and Development of an Age Hardenable Aluminum Alloy for Moderate Temperature Application: *E. A. Starke, Jr.*[1]; B. M. Gable[1]; G. J. Shiflet[1]; A. W. Zhu[1]; [1]University of Virginia, Dept. of Matls. Sci. & Engrg., Charlottesville, VA 22904-4745 USA

This presentation will describe both the alloy design methodology and our most recent experimental results directed towards the development of an age hardenable wrought aluminum alloy for moderate temperature applications. The design process involves extensive empirical research, theoretical simulation and modeling, first principle atomistic calculations, calculated phase diagrams and quantitative analytical techniques. Our research focuses on the Al-Cu-Mg-(Ag) system and we have used the CALPHAD (CALculation of PHAse Diagrams) method to determine the phase field that gives the most promise for having q' and W precipitates, without the interference of the S phase. First principle total energy calculations using VASP (Vienna Ab initio Simulation Program) were conducted to evaluate the proposed crystal structures and to determine interfacial energies and elastic constants of the strengthening phases. The elastic strain energy associated with the formation of plate-like elemental clusters in the aluminum matrix was determined. These clusters may function as either precursors or non-precursors for the desired precipitates. Combined with the strain energy considerations, thermodynamic analysis of short-range order was employed to identify useful trace elements and deleterious impurities for the formation of the desired precipitates having a fine and uniform distribution. Due to the competitive nature of precipitation in this alloy system the relative volume fractions of precipitates on {111} and {100} habit plans can be modified by thermal-mechanical treatments. Our modeling and predictions are being verified by experimental measurements. The ultimate objective of this program is to streamline alloy design and, therefore, to aid in early insertion of new high performance materials into aerospace systems. Sponsored by the Air Force Office of Scientific Research under Grant S49620-01-1-0090.

9:00 AM
Atomistic Design of Ductile Transition-Metal Solid Solution Alloys: *Kwai S. Chan*[1]; [1]Southwest Research Institute, 6220 Culebra Rd., San Antonio, TX 78238 USA

A computational approach is applied to design ductile bcc transition-metal solid solution alloys on the basis of a recent discovery that the d + s electrons affect the Peierls-Nabarro (P-N) barrier energy and dislocation mobility. For designing compositions of ductile alloys, the number of d + s electrons per atom in the solid solution alloy are tailored to achieve three objectives: (1) increasing the dislocation line energy, (2) decreasing the shear modulus in the slip direction, and (3) reducing the P-N barrier energy. Applications of this atomistic method to designing ductile bcc Ti-, Nb-, and Mo-base solid solution alloys are illustrated and compared against experimental data. The results indicates that design of ductile transition-metal solid solution alloys can be achieved solely by manipulating the number of d + s electrons to increase dislocation mobility via a reduction of the P-N barrier energy. Furthermore, the atomistic approach provides insights on the roles of electronic bonding in solid solution toughening and embrittlement in transition metals. Material parameters and electronic bonding that affect brittle-to-ductile fracture transition in transition metals are identified and discussed in conjunction with experimental observations. Work supported by AFOSR through contracts F4962001-C-0016 and F49620-01-1-0547, Dr. Craig S. Hartley, Program Manager.

9:30 AM
Atomic Level Modeling for the Computer Design of Naval Steels: *Michael J. Mehl*[1]; Dimitrios A. Papaconstantopoulos[1]; Yuri Mishin[2]; [1]Naval Research Laboratory, Ctr. for Computational Matls. Sci., Code 6390, Washington, DC 20375-5000 USA; [2]George Mason University, Sch. of Computational Scis., Fairfax, VA 22030 USA

Computer modeling on the electronic and atomic levels offers a way of reducing the costs and accelerating the development cycle of Naval steels. This paper reports on our recent efforts in that direction. First-principles (LAPW/GGA) calculations have been performed for many ordered fcc and bcc-based compounds of the Fe-Ni system. The results of these calculations are used to construct tight-binding and angular-dependent atomistic potentials for the Fe-Ni system. The potentials are applied to calculate the structures, energies and equilibrium segregation at several grain boundaries in fcc Fe-Ni alloys (austenite). Mechanical properties of the grain boundaries are evaluated by modeling the process of decohesion. The observed relations between the grain boundary energies, cohesive strength, temperature and the alloy composition provide useful input for mesoscopic continuum simulations of plastic deformation and fracture of Naval steels. The ongoing efforts to extend this work to multicomponent steel alloys are discussed.

10:00 AM Break

10:15 AM
Micro- and Nano-Contacts: Multi-Scale Models and Experiments: *W. O. Soboyejo*[1]; Z. Zong[1]; J. Lou[1]; A. Widjaja[2]; E. Van der Giesen[2]; E. Bittencourt[3]; A. Needleman[4]; [1]Princeton University, MAE Dept./PMI, Olden St., Princeton, NJ 08544 USA; [2]University of Gronigen, Dept. of Appl. Physics, Gronigen The Netherlands; [3]University of Sao Paulo, Sao Paulo Brazil; [4]Brown University, Div. of Engrg., Providence, RI 02912 USA

This paper presents a multi-scale framework for the modeling of micro- and nano-contacts that are relevant to micro-electro-mechanical systems (MEMS) and microelectronic devices. The framework includes the use of discrete dislocations models for the modeling of contact-induced plasticity in the nano-scale regime. However, in the sub-micron and micron-scale regimes, local and non-local strain gradient plasticity models are used to model the deformation induced by sharp and blunt contacts. Predictions of indentation size effects and material pile-up are compared with experimental measurements obtained from Berkovitch, cube-cornered and Vickers indenters. The implications of the results are then discussed for contact-induced deformation in MEMS switches and magnetic storage devices.

10:45 AM
Micromechanics Study of Gamma-TiAl Material: *Sheng Chang*[1]; Dan Hong[1]; *Fu-Pen Chiang*[1]; [1]State University of New York, Dept. of Mechl. Engrg., Stony Brook, NY 11794-2300 USA

The knowledge of the micromechanical properties of gamma Titanium Aluminide is essential to the design of high performance gas turbine engines or low emission-high efficiency internal combustion engines. A novel experimental micromechanics technique SIEM (Speckle Interferometry with Electron Microscopy) was employed to examine the inter-lamellar deformation and the deformation within the lamellar colony of gamma Titanium Aluminide. SIEM is a micromechanics measurement technique that has a spatial resolution approaching to a few nanometers. It is able to perform the full field displacement mapping over a region of only several microns in diameter. A dog-bone specimen was tested under uniaxial tension and intercolony shear was found in the displacement contours. Another fatigue-cracked specimen was tested under 3-point bending load at 1000x magnification. The deformation fields around the crack tip were obtained. It shows that the crack propagation path tends to align with the crystal orientation.

11:15 AM
Atomistic Modeling of High-Temperature Structural Aliminides: *Y. Mishin*[1]; [1]George Mason University, Sch. of Computational Scis., 4400 Univ. Dr., MSN 5C3, Fairfax, VA 22030-4444 USA

Overview of our recent work on the atomistic modeling of ordered intermetallic compounds of the Ni-Al and Ti-Al systems. Atomic interactions in these systems are modeled by semi-empirical potentials fit to both experimental and first-principles data. The methodology includes a large variety of techniques ranging from harmonic lattice dynamics to molecular dynamics and Monte Carlo simulations. The properties studied include lattice characteristics (elastic constants, phonons, thermal expansion), point-defect properties, atomic diffusion, generalized stacking faults, dislocations, surfaces, grain boundaries, interphase boundaries, phase diagrams and so on. It is discussed how the output of such calculations can be used for the computer design of advanced intermetallics.

Materials Education to Revitalize the Workforce: Session I

Sponsored by: TMS, Public & Governmental Affairs Committee, TMS-Education Committee
Program Organizers: Reza Abbaschian, University of Florida, College of Engineering, Gainesville, FL 32611-6400 USA; Iver E. Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA

Wednesday AM Room: 217D
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

8:30 AM Keynote
Training for a High Tech Society: *Larry Keen*[1]; [1]North Carolina Community College System, Economic & Workforce Dvlp., 5001 Mail Service Ctr., Raleigh, NC 27699-5001 USA

The dynamic nature of the world's economies require organizations, states and nations to provide training and educational services that result in rapid adaptation to changes organizationally and technologically by an adaptable workforce in high-tech societies. The educational enterprises' conceptualization by which the opportunities available in promising disciplines are organized, promoted and delivered may rest with greater inter-institution collaboration that could lead to the enhancement of a high-tech workforce in a high-tech society. Potential solutions to inform and train for a high-tech society may be an answer to consider and may result in seamless training and educational systems to meet the need.

9:00 AM
Making Science Exciting for the Future Workforce: *Robert A. Childs*[1]; [1]Massachusetts Institute of Technology, Plasma Sci. & Fusion Ctr., 190 Albany St., NW21-109, Cambridge, MA 02139 USA

One of the perceived notions that trouble us today is that working in a science related field is somehow less glamorous and less financially rewarding than say a career as a doctor, lawyer or sports hero. We have to show the youth of today that there can be excitement and comfortable earnings in a career in science that is more personally rewarding than the image they have today. Moderate strides have been made in the last couple of decades to correct this notion but more needs to be done. This talk will show samples of work being done by scientific societies, universities and local school systems in the K-12 range that have been successful. We need to continue making the jobs available but we also need to raise the level of excitement in those future workers if we are going to fill those jobs from within.

9:30 AM
Career Resource Center for MSE: *Gerald L. Liedl*[1]; [1]Purdue University, Sch. of Matls. Engrg., W. Lafayette, IN 47907 USA

In response to a NAS study that indicated initial career decisions are made in the absence of realistic information about life in science and engineering professions, the Alfred P. Sloan Foundation funded a group of professional societies to generate information for their fields in the early 1990's. TMS in collaboration with other materials-oriented societies generated the Career Resource Center for MSE. Components of the Center included surveys to provide statistical information about professionals in the field, development of a career guidance booklet, a web site, and a career CD ROM. The evolution and utilization of the components of the Center will be discussed as well as the impact of the integrated Sloan Career Cornerstone web site on distribution of information about MSE.

10:00 AM Break

10:20 AM
MSE Teach: An Undergraduate Recruitment Tool: *Paul H. Holloway*[1]; Elliot Douglas[1]; [1]University of Florida, Dept. of Matls. Sci. & Engrg., Gainesville, FL 32611-6400 USA

A number of techniques are necessary to successfully recruit undergraduates to MSE, including university information directed towards the potential student. However, the student is in daily contact with her/his high school or community college teachers, and this provides another avenue for supplying information about a career in materials. Since 1995, we have organized a workshop on materials science and engineering for 6th-12th grade and community college science teach-

ers. Using a combination of tutorial lectures, typical applications, hands-on demonstrations, and laboratories conducted by graduate students and faculty, we have introduced these teachers to the fundamental and exciting topics in MSE. The teachers in turn have developed lesson plans and passed this excitement to their students, resulting in applications from 10835 undergraduates designating MSE as their degree choice. Details of the MSE Teach program will be discussed and illustrated.

10:50 AM
Materials Engineering Technology: The Role of Community Colleges in Preparing the Technical Workforce of the 21st Century: John McKay[1]; Michael Kenney[2]; Pat Pohar[1]; John Pridgeon[3]; [1]South Piedmont Community College, Monroe, NC USA; [2]ASM International, Matls. Park, OH USA; [3]Allvac (retired), Monroe, NC USA

US companies involved in manufacturing are experiencing both a shortage of qualified workers and a need to retrain existing personnel on new technologies. Both of these situations can easily be addressed through two-year college educational programs. Advantages of the Community College include proximity to the workplace, knowledge and understanding of the needs of adult learners, and strong community ties. This presentation will present a case study of the efforts of South Piedmont Community College in collaboration with a local employer, Allvac, and ASM International. Lessons learned, both positive and negative, of a unique degreeprogram will be highlighted.

Materials Processing Fundamentals: Aqueous Processing

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control 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: 212B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

8:30 AM
Study on the Thermodynamic Equilibrium of the Ni(II)-Ammonia-Carbonate Aqueous System and its Application to the Precipitation of Basic Nickel Carbonate Particles: *Guo Xueyi*[1]; *Huang Kai*[1]; *Zhang Duomo*[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

Based on the principles of simultaneous equilibrium and mass equilibrium, the thermodynamic equilibrium equations of Ni(II)-ammonia-carbonate aqueous system at ambient temperature were deduced theoretically and the thermodynamic diagrams of lg[Ni2+] versus pH at different solution compositions were drawn. It was founded by experiments that the obtained theoretical diagrams were useful to explain the formation mechanism of precipitation particles with different microscopic shapes. When pH of the solution below 7.0, the loose flocculation particles were produced due to fast coagulation which was the dominant growth mechanism on this condition, while pH above 7.0, because of the coordination of nickel ion with ammonia, the precipitation proceeded slowly accompanying with the release of Ni2+ from the complexes, which easily results in the formation of the dense spherical particles.

9:00 AM
Application of Chemical Pattern Recognition to the Preparation of Monodispersed NiO Precursor Particles by Homogeneous Precipitation: *Huang Kai*[1]; *Guo Xueyi*[1]; Zhang Duomo[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

Mono-dispersed NiO precursor particles were prepared by homogeneous precipitation in the presence of urea. Optimal discrimination plan method, one of the chemical pattern recognition techniques, was applied to analyzing the experimental data and the calculating results were visualized in the mapping figures, in which the different class of samples were divided distinctly and the corresponding semi-experienced mathematic model was deduced. The model can be well used to predict the relationships of the characteristics of the precipitated particles with operational parameters. It proved experimentally that the model fit well with the experimental results and it was quite effective to guide the process design.

9:20 AM
Study on the Thermodynamic Equilibrium of the Ni(II)-Ammonia-Oxalate Aqueous System and its Application to the Precipitation of Fine Nickel Oxalate Particles: *Huang Kai*[1]; *Guo Xueyi*[1]; *Zhang Duomo*[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Engrg., Yuelu Dist., Changsha, Hunan 410083 China

Based on the principles of simultaneous equilibrium and mass equilibrium, a series of thermodynamic equilibrium equations of the complex system of Ni(II)-ammonia-oxalate aqueous system at ambient temperature were deduced theoretically and the relationship of lg[Ni(II)] versus pH at different solution compositions was established quantitatively. From the thermodynamic diagrams, it was indicated that when pH less than 8.0, free Ni(II) was the dominant metal ion in the solution; while pH higher than 8.0, most of the nickel ions coordinate with the ammonia as the complex in the solution. The experimental results show that when pH in solution less than 8.0, the nickel oxalate particles in cubic morphology precipitated; while pH higher than 8.0, the needle-like nickel oxalate was obtained. Comparing the thermodynamic diagram with the experimental data, it was founded that the different existing forms of nickel ion led to the formation of the above-obtained two kinds of particles.

9:40 AM Break

10:10 AM
Viscosity and Drift Flux Analysis: *Ramiro Escudero*[1]; Francisco J. Tavera[1]; [1]Universidad Michoacana de San Nicolás de Hidalgo, Inst. de Investigaciones Metalúrgicas, Santiago Tapia 403, C.P. 58000, Morelia, Mich. México

Column flotation are enjoying renewed interest due to new applications such as de-inking of recycled paper, industrial effluents treatment, and de-oiling of water. According to reported results, the efficiency of a given dispersion process depends on the characteristics of the dispersed phase. In order to predict an appropriate gas holdup, bubble surface area flux, and bubble size for a given duty, a mathematical model known as Drift Flux Analysis is currently applied. Drift Flux Analysis assumes a constant dynamic viscosity of the continuous phase (1 grame/centimetre-second) not matter the changes as result of the pulp consistency or solids content. This paper shows the relevance of considering the real value of the dynamic viscosity in terms of the characteristics of a gas dispersion. Viscosity of water was varied by using a polymer and the bubble size, bubble surface area, and gas holdup were calculated through the Drift Flux Model.

10:40 AM
Phase Transformation Relationships in the CaSO4-H2SO4-H2O System: *Yuanbing Ling*[1]; George P. Demopoulos[1]; [1]McGill University, Dept. of Mining & Metallurgl. Engrg., 3610 Univ. St., Montreal, Quebec H3A 2B2 Canada; [1]McGill Univeristy, Metals & Matls. Engrg., 3610 Univ. St., Montreal, Quebec H3A2B2 Canada

As part of a boarder research topic, seeking the development of a cost-effective technology for direct production of a high value building/dental material, alpha-calcium sulfate hemihydrate, from sulfuric acid at atmospheric as opposed to elevated pressure conditions, some fundamental work has been done. This includes experimental monitoring of phase transformation reactions and thermodynamic analysis of the relationships among the various phases, namely, calcium sulfate dihydrate, hemihydrate and anhydrite in aqueous sulfuric acid solution at temperatures ranging from 20°C to boiling point. Account of these fundamental studies is presented in this paper.

11:00 AM
Leaching of Cu-Co Oxidized Ore and Concentrate as Alternative to Conventional Sulphidization: *Eduardo Patino*[1]; [1]University Arturo Prat, Engrg. Dept., Av Arturo Prat 2120, Iquique, 57 Chile

The Luiswishi deposit belongs to the copper-cobalt oxides deposit of the Southern Group of the Katangian Copper Belt and is operated by a joint venture between EGMF (Enterprise Générales Malta Forrest) and Geacamines (Générale des Carrières et des Mines). The coopercobal ore is beneficiated by the sulphidization process at the NCK (New concentrator of Kipushi) at Kipushi. Sulphidization followed by flotation with sulphydryl collectors is a common practice for the treatment of oxidized base metals ores. However, this process is very complex and its application at the industrial scale is quite sensitive. In central Africa it is also a cost intensive treatment as the sulphidizing agent has to be imported. A new laboratory research program was carry out to find an alternative route of treatment using hydrometallurgical approach. Ore is composed by malachite, chrysocol, tenorite, and

heterogenite with grades of 1.98% and 1.51% in copper and cobalt, respectively. The study was focussed in leaching techniques using sulphuric acid and ferrous iron as reductant to extract copper and cobalt, respectively. Two methods were compared: agglomerate-curate-leach of ore and ore Geocoat-ed with conventional concentrate. Results obtained reach copper and cobalt extractions up 80%.

Metals for the Future: Structural Materials

Sponsored by: TMS
Program Organizers: Manfred Wuttig, University of Maryland, Department of Materials & Nuclear Engineering, College Park, MD 20742-2115 USA; Sreeramamurthy Ankem, University of Maryland, Department of Material & Nuclear Engineering, College Park, MD 20742-2115 USA

Wednesday AM Room: 215
March 17, 2004 Location: Charlotte Convention Center

Session Chair: K. L. Murty, North Carolina State University, Raleigh, NC 27695-7909 USA

8:30 AM Opening Remarks by Dr. Haworth

8:40 AM Invited
Research on Structural Materials: Quo Vadis?: *Ronald Gibala*[1]; [1]University of Michigan, Dept. of Matls. Sci. & Engrg., 2300 Hayward, 2026 H.H. Dow Bldg., Ann Arbor, MI 48109-2136 USA

In spite of irregularly appearing discussions and opinions to the contrary, the future of applications of and research on structural materials, especially metals, appears to be substantial. Examination of data from several industrial sectors for applications and from funding agencies for applied and fundamental research demonstrates that, at worst, a few specific areas of research on metallic materials have declined and a few others may have stabilized. In general, however, the application of new techniques - experimental, theoretical, computational - offers many innovative opportunities for research on all types of structural materials. This talk will amplify on these points, offer several examples of current and exciting research on structural materials (at least to the author), and suggest areas of future research opportunities.

9:10 AM Invited
Recent Developments on the Low Temperature Creep Deformation Behavior of Titanium Alloys: *Sreeramamurthy Ankem*[1]; [1]University of Maryland, Matls. Sci. & Engrg., Stadium Dr., Bldg. 090, College Park, MD 20742-2115 USA

Recent investigations revealed that alpha beta and beta titanium alloys could creep at low temperatures, such as room temperature. The factors that effect creep deformation include stress level, grain size, morphology of phases, and stability of phases. In this presentation, these developments will be reviewed and the future challenges that must be addressed in this area will be outlined. This work is being supported by the National Science Foundation under grant number DMR-0102320.

9:40 AM Invited
Morphological Control and Applications of Nanoporous Gold: *Jonah D. Erlebacher*[1]; [1]Johns Hopkins University, Matls. Sci. & Engrg., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

The morphological evolution and applications of nanoporous gold (NPG) will be reviewed. NPG is a material with nanometer-scale porosity formed by selective dissolution of silver from silver/gold alloys. NPG has a huge surface area to volume ratio, and is chemically modifiable so as to change both its morphology and also the chemical reactivity of its surface. We will specifically address methods to create NPG with a hierarchy of pore sizes (i.e., large channels with porous sidewalls), methods to coat the surface of NPG with catalytically active nanoparticles, and applications for surface modified-NPG as fuel cell electrodes and sensor components.

10:10 AM Break

10:25 AM Invited
Nanostructured Intermetallic Alloys - Annealing Behavior, Microstructural Control and Influence of Scale in Reversibly Ordering Systems: *Jorg M.K. Wiezorek*[1]; [1]University of Pittsburgh, Matls. Sci. & Engrg., 848 Benedum Hall, Pittsburgh, PA 15261 USA

The annealing behavior of intermetallics after cold-deformation represents an underdeveloped field of physical metallurgy. This research uses combinations of measurements by DSC, magnetometry and mechanical testing together with microstructural observations by SEM, TEM and XRD to study the annealing behavior of deformed binary intermetallics that undergo the ordering transformation in the solid state (reversibly ordering systems). The L1o- and L12-ordering Fe-Pd alloys used here are excellent model systems for many other intermetallics and offer attractive properties for permanent magnet applications. Furthermore, utilizing severe plastic deformation (SPD) processing bulk nanostructured Fe-Pd alloys will be fabricated, thereby enabling the systematic investigation of microstructural scale effects. The main goals of this effort are to: a) Develop physical mechanism based descriptions of the annealing behavior; b) Fabricate nanostructured bulk Fe-Pd alloys; c) Control alloy microstructures across the length scales; d) Determine processing-microstructure-property relationships for the Fe-Pd intermetallics. Thus, knowledge pertaining to this important area of physical metallurgy will be advanced.

10:55 AM Panel Discussion with R. Gibala, H. Fraser, S. Ankem and K. L. Murty

Multiphase Phenomena in Materials Processing: Session I

Sponsored by: Extraction & Processing Division, Light Metals Division, Materials Processing and Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee, MPMD-Solidification Committee
Program Organizers: Ben Q. Li, Washington State University, School of Mechanical and Materials Engineering, Pullman, WA 99164-2920 USA; Stavros A. Argyropoulos, University of Toronto, Department of Materials Science and Engineering, Toronto, Ontario M5S 3E4 Canada; Christoph Beckermann, University of Iowa, Department of Mechanical Engineering, Iowa City, IA 52242 USA; Bob Dax, Concurrent Technologies Corporation, Pittsburgh, PA 15219 USA; Hani Henein, University of Alberta, Edmonton, AB T6G 2G6 Canada; Adrian S. Sabau, Oak Ridge National Laboratory, MS-602, Oak Ridge, TN 37831-6083 USA; Brian G. Thomas, University of Illinois, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Srinath Viswanathan, Sandia National Laboratories, Albuquerque, NM 87185-1134 USA

Wednesday AM Room: 218B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Ben Q. Li, Washington State University, Sch. of Mech. & Matls. Engrg., Pullman, WA 99164-2920 USA; Stavros A. Argyropoulos, University of Toronto, Dept. of Matls. Sci. & Engrg., Toronto, Ontario M5S 3E4 Canada

8:30 AM Invited
Application of an Online Iterative ECT Image Reconstruction Method in Multiphase Flow Measurement: *Shi Liu*[1]; Fan Jiang[1]; Haigang Wang[1]; [1]Chinese Academy of Sciences, Inst. of Engrg. Thermophysics, Beijing 100080 China

Electrical capacitance tomography (ECT) has been applied in imaging multiphase distributions since the 90's of the last century. The most widely used simplistic image reconstruction method, namely linear-back-projection (LBP), is fast (e.g 140 frames per second) but often produce unsatisfactory results. Therefore, enhanced image reconstruction methods, such as iterative methods, are required to improve the quality of the images. While iterative image reconstruction algorithms may produce improved tomographic images, they are time-consuming and can be used off-line only, which is not a favourable feature for monitoring rapidly changing industrial processes. This paper describes the development of a new image reconstruction algorithm and its applications in gas-solid two phase flows. For the new algorithm, firstly a coefficient matrix is generated through an off-line iteration sequence. The matrix is then used for on-line image reconstruction, in the same way as a sensitivity map is used in the popular LBP algo rithm. This new algorithm can produce similar quality images to the Landweber iteration algorithm with a constant sensitivity map, but at the same speed as the LBP. An optimal step length has been incorporated into the new algorithm for improved convergence. The new algorithm was evaluated by both simulation and experiment. The new scheme has been applied to the online measurement of solids distribution in square circulating fluidized beds (CFBs), in gas-solids wavy flow in pipes, and in a cyclone gas-solids separator. From the measurement of solids distribution in CFBs in different flow regimes,

the solids concentration profile, temporal variation of average solids concentration and frequency spectra are obtained. The results reveal noticeable solids fraction in the central region of the CFBs, which agrees with the nature of bubbling or turbulent fluidization in the bottom zone but could not be reconstructed by LBP. Meanwhile, closely distributed multiple voids (gas bubbles) are distinguished that is not pos sible for LBP. Solids concentrations in the conical part of a cyclone separator are also measured. The cross-correlation technique was combined with the solids concentration measurements to obtain the flow rate through the dip-leg of the cyclone. The same technique applied in a dense phase pneumatic transport pipe provides the solids concentration, and the flow rate of a wavy gas-solids flow. The capability of the online iterative ECT method in providing much improved instantaneous spatial and temporal information on two-phase flow is unique, and can be of great value in the study of multiphase processes. Also, the obtained data are valuable references for further studies.

8:55 AM
Study on the Air Flow and Heat Transfer in Gravel Embankments in the Permafrost Areas Between Qinghai and Tibet: *Jiang Fan*[1]; [1]Chinese Academy of Sciences, Inst. of Engrg. Thermophysics, PO Box 2706, Beijing 100080 China

Winter-time natural convection in open-graded gravel embankments has been suggested as a technique which can be used to provide passive cooling and thereby avoid thaw-settlement of roadways located in permafrost areas. Heat transfer in open-graded gravel embankment is studied using numerical simulation, based on unsteady two-dimensional momentum and energy equations. Two different models, one being porous media model, the other gravels model in which the embankment is composed of stones and air, have been adopted to investigate the temperature and velocity fields in embankment. Simulation results show that in summer, a gentle clockwise rotation of the pore air extending throughout most of the embankment. The pore-air motion is very weak which results in relatively straight horizontal isotherm lines. And heat transfer is mainly maintained through conduction. Whereas in winter, pore-air velocities are higher and multiple vortexes are formed in the embankment. Natural convection then becomes the dominant influence on the isotherm shapes within the embankment. As a result of low ambient temperatures acting on the embankment surface during winter months, an unstable air density gradient develops within the embankment. This convection increases the heat flux out of the embankment. Therefore, the winter-time convection can lower the temperature of the foundation soil beneath the open-graded embankment. In addition, stones within the embankment with different dimensions have been analyzed and compared using the gravel model. It shows that in winter, Ö200mm stones lead to stronger vortexes in the embankment compared with that of Ö60mm stones. Consequently, the zone of low-temperature beneath the embankment is broadened.

9:15 AM
A Two-Phase Flow Model for Particle-Gas Flows and Comparison with Experimental Measurements: W. Song[1]; *Ben Li*[1]; [1]Washington State University, Sch. of Mech. & Matls. Engrg., Pullman, WA 99164 USA

Many materials processing systems involve the gas-particle two phase flow phenomena. In this presentation, a two-phase flow model is developed for spouting gas-particle reactors used for chemical vapor deposition processes. A physical model is also developed and the results are compared with the numerical model predictions. The predictions of various gas-particle flow models such as gas-kinetic models, granular flow models and two phase Eulerian models are presented and their advantages and limitations in the gas-particle flow modeling are discussed in light of the comparison with the measurements taken on the physical model.

9:35 AM
Optimization of Flow Modifiers in McGill Heat Pipes: *Pietro Navarra*[1]; Hujun Zhao[1]; Frank Mucciardi[1]; [1]McGill University, Dept. of Mining, Metals & Matls. Engrg., 3610 Univ., Montreal, Quebec H3A 2B2 Canada

A heat pipe is a two-phase heat exchanger with a very high effective thermal conductivity. Such devices were developed extensively in the 1960's and have become standard heat extraction devices in many fields. The metallurgical industry has traditionally been beyond the operational range of these devices as the heat fluxes encountered are often above the critical heat flux, causing film boiling within conventional heat pipes. Recent research at McGill University has led to a new type of heat pipe capable of overcoming the film boiling limitation encountered at high heat fluxes. Several industrial-scale tests have proven the commercial viability of the new McGill Heat Pipe in applications such as oxygen lance injection, furnace equipment cooling and permanent mould casting. A heat pipe consists of three main components: a condenser and an evaporator section, and a working fluid contained within. Condenser design is somewhat trivial, whereas the evaporator section encompasses the key behind McGill Heat Pipes. The evaporator section contains a flow modifier which generates a centrifugal force that suppresses the formation of a vapour film at the wall/fluid interface during operation. Thus the "swirler" flow modifier is the key to extending the operational range of heat pipes in terms of maximum heat flux capability. This paper summarizes a study to model fluid flow within the evaporator section of McGill Heat Pipes and optimize the flow modifier design parameters to attain the maximum critical heat flux.

9:55 AM Break

10:10 AM
An Integrated Model for Microwave Heating of Ceramics and Dielectric Materials: *Y. Huo*[1]; *Ben Q. Li*[1]; Ravindra Akarapu[1]; J. Tang[1]; [1]Washington State University, Sch. of Mech. & Matls. Engrg., Pullman, WA 99164 USA

An integrated model is developed to represent the three-dimensional electromagnetic wave propagation and thermal phenomena during microwave heating of ceramics and dielectric/biological materials. The model development is based on the edge finite element formulation for electromagnetic fields and the node finite element formulation for thermal phenomena. Results are presented for both simple systems and complex real processes used for ceramic processing and biomaterials processing.

10:30 AM
Undercooling and Demixing of Cu-Co Melts in the TEMPUS Facility During Parabolic Flight: *M. Kolbe*[1]; S. Reutzel[2]; A. Patti[3]; I. Egry[1]; L. Ratke[1]; D. M. Herlach[1]; [1]Institute of Space Simulation, German Aeros. Ctr., DLR, D-51170 Köln Germany; [2]Ruhr-University Bochum, Inst. of Experimental Physics IV, D-44780 Bochum Germany; [3]Royal Melbourne Institute of Technology, Melbourne Australia

Cu-Co has a metastable miscibility gap in the region of the undercooled melt. Undercooling of the melt below the binodal leads to separation of the melt into a Co-rich L1-phase and a Cu-rich L2-phase. Experimentally, undercooling into the metastable miscibility gap can be realised by means of electromagnetic levitation (EML), processing in drop tube (DT) and by melt flux embedding. As undercooling is large in these experiments, the velocity of the solidification front is high and the microstructure is frozen in instantaneously. Samples have been undercooled and solidified in the TEMPUS-facility (containerless electromagnetic processing under weightlessness) during parabolic flight under reduced gravity conditions. Microstructures of solidified samples have been analysed and compared to those processed under terrestrial conditions. The influence of convection on phase growth is much less in the TEMPUS-facility than in terrestrial processing: The microstructure shows spherical droplets of the minority phase, statistically dispersed in the majority phase.

10:50 AM
Effects of Magnetic Field and Internal Radiation on Flow and Solidification of Oxide Melts: Yan Shu[1]; *Ben Q. Li*[1]; Kelvin Lynn[1]; [1]Washington State University, Sch. of Mech. & Matls. Engrg., Pullman, WA 99164 USA

This paper discusses the effects of magnetic field and internal radiation on the melt flow and solidification morphology during solidification processing of oxide crystals for optoelectronic and remote sensing applications. The numerical solution of the integral differential equation characterizing the internal radiation and the magnetohydrodynamic equations describing the magnetic and transport phenomena is obtained by applying the combined discontinuous and continuous finite element method. The results show that both internal radiation and an externally applied magnetic field can have important effects on the melt flow and solidification behavior during the melt processing of oxide materials.

11:10 AM
Influence of an External Electric Field Applied During the Solution Heat Treatment of the Al-Mg-Si Alloy AA 6022: Kang Jung[1]; Hans Conrad[1]; [1]North Carolina State University, Matls. Sci. & Engrg. Dept., Raleigh, NC 27695-7907 USA

The effects of an external dc electric field E = 5 kV/cm applied during solution heat treatment (SHT) of AA 6022 on the resistivity r and hardness H following quenching were determined. Both r and H increased with E. The results are analyzed in terms of the influence of the field on the enthalpy deltaHs and entropy deltaSs of solution and the constants relating r and H to the concentration of Mg2Si in solution.

11:30 AM
Peculiarities of Phase Creating in the Ag-Fe-Graphite Compositions During Processing by Electric Current: *O. I. Raychenko*[1]; V. P. Popov[1]; O. V. Derev'yanko[1]; [1]IPMS, NAS of Ukraine

Two series of experiments with powder mixtures Ag-graphite and Ag-Fe-graphite were performed. Equipment for experiment was installation for electrodischarge sintering ERAN 2/1. The processing was carried out in moulds of the dense graphite and of the alloy on Ni-Cr basis. The objects of processing were 1)graphite powder cladded by Ag, and 2)cladded graphite powder-Fe. During processing the metal components in compositions were solid or melted. Behavior of melted metal happens under electromagnetic forces. Addition of Fe results in hardening.

Nanostructured Materials for Biomedical Applications: Session V

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA; J. Michael Rigsbee, North Carolina State University, Department of Materials Science and Engineering, Raleigh, NC 27695-7907 USA; Xinghang Zhang, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Wednesday AM Room: 219A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Xinghang Zhang, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Marc Meyers, University of California, Mech. & Aeros. Engrg., La Jolla, CA 92093-0411 USA

8:30 AM Invited
Nanostructured Calcium Phosphates: Opportunities and Challenges for Biomedical Applications: *Prashant Nagesh Kumta*[1]; Charles Sfeir[2]; Daiwon Choi[1]; Sarah Petricca[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 4309 Wean Hall, 5000 Forbes Ave., Pittsburgh, PA 15213 USA; [2]University of Pittsburgh, Dept. of Oral Medicine & Pathology, 574 Salk Hall, 3501 Terrace St., Pittsburgh, PA 15261-1964 USA

Nanotechnology is a revolutionary area that has impacted several areas of materials science. Since the discovery of apatite and their close structural similarity to natural bone, calcium phosphates (CaP) have become synonymous to natural bone. Hydroxyapatite (HA) is the most well-known CaP studied for biomedical applications. Engineering bone at a defective site is largely dependent on the material displaying favorable characteristics promoting growth of healthy osseous tissue. The higher reactivity of nano-sized materials has led to significant activity in the synthesis and design of nanostructured materials. Our research has focused in the development of novel chemical and mechanochemical strategies for synthesizing stoichiometric, nanostructured HA for a number of bone tissue engineering applications ranging from scaffolds to potential bone sensor as well as novel carriers of plasmid DNA for gene delivery. Results of these studies and the potential biomedical benefits and challenges of nanostructured HA will be presented.

9:15 AM Invited
A Novel Technique for Processing Functionally Graded HA Coatings: *Afsaneh Rabiei*[1]; [1]North Carolina State University, Mech. & Aeros. Engrg. & Biomed. Engrg. Depts., 2417 Broughton Hall, Campus Box 7910, Raleigh, NC 27695-7910 USA

A new generation of calcium phosphate coatings for dental implants, in which film crystallinity and structure is optimized, for enhanced bioactivity, osseointegration, and adhesion for promotion of the coating lifetime has been developed. The osseointegration provides mechanical stability to an implant in situ, minimizes motion-induced damage to surrounding tissues, and is imperative for the clinical success of bone implants. In this manner, the health relatedness of this project is to increase bonding between an implant and juxtaposed bone so that a patient who has received joint or dental replacement surgery may quickly return to a normal active lifestyle. Furthermore, the present study aimed to increase the service-life of an orthopedic material by creating materials that form a strong, long lasting, bond with juxtaposed bone. A functionally graded Hydroxyapatite coating with graded Crystallinity and grain size has been produced using Ion Beam Assisted Deposition with insitu heat treatment method. HA coating shows larger grain size and Crystallinity at the interface of the coating with the substrate. The Crystallinity and the grain size decreases towards the surface of the coating. Both early and long-term bone responses have been assessed in small rat models, both prior to and after prosthesis loading. The desire to introduce this innovative technology to the field of dental implantology is based on two primary advantages of the process over existing technology used to apply HA coatings to implants. First, the chemical composition, Crystallinity and grain size of the applied coating can be precisely controlled over a wide range of values, without the need for post deposition annealing. Second, the HA coatings can be deposited as thin films from 1 to 5μm thick, much thinner than coatings applied using plasma spray technology. Thinner coatings can provide a higher interfacial strength and better fracture resistance than thicker plasma spray coatings. Additionally, this processing technique is causing less residual stresses at the interface of the coating and the substrate that will lead to a better adhesion bonding.

10:00 AM Cancelled
Synthesis of Hydroxyapatite/Gelatin Nanocomposites for Orthopedic Applications and Their Properties

10:45 AM Invited
Effects of Diamond Like Carbon Coating and Surface Treatment on Bioactivity of Ti-6%Al-4%V: *Janet Hamipikian*[1]; S. N. Dunham[1]; J. L. McKillip[1]; B. F. Bell[1]; D. Scholvin[1]; J. Hampikian[1]; Roger J. Narayan[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

In recent years there has been a growing need for biomedical implants with excellent surface properties. Titanium alloys have been of particular interest due to their high strength, wear resistance, and biocompatibility. Several surface treatments of Ti-6%Al-4%V alloy are presented in this work. These coatings have been characterized using SEM, XRD, Raman spectroscopy, and simulated body fluid studies.

11:30 AM Plenary
Nanostructured Materials for Orthopaedic/Dental Applications: *Rena Bizios*[1]; R. W. Siegel[2]; P. M. Ajayan[2]; L. S. Schadler[2]; [1]Rensselaer Polytechnic Institute, Dept. of Biomed. Engrg., 110 8th St., Troy, NY 12180-3590 USA; [2]Rensselaer Polytechnic Institute, Depts. of Matls. Sci. & Engrg., & Rensselaer Nanotech. Ctr., Troy, NY 12180-3590 USA

New tissue formation in vitro and in vivo can be enhanced by biomaterials designed with properties similar to those of physiological bone, which is characterized by ceramic grains in the nanometer range connected by polymer. It is now possible to design and formulate nanostructured ceramic/polymer composites, which exhibit improved cytocompatibility and mechanical behavior that simulate those properties of natural bone. Nanoceramics (specifically, alumina, titania, and hydroxylapatite), composites of these nanoceramics with polymers (specifically, poly(L-lactic) acid and poly(methylmethacrylate)), as well as composites of poly(L-lactic) acid and carbon nanotubes were evaluated using cellular in vitro models. The results provide evidence that, compared to pure ceramics, nanoceramic composites exhibit improved mechanical properties and that carbon-nanotube containing composites can be successfully used to expose osteoblasts (the bone-forming cells) to electrical stimulation. The most significant result, however, was that all of the nanostructured materials tested promote osteoblast functions pertinent to new bone formation. Thus, nanostructured materials can promote osseointegration, a critical requirement for the clinical success of orthopaedic/dental implants in vivo. This research exemplifies alternative strategies and novel approaches that, although extremely promising, remain as yet unexplored for bone regeneration purposes and in tissue engineering applications. This work was supported by Philip Morris USA and the Nanoscale Science and Engineering Initiative of the National Science Foundation under NSF Award No. DMR-D117792.

Phase Stability, Phase Transformation, and Reactive Phase Formation in Electronic Materials III: Session V

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

Program Organizers: C. Robert Kao, National Central University, Department of Chemical and Materials Engineering, Chungli City 32054 Taiwan; Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu 300 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: 214
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Sinn-Wen Chen, National Tsing-Hua University, Dept. of Chem. Engrg., Hsin-chu 300 Taiwan; Eric J. Cotts, Binghamton University, Dept. of Physics & Matls. Sci., Binghamton, NY 13902 USA

8:30 AM Invited
Evolution of the Microstructure of Pb Free Solder Joints: *Eric J. Cotts*[1]; Lawrence P. Lehman[1]; Lubov Zavalij[1]; Robert Kinyanjui[1]; [1]Binghamton University, Physics & Matls. Sci., Sci. 2, Vestal Pkwy. E., PO Box 6000, Binghamton, NY 13902 USA

A study of how the adoption of Pb-free solders affects manufacturing processes for electronics packages is in progress. Focus is on issues affecting the mechanical properties of solder joints, to facilitate the development of reliability models. The composition and microstructure of solder joints are characterized as a function of solder joint metallurgy and heat treatment, including reflow schedule and subsequent annealing treatments. Solder joints are constructed in ball-on-substrate geometries, annealed either in a calorimeter or by standard reflow techniques. The evolution of the microstructure of solder joints is determined by optical microscopy and scanning electron microscopy. We characterize grain and precipitate sizes and orientations as a function of cooling rate and composition. Our goal is to characterize the effect of thermal anneals and associated microstructures on joint reliabilities. Support from the National Science Foundation, DM0218129 is gratefully acknowledged.

8:50 AM Invited
Effects of Phosphorus Content on the Phase Transformation and Stress Evolution During the Reaction Between Electroless Ni(P) and Sn Film: J. Y. Song[1]; Y. C. Sohn[1]; *Jin Yu*[1]; [1]KAIST, Ctr. for Elect. Pkgg. Matls., MS&E, 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701 Korea

Electroless Ni(P) has been widely used as under bump metallization (UBM) for low cost flip chip technology because of its merits such as maskless process and slow reaction rate, etc. In this study, the reactions between electroless Ni(P) and Sn film were investigated by varying phosphorous content (low, medium and high) in the Ni(P) film. The phase transformation of the Ni(P) film during the reactions was examined by thermal analysis using differential scanning calorimetry. Also, the intrinsic stress evolutions, which accompanied the amorphous-crystalline phase transformation and the intermetallic compound (IMC) formation between Ni(P) and Sn, were investigated by conducting the isochronic and isothermal heat treatment and measuring the curvature change of the Si substrate. Results indicate that: 1) solder reaction facilitated phase transformation of Ni(P) into Ni3P and Ni by reducing the transformation temperature and released heat, 2) isochronic thermal cycling tests showed that formation of Ni3Sn4 and Ni3P out of Ni(P) and Sn films produced tensile stresses for the high P content films, but compressive stresses for the low P content films, 3) stress evolution caused by IMC formation was larger than that produced by Ni(P) crystallization, 4) thick and dense Ni3Sn4 compound with strong (111) texture formed on nanocrystalline Ni(low P) compared to that formed on amorphous Ni(high P).

9:10 AM Invited
The Effects of Bath Composition on the Morphologies of Electroless Nickel UBM on Al I/O Pad: *Jae-Ho Lee*[1]; Jinsoo Bae[1]; Ingun Lee[2]; Tak Kang[2]; Namseog Kim[3]; Seyong Oh[3]; [1]Hong-Ik University, Dept. of Met. Engrg. & Matl. Sci.; [2]Seoul National University, Div. of Matls. Sci.; [3]Samsung Electronics Co., Div. of Pkg. Dlvp.

For the flip chip packaging, the interests on UBM have been increased. Even though lead free solder bumps were obtained successfully, the failure in flip chip could be occurred on the interface of pad and bump if the interphacial regions were not well prepared. The adhesion of nickel on aluminum pad is very important in reliability of bump. The electroless nickel plating on zincated aluminum pad was investigated. The nickel deposition rate with bath composition and operating conditions were measured. The surface morphlogies of electroless plated nickel were influenced by the nature of zinc on aluminum. The deposition potential with time was also observed.

9:30 AM
Phase Equilibria of the Ag-Sn-Cu-Ni Quaternary System at the Sn-Rich Corner: *Sinn-wen Chen*[1]; Cheng-An Chang[1]; [1]National Tsing-Hua University, Chem. Engrg. Dept., #101, Sec. 2, Kuang-Fu Rd., Hsin-Chu, Taiwan 300 Taiwan

Knowledge of phase equilibria of the Sn-Ag-Cu-Ni quaternary system at the Sn-rich corner is important for the understanding of the interfacial reactions at the Sn-Ag-Cu/Ni contacts which are frequently encountered in the recent microelectronic products. Various Sn-Ag-Cu-Ni alloys were prepared and equilibrated at 250°C. The alloys were then quenched and analyzed. The existed phases were determined both by metallography and compositional analysis. No quaternary phases were found. The phase equilibrium relationship was proposed from the experimental results and the 250°C isothermal sections of the three constituent ternary phases, Sn-Ag-Cu, Sn-Ag-Ni, and Sn-Cu-Ni. Since there are no ternary phases in all these three systems, all the compounds are in fact binary compounds with various solubilities of the other two elements.

9:45 AM
Phase Stability of Sn-Cu Intermetallic Compounds Upon Current Stressing: *Ching Jung Yang*[1]; Ying Chao Hsu[1]; Chia Hui Lin[1]; Chih Chen[1]; [1]National Chiao Tung University, Dept. of Matls. Sci. & Engrg., 1001 Ta Hsueh Rd., Hsinchu Taiwan

The evolution of Sn-Cu intermetallic compound (IMC) within the eutectic SnPb solder was studied under the current density of 1.0 -~ 10^4 A/cm^2 in the temperature range from room temperature to 100°. The measurement of growth rate and grain size change of IMC indicated the dependence on temperature effect, current density, and electric polarity. The contribution of electromigration to the microstructure change of IMC is emphasized in our study.

10:00 AM
Effect of Intermetallic Compound Formation on Electrical Properties of Cu/Sn Interfaces During Thermal Treatment: *Chien-Neng Liao*[1]; *Chung-Ting Wei*[1]; [1]National Tsing Hua University, Matls. Sci. & Engrg., 101, Kuang Fu Rd., Sec. 2, Hsinchu 300 Taiwan

The influence of intermetallic compound (IMC) formation on electrical properties of Cu/Sn interfaces is investigated using an in-situ electrical probing method. A Cu/Sn bilayer thin film was deposited on thermally oxidized silicon wafers using a sputtering and thermal evaporation method, respectively. The resistance of the bimetallic films was measured while heating the specimen at a fixed ramping rate from 25°C to 225°C. It is found that the resistance change of the specimen depends on the formation of Cu6Sn5 and Cu3Sn phases during thermal treatment. The activation energy of Cu-Sn IMC formation is found to be 1.1 eV. The micro-structural and compositional evolution of the Cu/Sn thin film couple was studied by Rutherford Backscattering Spectrometry, x-ray diffraction, scanning electron microscopy, secondary ion mass spectrometer, respectively. In addition, a lithographically-patterned structure is also fabricated to investigate the reactions of solder/UBM thin films during electrical stressing.

10:15 AM Break

10:30 AM Invited
Interaction of Ag-In-Sn Solders with Pd Contacts: A Phase Diagram Approach: *Herbert Ipser*[1]; Jan Vrestal[2]; Ales Kroupa[3]; [1]University of Vienna, Inst. f. Anorganische Chemie, Waehringerstr. 42, Wien A-1090 Austria; [2]Masaryk University, Fac. of Scis. - Dept. of Theorical & Phys. Chmst., Kotlárská 2, Brno CZ-61137 Czech Republic; [3]Czech Academy of Sciences, Inst. of Physics of Matls., Zizkova 2, Brno CZ-61662 Czech Republic

The use of lead-containing solders will be ruled out within the European Union by July 1, 2006. Currently, Sn-Ag and Sn-Ag-Cu are

the leading candidates as lead-free solder materials, however, it will be of interest to search for alternative materials. One possible solution - at least for special applications - could be Sn-Ag-In alloys where the melting temperature can be tailored over a considerable range by varying the composition. In order to understand the interaction of such Sn-Ag-In solders with contacts containing Pd, the quaternary phase diagram Ag-In-Pd-Sn should be well known, at least in the part rich in In and Sn. As a first step toward this goal, the ternary systems In-Pd-Sn and Ag-In-Pd were investigated by means of X-ray diffraction (XRD), electron probe microanalysis (EPMA), and differential thermal analysis (DTA). The corresponding experimental results, together with all available thermodynamic information from literature, were used as input data for CALPHAD-type optimizations.

10:50 AM Invited
Formation and Characterization of SnBi and Bi Coated Solder Alloy: Jae-sik Lee[1]; Woong-ho Bang[2]; *Jae-Pil Jung*[1]; Kyu-hwan Oh[2]; Norman Zhou[3]; [1]University of Seoul, Matls. Sci. & Engrg., Seoul Korea; [2]Seoul National University, Matls. Sci. & Engrg., Seoul Korea; [3]University of Waterloo, Mech. Engrg., Waterloo, Ontario Canada

Electroplating method was used to form SnBi and Bi coated solder alloy. To produce a uniformed SiBi alloy, applied current density, bath temperature and stirring rate were carefully controlled. The compositions of plated SiBi layer was investigated by EPMA. Soldered Samples were tested to evaluate Microstructures and characteristics of intermetallic compounds. Test temperatures were 200C, 220C, and 250C. Further investigation by EPMA area mapping to study Bi distribution on the intermetallic compounds and inner solder, which affects mechanical and thermal property of Solder, was performed after soldered at 220C. According to SEM observation, coated solder at 2 and 4A/dm2 showed intermetallic compounds developed well on the interface of Cu plate at 200C, due to abundant Bi amount in the plated solder where forming transient liquid phase bonding. Bi was not observed on the intermetallic compounds and inner solder, and it implies that the solder was homogenized after soldering.

11:10 AM
A Study of Eutectic Sn-Pb Solder Wetting Behavior on Cu Strips Under Thermal Stressing: *Chien-Neng Liao*[1]; *Wen-Tai Chen*[1]; [1]National Tsing Hua University, Matls. Sci. & Engrg., 101, Kuang Fu Rd., Sec. 2, Hsinchu 300, Taiwan China

The wetting reaction between the eutectic Sn-Pb solder and copper metallization has been an important topic owing to academic and industrial research interests. In this study we will investigate the wetting behavior of eutectic Sn-Pb solder on a patterned Cu strip that is subjected to a thermal gradient. The non-uniform temperature distribution on the Cu strip is generated by applying an electric current through a fractional segment of the Cu strip. The preliminary results show that the capillary flow direction and moving distance of the eutectic solder depend on the geometry of the Cu strip and can be modulated by the electric current applied. The details of the testing structure and experimental results will be presented in this paper. The "thermo-migration" phenomenon of solder on Cu metallization will also be discussed.

Phase Transformations and Deformation in Magnesium Alloys: Creep Deformation

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizer: Jian-Feng Nie, Monash University, School of Physics and Materials Engineering, Victoria 3800 Australia

Wednesday AM Room: 205
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Barry Mordike, Technical University of Clausthal, Inst. of Matls. Engrg. & Tech. Germany; Wolfgang Blum, Universität Erlangen-Nürnberg, Inst. f. Werkstoffwissenschaften, Erlangen 91058 Germany

8:30 AM Invited
Creep Deformation Mechanisms in High Pressure Die Cast Magnesium Alloys: *Wolfgang Blum*[1]; Yujiao Li[1]; Xiaohui Zeng[1]; Berthold von Grossmann[2]; Christoph Haberling[2]; H.-G. Haldenwanger[2]; [1]University Erlangen-Nurnberg, Inst. f. Werkstoffwissenschaften, Martensstr. 5, Erlangen 91058 Germany; [2]Audi AG, Abt. I/EG-34, Ingolstadt 85045 Germany

Creep is described as an integral part of plasticity in terms of stress-strain rate-strain relations. Creep tests yield information on yield stress, work hardening, maximum deformation resistance (minimum creep rate) and work softening. Testing in compression avoids influences by fracture. Data on the new creep resistant alloy AJ52 (5Al, 2Sr) in the temperature range between 100 and 200°C are presented. Comparison with AZ91 and AS21 demonstrates that AJ52 has the highest creep resistance among the three. Electron microscopy shows that the microstructures of die cast Mg-Al-alloys consist of fine grains with a mantle containing large intermetallic precipitates. Dislocation structure evolution is different in both regions with stronger tendency to subgrain formation in the mantle. The difference in creep resistance are discussed in terms of differences in strength and stability of the intermetallic phases.

9:05 AM Invited
The Role of Precipitates for the Creep Behavior of Magnesium Die-Cast Alloys: *Oliver Kraft*[1]; Michael Vogel[2]; Eduard Arzt[3]; [1]Forschungszentrum Karlsruhe, Inst. fuer Materialforschung II, Postfach 3640, Karlsruhe 76021 Germany; [2]SiCrystal AG, Paul-Gossen-Str. 100, Erlangen 91052 Germany; [3]Max-Planck-Institut fuer Metallfoschung, Heisenbergstr. 3, Stuttgart 70569 Germany

Microstructure and creep behavior of ZA85 and two alloy modifications with 0.3 and 0.9 wt.% Ca (ZACa8503 and ZACa8509) were investigated in die-cast and annealed conditions. In general, it was found that the addition of Ca leads to a more creep resistant material. High temperature dislocation creep controls the deformation at low stresses as well as high stresses in all conditions. Further, the creep behavior is dominated by the formation and coarsening of τ-phase precipitates near grain boundaries. It will be shown that the creep behavior of all alloys and conditions can be described phenomenologically by a threshold concept for creep deformation. It is argued that the addition of Ca increases the threshold leading to the increased creep resistance. However, for long-term applications at elevated temperatures, our experiments highlight the role of microstructural stability for the creep deformation of Mg die-cast alloys as the precipitates will coarsen and the threshold is no longer effective.

9:40 AM Invited
On the Creep Resistance of Magnesium Die Casting Alloys: *Qingyou Han*[1]; Bimal K. Kad[2]; Srinath Viswanathan[3]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., One Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6083 USA; [2]University of California, Dept. of Structural Engrg., 409 Univ. Ctr., La Jolla, CA 92093-0085 USA; [3]Sandia National Laboratories, MS 1134, Org. 1835, PO Box 5800, Albuquerque, NM 87185-1134 USA

The microstructure of die cast magnesium alloys is highly non-uniform, which leads to a non-uniform distribution of the solidus/homologous temperature in the α(Mg) phase and a non-uniform distribution of stress in the specimen during creep testing. As a result, the creep deformation in the specimen is also non-uniform. More creep deformation occurs in the α(Mg) phase in and adjacent to the eutectic regions than that in the primary α(Mg) dendrites. This article addresses the effect of the non-uniformity in the microstructure on the creep resistance of die cast magnesium alloys. Computational thermodynamic simulations were carried out to determine solute segregation, solidus temperature, and the corresponding homologous temperature distribution in the α(Mg) phase. Transmission electron microscopy (TEM) studies provided evidence of non-uniform creep deformation in the creep tested specimens. The results suggest that the creep resistance of magnesium alloys is determined by the weakest phase in the alloy, viz., the α(Mg) phase in and adjacent to the eutectic regions. Factors that increase the homologous temperature or reinforce the eutectic α(Mg) phase increase the creep resistance of the magnesium alloys.

10:15 AM Break

10:30 AM Invited
Mechanisms of Creep Deformation in Mg-Sc Based Alloys: *Barry Mordike*[1]; I. Stulíková[2]; B. Smola[2]; [1]Technical University, Clausthal, Inst. of Matls. Engrg. & Tech. Germany; [2]Charles University, Faculty of Math. & Physics Czech Republic

Binary Mg-Sc alloys exhibit only poor age hardening due to the low diffusivity of Sc in Mg and consequently are less resistant to creep than the WE alloys. A small addition of Manganese (< 1.5 wt. %) improves the creep behaviour significantly. The minimum creep rates are up to two orders of magnitude lower than those of commercially available WE alloys at temperatures over 300°C. This is due to the finely dispersed Mn_2Sc phase in the form of discs on the basal plane, which are very effective obstacles in restricting creep. At these temperatures, this implies restricting cross slip of basal dislocations and non

basal slip. The addition of Ce improves the creep resistance even further due to the effect of a grain boundary eutectic. The effect of the Mn_2Sc discs is observed also in alloys with a low content of Sc (~ 1 wt. %) and the addition of a rare earth element (Gd, Y, Ce ~ 4 wt. %). Very thin hexagonal plates containing the rare earth and Mn and also parallel to the basal plane of the Mg matrix strengthen the effect of Mn_2Sc precipitates at elevated temperatures (~ 250°C). A triangular arrangement of prismatic plates of metastable or stable phases of Mg-rare earth systems controls effectively the movement of basal dislocations during creep of these alloys at elevated or high temperatures. The combined control of basal slip, cross slip of basal dislocations and of non basal slip in these alloys ensures the minimum creep rates of one order of magnitude lower than those in WE alloys both at elevated and high temperatures.

11:05 AM Cancelled
Recent Studies of Diffusional Creep in Magnesium Alloys and Other Materials

11:40 AM Invited
Creep Behavior and Deformation Substructure of Mg-Y and Mg-Y-Zn Alloys: *Mayumi Suzuki*[1]; Tsuyoshi Kimura[2]; Noritsugu Nakamura[1]; Jun-ichi Koike[1]; Kouichi Maruyama[1]; [1]Tohoku University, Grad. Sch. of Environmental Studies, Ecomatl. Design & Proc. Engrg. Course, 02 Aobayama, Aoba-Ku, Sendai, Miyagi 980-8579 Japan; [2]Tohoku University, Grad. Sch. of Engrg., Matls. Sci., 02 Aobayama, Aoba-Ku, Sendai, Miyagi 980-8579 Japan

Compressive creep behavior of Mg-Y and Mg-Y-Zn alloys was investigated at 550K to 650K. Deformation substructures have been observed by using TEM. Creep strength of Mg-Y alloys is significantly higher than other Mg based alloys. Furthermore, Creep rate of Mg-Y alloys decreased to 1/10 by the addition of zinc.High activation energy for creep was observed in Mg-Y and Mg-Y-Zn alloys even in dilute solid solution range. In microstructural observation, the activation of non-basal slip of a-dislocations was observed in Mg-Y alloys. In Mg-Y-Zn alloys, a high density of planar fault was observed on (0001) matrix planes, which suppress the non-basal slip of a-dislocations. Furthermore, many extended a-dislocations on basal planes were observed due to the decrease of the stacking fault energy. The simultaneous addition of yttrium and zinc can reduce the content of expensive yttrium without losing creep strength.

WEDNESDAY AM

Roytburd Symposium on Polydomain Structures: Elastic Domains in Structural Materials

Sponsored by: TMS, MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Julia Slutsker, National Institute of Standards and Technology, CTCMS, Gaithersburg, MD 20899 USA; Greg B. Olson, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Wednesday AM Room: 216A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Greg B. Olson, Northwestern University, Dept. of Matls. Sci. & Engrg., Evanston, IL 60208 USA; Julia Slutsker, National Institute of Standards and Technology, CTCMS, Gaithersburg, MD 20899 USA

8:30 AM Invited
From "Old" Martensite to Engineering of Polydomain Nanostructures: *Alexander L. Roytburd*[1]; [1]University of Maryland, Matls. & Nucl. Engrg., College Park, MD 20742 USA

The talk focuses on developing a new approach for materials engineering: using mescoscopic design of composites with transformable components to control self-assembling micro- and nano-structures arising during structure phase transformations. Such self-assembling polydomain structures are natural products of phase transformations in solids. The trend to minimize the energy of long-range elastic interactions leads to the formation of self-organized arrangements of domains of different phases or differently oriented domains of the same phases (twins). Transformations in bulk materials usually result in the formation of complex irregular polydomain structures that are difficult to control. In contrast, it is possible to obtain a completely controlled structure in a single crystalline film through constraint. The constraint can be imposed by embedding transformable materials in a composite architecture. Thus, the combination of engineered mesostructures and self-organized micro- and nano- polydomain structures presents broad opportunities to design new materials with well-controlled structures.

9:05 AM Invited
Combining Thermodynamics, Elasticity, Interfaces and Kinetics for Interpreting the Evolution of Microstructures and the Materials Properties Which Result: *John W. Cahn*[1]; [1]NIST, MESL, Gaithersburg, MD 20899-8555 USA

For the inquisitive and thoughtful materials theorist we honor today, microstructures and their evolution have displayed an amazingly rich variety of interesting phenomena. Phase diagrams provide only a partial explanation of what is seen. The free energies that form the basis of the phase diagrams greatly extend the predictive power of thermodynamics, especially if these energies are augmented with those due to the induced stresses and interfaces that form en route. Concepts of local and coherent equilibrium, polydomain and other energy-minimizing structures all have developed from careful observation and thought. The introduction of kinetics consistent with this richly augmented thermodynamics has provided an expanding basis for understanding and predicting microstructural processes and new properties in materials as diverse as steels, magnetic alloys, ferro-electrics, and thin films. The progress over the last 40 years has been astonishing, but much remains to be done.

9:40 AM Invited
Self-Assembling of Structural Domains in Phase Transformations: *Armen G. Khachaturyan*[1]; Yongmei M. Jin[1]; Yu U. Wang[1]; Dwight Viehland[2]; Jie-Fang Li[2]; [1]Rutgers University, Ceram. & Matls. Engrg., 607 Taylor Rd., Piscataway, NJ 08854 USA; [2]Virginia Polytechnic Institute and State University, Matls. Sci. & Engrg., 201 Holden Hall (0237), Blacksburg, VA 24061 USA

Given the similarity of stress-induced interaction with dipole-dipole interaction, the coherent phase transformations producing crystallographically equivalent orientation variants form domain structure similar to those of ferromagnetic and ferroelectrics. 3D simulations using Phase Field Microelasticity were performed for the domain structures formed in single-crystals, polycrystals, and thin films. Since the domain size is proportional to the square root of the domain wall energy, a drastic reduction of this energy results in domain miniaturization to nano- or subnano-scale and formation of a mixed adaptive state that usual diffraction methods see as a homogeneous phase with a microdomain-averaged lattice. This phase has special property - the high adaptivity provided by a high mobility of the low energy domain walls and the multivariant character of the polydomain structure. If the transformation is ferroelectric, it is an adaptive ferroelectric. Evidence of a ferroelectric adaptive phase in PMN-PT near the morphotropic boundary will be presented.

10:15 AM Break

10:30 AM Invited
Coherent Phase Equilibria in Ag-Au-Cu Nanoparticles: *William C. Johnson*[1]; James M. Howe[1]; Jooyoul Huh[2]; [1]University of Virginia, Dept. of Matls. Sci. & Engrg., PO Box 400745, Charlottesville, VA 22904-4745 USA; [2]Korea University, Dept. of Matls. Sci. & Engrg., 5-1, Anam-Dong, Seoul, Sungbuk-Ku 136-701 Korea

Professor Roytburd has made many contributions to our understanding of solid-state phase transformations. Of particular interest to us is his clarification of the interplay between compositional strain, misfit strain, and imposed geometrical constraints on the characteristics of phase equilibria in coherent systems. Here, we extend his work to consider the effect of particle size on coherent phase equilibria in small (10-50 nm diameter) Au-Cu-Ag nanoparticles. Transmission electron microscopy observations showing the presence of an L1_2 ordered phase in the nanoparticles at temperatures more than 250K above the bulk ordering temperature are investigated by considering the influence of surface stress on phase equilibria. This work is supported by the U.S. National Science Foundation.

11:05 AM Invited
Engineering of Elastic Domain Structures in Constrained Layers: Julia Slutsker[2]; *Andrei Artemev*[1]; Alexander L. Roytburd[3]; [1]Carleton University, Mech. & Aeros. Engrg., 1125 Colonel By Dr., Ottawa, ON K1S 5B6 Canada; [2]NIST, Gaithersburg, MD 20899 USA; [3]University of Maryland, Matls. Sci. & Engrg., College Park, MD 20742 USA

The formation and evolution of polydomain microstructure under external stress in the constrained layer are investigated by phase-field simulation. The reversible superelastic deformation of two-phase austenite/martensite mixture has been modeled as well as superplastic deformation of martensite polydomain structure.

Solidification of Aluminum Alloys: Solidification Cracking/Mechanical Properties

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Solidification Committee
Program Organizers: Men Glenn Chu, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Douglas A. Granger, GRAS, Inc., Murrysville, PA 15668-1332 USA; Qingyou Han, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Wednesday AM Room: 207B/C
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: David R. Poirier, University of Arizona, Matls. Sci. & Engrg., Tucson, AZ 85721 USA; Men G. Chu, Alcoa Inc., Alcoa Technical Center, Alcoa Ctr., PA 10569 USA

8:30 AM Keynote
How Does Coalescence of Dendrite Arms or Grains Influence Hot Tearing?: *M. Rappaz*[1]; V. Mathier[1]; P.-D. Grasso[1]; J.-M. Drezet[1]; A. Jacot[1]; [1]EPFL, Inst. of Matls., Computational Matls. Lab., MXG, Lausanne CH-1015 Switzerland

Hot tearing, a severe defect occurring during solidification, is the conjunction of tensile stresses that are transmitted to the mushy zone by the coherent solid underneath and of an insufficient liquid feeding to compensate for the volumetric change. In most recent hot tearing criteria, one of the critical issues is the definition of a coherency point which, in low-concentration alloys, corresponds to the bridging or coalescence of the primary phase. A coalescence model has been developed recently using the concept of the disruptive pressure in thin liquid films.[1] It has been shown that large-misorientation grain boundaries, which are characterized by an interfacial energy, γ_{gb}, larger than twice the solid-liquid interfacial energy, γ_{sl}, solidify at an undercooling $\Delta T_b = (\gamma_{gb} - 2\gamma_{sl})/(\Delta s_f \delta)$, where Δs_f is the entropy of fusion and δ the thickness of the diffuse interface. When $\gamma_{gb} < 2\gamma_{sl}$ (e.g., weak-misoriented grain boundaries), dendrite arms coalesce as soon as they impinge on each other. Using such concepts and a back-diffusion model, the percolation of equiaxed, randomly oriented grains has been studied in 2D: it is shown that the grain structure gradually evolves from isolated grains separated by a continuous interdendritic liquid film, to a fully coherent solid with a few remaining wet boundaries. The implication of such findings for the hot cracking tendency of aluminum alloys will be discussed. [1]M. Rappaz, A. Jacot and W. J. Boettinger, Last stage solidification of alloys: a theoretical study of dendrite arm and grain coalescence. Met. Mater. Trans. 34A (2003) 467-79.

9:00 AM
A New Two-Phase Thermo-Mechanical Model and its Application to the Study of Hot Tearing Formation During the Start-Up Phase of DC Cast Ingots: M. M'Hamdi[1]; *H. G. Fjær*[2]; A. Mo[1]; D. Mortensen[2]; S. Benum[3]; [1]SINTEF Materials Technology, PB 124 Blindern, N-0314 Oslo Norway; [2]Institute for Energy Technology, PB 40, N-2007 Kjeller, Oslo Norway; [3]Hydro Aluminium, N-6600 Sunndalsøra Norway

The purpose of this work is to present a new 2D two-phase simulator, TearSim, for the prediction of hot tearing formation in DC casting of aluminum ingots. In this model, the main mechanisms for the formation of hot tears during solidification, namely too severe thermally-induced deformations, and the lack of liquid feeding to compensate for solidification shrinkage and viscoplastic volumetric dilatation, are addressed simultaneously. In the modelling, the liquid flow, the stresses and the strains in the two-phase mushy zone are calculated using an advanced model for the viscoplastic deformation of the semi-solid material. In the present article, the modeling equations are solved numerically for the direct-chill casting of axi-symmetric aluminum extrusion ingot. In order to study the effect of the casting speed on the formation of center hot-cracks, new hot tearing criteria based on the mechanical quantities resulting from the simulations are compared to experimental results from casting trials with varying the casting speed ramping during the casting process.

9:20 AM
The Influence of Hydrogen on the Hot Tearing Susceptibility of Aluminium-Silicon Alloys: *Russell S. Barnett*[1]; John A. Taylor[1]; David H. StJohn[1]; [1]University of Queensland, FAST Casting Ctr. of Excellence, Brisbane, Queensland 4072 Australia

An automotive company has recently adopted procedures to control the melt hydrogen content of their cast aluminium air-intake manifolds. This arose because the hydrogen content showed a strong correlation with the incidents of hot tearing. Although the literature typically reports a negative impact of hydrogen on hot tearing it was clearly observed that higher hydrogen levels decreased hot tearing susceptibility in the manifold. Based on these in-house observations a series of experiments have been conducted using the common ring test mould. The impact of hydrogen content on hot tearing with varying solute (silicon) content and eutectic modification was closely examined using a hot tearing severity index that was established from ring crack dimensions. Results from the hydrogen trials showed the typical lambda curve for hot tearing versus solute content that has been well documented. The relationship between gas content and hot tearing is analysed with a view to establishing new insight into the mechanistic processes of hot tear formation.

9:40 AM
An Experimental Method for Determining Mechanical Properties of Non-Equilibrium Mushy Zone: *Qingyou Han*[1]; Mohamed I. Hassan[2]; Srinath Viswanathan[3]; Subodh K. Das[4]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., One Bethel Valley Rd., Oak Ridge, TN 37831-6083 USA; [2]University of Kentucky, Dept. of Mech. Engrg., 151 RGAN Bldg., Lexington, KY 40506-0108 USA; [3]Sandia National Laboratories, PO Box 5800, Albuquerque, NM 87185-1134 USA; [4]Secat Inc., 1505 Bull Lea Rd., Lexington, KY 40511 USA

The mushy zone of an alloy during solidification is non-equilibrium and the mechanical properties of this non-equilibrium mushy zone, especially at small liquid fractions, are closely related to the formation of hot tearing during solidification of castings. Experimentally it is very difficult to measure the mechanical properties of the non-equilibrium mushy zone with a small liquid fraction because the liquid fraction decreases rapidly due to back diffusion in the solid. This paper describes a new experimental method for determining the mechanical properties of the non-equilibrium mushy zone. Back diffusion in the solid of the mushy zone is considered in order to prevent liquid from disappearing. Specimens were taken from castings so that their grain sizes are same to the relevant casting. Initial experimental results indicate that the method can be used to capture the brittleness of an alloy at temperatures close to the non-equilibrium solidus temperature of the alloy.

10:00 AM Break

10:20 AM Keynote
Constitutive Behaviour and Hot Tearing During Aluminum DC Casting: *Laurens Katgerman*[1]; Willem-Maarten Van Haaften[2]; Pim Kool[1]; [1]Delft University of Technology, Matls., Rotterdamseweg 137, Delft 2628AL The Netherlands; [2]Corus RD&T, IJmuiden The Netherlands

The constitutive behaviour of two non-heat treatable industrial aluminum alloys (AA3104 and AA5182) is investigated. This is done by testing the as-cast material in tension at low strain rates and from room temperature to semi-solid temperatures, similar to the conditions during DC casting. The parameters of two constitutive equations, the extended Ludwik equation and a combination of the Sellars-Tegart equation with a hardening law, were determined. To evaluate the quantified constitutive equations, tensile tests were performed simulating the deformation and cooling history experienced by the material during casting. In the semi-solid state the behavior is dominated by the solid network but the geometry of the liquid determines how much of the solid network contributes to the strength. A simple modification of a standard creep law, which takes into account the geometry of the liquid film, provides a continuous description of the constitutive behaviour of these alloys from the creep regime into the semi-solid state. It is concluded that the constitutive behaviour of the alloys in as-cast condition is well described by both the extended Ludwik equation and an adapted form of the Sellars-Tegart equation. Although the extended Ludwik equation describes the data better, the adapted form of the Sellars-Tegart equation is easier to implement in DC casting models, because the temperature appears explicitly in this equation. In order to study the hot tearing mechanism, tensile tests are carried out in semi-solid state and at low strain rates and crack propagation is studied in-situ by SEM. Microstructural investigations of these cracks indicates that they initiate at any weak spot such as a pore or partially liquid grain boundary and occur by a combination of fluid film separation and rupture of solid bridges. This leads to brittle behaviour on the large scale although local deformation can be very ductile. Similarities between hot tears in the industrial ingot and cracked specimens indicate that important aspects of hot tearing during casting can be simulated by tensile experiments at semi-solid temperatures.

10:50 AM
Tensile Properties of As-Cast AA5182 Aluminum Alloy Close to the Solidus Temperature: *L. J. Colley*[1]; M. A. Wells[1]; D. M. Maijer[1]; S. Cockcroft[1]; [1]University of British Columbia, Dept. of Metals & Matls. Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

In order to address the demand for accurate mechanical property data in the partially solidified state, an experimental apparatus has been developed to perform tensile measurements of aluminum alloys at temperatures close to the solidus temperature. Measurements of the tensile properties of an industrially DC cast AA5182 aluminum alloy have been carried out between 500ºC and 580ºC, under strain rate conditions of ~10-4s^{-1} to ~10-2s^{-1}. The fracture surfaces and microstructures of the tested specimens have been examined using optical microscopy, SEM and EDX analysis to develop a relationship between tensile properties, fracture behaviour and changes in microstructure. Literature values of the solidification characteristics of AA5182 have also been used to relate the properties and microstructure with fraction liquid.

11:10 AM
Effects of 1-4% Copper Additions on Semi-Solid Aluminum Alloy 357: *Jon T. Carter*[1]; Vjekoslav Franetovic[1]; [1]General Motors, R&D Ctr., Matls. & Processes Lab., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

The effects of copper additions on aluminum alloy 357 were studied in the course of alloy development for semi-solid forming. Copper additions of 1-4% were found to (a) increase the overall hardness and greatly increase the microhardness of the regions between the primary alpha grains, (b) inhibit spheroidization of alpha dendrites during the heat treatment in which the interdendritic constituents are melted, and (c) slightly refine the dendritic grains and promote a divorced form of the eutectic constituent in the as-cast microstructure. Data are presented in the forms of hardness plots, micrographs, solidification cooling curves, and heating curves.

11:30 AM
Rheocasting of Aluminum Alloys: *Sungbae Park*[1]; Ian C. Stone[1]; Brian Cantor[2]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK; [2]University of York, York UK

Many processes have been developed for semi-solid casting. Recently, UBE Industries has introduced a new semi-solid casting process which comprises the preparation of the semi-solid slurry directly from the melt in-line with the casting machine. In the UBE rheocasting process semi-solid slurries with globular microstructures are formed using continuous controlled cooling, and in this study, the resulting semi-solid microstructures have been characterised quantitatively as a function of different slurry making conditions. In order to determine the deformability of the semi-solid slurries, thermomechanical analysis (TMA) has been carried out. This paper describes the optimum slurry making conditions in the UBE rheocasting process.

Solidification Processes and Microstructures: A Symposium in Honor of Prof. W. Kurz: Phase Field

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Solidification Committee
Program Organizers: Michel Rappaz, Ecole Polytechnique Fédérale de Lausanne, MXG, Lausanne Switzerland; Christoph Beckermann, University of Iowa, Department of Mechanical Engineering, Iowa City, IA 52242 USA; R. K. Trivedi, Iowa State University, Ames, IA 50011 USA

Wednesday AM Room: 207D
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Andreas Ludwig, University of Leoben, Dept. of Metall., Leoben 8700 Austria

8:30 AM Invited
Fractal Solidification Patterns: *Heiner Mueller-Krumbhaar*[1]; [1]Forschungszentrum Juelich, Inst. Festkoerperforschung, D-52425 Juelich Germany

Fractal structures are frequently observed in solidification processes. A short overview is given starting with dendrites and seaweed structures[1] and covering also our recent results on elastic effects in segregation and in fractal layer growth on substrates.[2] [1]E.Brener, H. Mueller-Krumbhaar, D. Temkin, T. Abel, Morphology Diagram of Possible Structures in Diffusional Growth Physica A 249, 73 (1998); H. Mueller-Krumbhaar, T. Abel, E. Brener, M. Hartmann, N. Eissfeldt, D. Temkin; Growth-Morphologies in Solidification and Hydrodynamics; JSME International Journal B 45, 1, 2002. [2]H. Mueller-Krumbhaar, F. Gutheim, R. Spatschek, E. Brener; Elastic effects on growth processes, Appl. Surface Science, 7160, 1 (2001); F. Gutheim, H. Muller-Krumbhaar, E. Brener, V. Kagan; Thermal Roughening of a Solid-On-Solid Model with Elastic Interactions, Phys. Rev. B 67, 195404 (2003).

9:00 AM Invited
Phase Field Simulation of Directional Solidification: *Jonathan A. Dantzig*[1]; Bari Athreya[1]; [1]University of Illinois, Dept. of Mech. & Industrial Engrg., MC-244, 1206 W. Green St., Urbana, IL 61801 USA

Prof. Kurz has made numerous important contributions to the understanding of microstructure development. Much of this work has been done by studying transparent binary alloys in directional solidification, where the temperature gradient and growth velocity are independently controlled, and the ensuing microstructure is examined. The sample is confined between microscope slides to make observation easier. In this work, we examine the directional solidification process in binary alloys. In particular, we examine the role of the confinement of the diffusion fields by the microscope slides on the development of microstructure. We demonstrate that 3-D effects are important, and compare our results to those found in experiments, and in simulations performed using 2-D approximations.

9:30 AM Invited
Globular-Dendritic Transition in Equiaxed Alloy Solidification: Hermann-Josef Diepers[1]; *Alain S. Karma*[1]; [1]Northeastern University, Dept. of Physics, Boston, MA 02139 USA

Depending on the growth conditions, equiaxed grains can develop dendritically or form a globular morphology that is often associated with a high density of nucleants. Despite the widespread observation of both dendritic and globular grains, the transition between these two morphologies remains poorly understood. Existing equiaxed growth models assume Ivantsov-like dendrite tip kinetics that is only valid for well-developed dendritic grains, and Zener's classical theory of growth transients only describes the initial development of spherical nuclei. We have carried out a phase-field study of the unsteady growth regime in between these two extremes that provides new insight into the globular-dendritic transition. The results reveal the existence of robust scaling laws that relate quantitatively the critical total grain size for this transition with the cooling rate and alloy parameters. These laws are interpreted with the help of simple analytical models.

10:00 AM Break

10:30 AM
Examination of Binary Alloy Dendrite Tip Operating State with a Phase-Field Model: Juan Ramirez[1]; *Christoph Beckermann*[1]; [1]University of Iowa, Dept. Mech. & Industrial Engrg., 2412 SC, Iowa City, IA 52242 USA

The description of free dendritic growth into an undercooled binary alloy melt is most commonly performed with the Lipton, Glicksman and Kurz (LGK) model. Taking into account heat and solute diffusion, this model describes the variation of the steady-state dendrite tip velocity and radius with solute concentration. The LGK model assumes the existence of a selection parameter that remains constant and independent of undercooling and initial melt concentration. The LGK model is re-examined using a recently developed phase-field model. This thermosolutal phase-field model allows for the simulation of dendritic growth without interface kinetics and is used to examine the variation of the dendrite tip operating state with initial melt solute concentration. As predicted by the LGK theory, a maximum in the steady-state tip velocity is observed for small but finite melt concentrations, but other results indicate that additional study is needed.

10:45 AM
Microstructure Evolution in the Presence of Foreign Particles: *James A. Warren*[1]; Laszlo Granasy[2]; [1]NIST, CTCMS, 100 Bureau Dr., Stop 8554, Gaithersburg, MD 20899-8554 USA; [2]RISSPO, PO Box 49, H-1525, Budapest Hungary

The evolution of microstructure during solidification is the focus of much of Wilfried Kurz's research. While great progress has been made in the theory of perfect single crystals of either a single component or a binary alloy, Nature has not obliged us with circumstances where such systems are easily realized. A major focus of our research has been the extension of phase field models of solidification to include polycrystalline materials. In this talk we will assess the range of applicability of this model, in particular examining the role that foreign particles can play during dendritic growth, as well as the occurrence of spherulitic growth during solidification of both metals and polymers.

11:00 AM

Three-Dimensional Phase-Field Simulations of Directional Solidification: Marcus Dejmek[1]; Roger Folch[1]; Andrea Parisi[1]; *Mathis Plapp*[1]; [1]CNRS/Ecole Polytechnique, Lab. PMC, Rte. de Saclay, Palaiseau 91120 France

The phase-field method has become in recent years the method of choice for simulating microstructural pattern formation during solidification. One of its main advantages is that time-dependent three-dimensional simulations become feasible. This makes it possible to address long-standing questions of pattern stability. Here, we investigate the stability of hexagonal cells and eutectic lamellae. For cells, it is shown that the geometry of the relevant instability modes is detemined by the symmetry of the steady-state pattern, and that the stability limits strongly depend on the strength of crystalline anisotropy, as was previously found in two dimensions. For eutectics, preliminary investigations of lamella breakup instabilities are presented that are carried out with a newly developed phase-field model of two-phase solidification.

11:15 AM

Experimental Observation and Phase-Field Modeling of Interface Morphological Transition in Solidification: *Taiming Guo*[1]; Haijun Xu[2]; Thein Kyu[2]; G.-X. Wang[1]; [1]University of Akron, Dept. of Mech. Engrg., Akron, OH 44325-3903 USA; [2]University of Akron, Dept. of Polymer Engrg., Akron, OH 44325-0301 USA

Depending on the relative extent of the anisotropy of the interface energy, a growing solidification interface may develop with various distinguished patterns, such as normal dendrites, degenerate dendrite, and seaweed. This presentation will present experimental observations of the dynamic variation of various interface morphologies in a horizontal unidirectional solidification system using succinonitrile. Under certain circumstances, non-dendritic interface morphologies have been observed. It is found that formation of any specific morphology depends on the interface growth velocity and the temperature gradient. Dynamic transition from one pattern to another has also been observed and documented. The formation and transition of these non-dendiritc patterns are then simulated by using a 2-D phase-field model coupled with heat conduction equation in which the anisotropy of the interface energy can be artificially set and adjusted. Both unidirectional and unconstrained crystal growth process have been simulated using the model. It is found that the interface morphology could transit from seaweed to dendritic pattern because of the variation of parameters K and e of heat conduction equation, where K is proportional to the latent heat released at the interfacial front and inversely proportional to the supercooling, and e is related to the thermal diffusivity. These numerical results confirm qualitatively the experimental observations. Quantitative comparisons between the numerical and experimental results are also attempted and will be presented.

11:30 AM

Phase Field Modeling of Step Flow With Dendritic Pattern: *Seong Gyoon Kim*[1]; *Won Tae Kim*[2]; [1]Kunsan National University, Dept. of Matls. Sci. & Engrg., Kunsan 573-360 Korea; [2]Chongju University, Appl. Sci. Div., 36 Naedok Dong, Chongju 360-764 Korea

Macrosteps with dendritic pattern have been observed on the vicinal (0001) surface of sapphire during evaporation in vacuum at temperatures between 1923K and 2223K. To clarify the pattern formation mechanism of the steps, we perform the linear stability analysis, as well as phase-field computations, based on the classical Burton-Cabrera-Frank model. With increasing annealing temperature evaporation rate and dendritic growth rate increased. The growth rate dependence of the dendritic tip radius was different from the well known scaling law (R^2V with R: tip radius, V: growth rate) observed in solidification process. Tip radius was not strongly dependent of the growth rate. This could be explained by the co-existence of adatom evaporation and adatom diffusion within the diffusional field on upper terrace, which is in contrast with the solute diffusion only in liquid phase during solidification. A 2-D phase-field modeling including the multiple step interaction via diffusional field and the decomposition of accelerating steps near the step source could reproduce all the characteristics of the patterns with temperature.

11:45 AM

Phase Field Modeling of Dendritic Growth With Facets in an Undercooled: *Seong Gyoon Kim*[1]; Won Tae Kim[2]; Toshio Suzuki[3]; [1]Kunsan National University, Dept. Matls. Sci. & Engrg., Kunsan 573-360 Korea; [2]Chongju University, Appl. Sci. Div., 36 Naedok Dong, Chongju 360-764 Korea; [3]University of Tokyo, Dept. of Metall. Sys. Engrg. for Matls., Tokyo 113-8656 Japan

We extend our new phase field model of alloy solidification, characterized by localization of solute redistribution into a narrow region to minimize anomalous interface effects in the thin interface model, to model the solidification of faceted materials. This approach consists of finding missing orientations in an equilibrium shape, regularization of anisotropy based on the method proposed by Eggleston et al [Physica D, 150(2001),91]. The phase field equation was solved on 2D geometry at the vanishing interface kinetics condition with various anisotropies between $0< \delta <0.3$. When $\delta < 1/15$, a normal dendrite without cusps appeared as expected and dendrite with cusps appeared at $\delta > 1/15$. As the anisotropy δ increases from 0 to 1/15, steady state tip radius decreased while tip growth rate increased. A transition in tip growth rate appeared when normal dendrite changes to dendrite with cusps. With further increase in anisotropy δ above 1/15, steady state growth rate increased and reached a saturation value. Scaling laws between tip radius and tip growth rate will be discussed.

12:00 PM

A 2-Dimensional Model Coupled to a Thermodynamic Database for the Prediction of Solidification Microstructures in Multi-Component Alloys: *Alain Jacot*[1]; Qiang Du[1]; [1]Ecole Polytechnique Fédérale de Lausanne, Matls. Inst., Computational Matls. Lab, Lausanne 1015 Switzerland

A two-dimensional model based on the pseudo-front tracking method was presented recently as an interesting approach to predict the formation of globulo-dendritic grains during solidification in multi-component systems.[1] This method permits to calculate the evolution of solid/liquid interfaces which are governed by solute diffusion and anisotropic surface tension. It is based on a finite volume method for the resolution of the diffusion equations and a geometrical approach for the calculation of the interface curvature. The present contribution deals with a recent extension of the pseudo-front tracking method to the formation of secondary phases. The secondary phase model is based on a mixture approach and the assumption that the interdendritic regions are composed of liquid and secondary particles locally in thermodynamic equilibrium. The model accounts for back-diffusion in the primary phase, which continuously modifies the composition of the interdendritic regions. The phase diagram software Thermo-Calc is used to obtain the equilibrium concentrations to be prescribed as boundary conditions at the primary phase/liquid interface and, during the second stage of solidification, to calculate the composition of the mixture in the interdendritic region. The model is applied to the description of microstructure formation in commercial aluminum alloys solidified under different cooling conditions. Comparisons between experimental and calculated microstructures will be shown. [1]A. Jacot and M. Rappaz, Acta Materialia 50, (2002), pp. 1909-1926.

12:15 PM

A Cellular Automaton for Growth of Solutal Dendrites: Factors Influencing Artificial Anisotropy in Growth Kinetics: *Srinivasan Raghavan*[1]; Matthew John M. Krane[1]; David R. Johnson[1]; [1]Purdue University, Sch. of Matls. Engrg., 501 Northwestern Ave., W. Lafayette, IN 47907-2036 USA

Cellular automaton is used to simulate the dendritic growth controlled by solutal effects. The model does not explicitly track the interface velocity by the flux equations; the interface motion results from the combined effect of maintaining equilibrium composition, solute rejection and the diffusion of solute at the interface. The grid of the cells introduces artificial anisotropy in the growth of dendrites, favoring growth at angles 0° or 45° from the vertical on a square grid. We propose a solution to reduce the anisotropy by suitable choice of growth rules and by identifying the appropriate dependence of cell size and time step on material properties and process parameters. The cell size is limited by the diffusion length and the imposed velocity. Finally, the simulated results are compared with analytical and experimental results for the tip radius, undercooling and primary dendritic arm spacing and fraction primary solid to test the validity of the model.

Surfaces and Interfaces in Nanostructured Materials: Synthesis & Processing

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Sharmila M. Mukhopadhyay, Wright State University, Department of Mechanical and Materials Engineering, Dayton, OH 45435 USA; Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Wednesday AM Room: 217A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Arvind Agarwal, Florida International University, Dept. of Mech. & Matls. Engrg., Miami, FL 33174 USA

8:30 AM Invited
The Formation of Nanoparticles in Sapphire and Silica by Ion Implantation: *Janet M. Hampikian*[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Engrg., 771 Ferst Dr., NW, Atlanta, GA 30332-0245 USA

The formation of nano-composites comprising metal clusters (5 to 20 nm in diameter) embedded in dielectric hosts via ion implantation is presented. Two basic approaches yield embedded nanoparticles: first, the implantation of noble metal ions, and second, the implantation of highly reactive metal ions that reduce the substrate cations to form metal atoms. An example of the former approach is the formation of gold clusters via gold ion implantation of sapphire. The latter approach relies on the reduction of the substrate. An example of this is the formation of aluminum clusters in sapphire via yttrium ion implantation. The thermodynamics and kinetics of the latter approach are presented, including the effects of ion energy, ion fluence and implantation temperature. The metal particles so formed are amorphous and do not contribute to electron diffraction. Therefore, energy filtered transmission electron microscopy is used as a characterization tool in conjunction with TEM, PEELS and EDS. Optical absorption measurements of the modified surfaces are also presented.

8:55 AM
Synthesis and Processing of Nanocrystalline Magnetic Materials: *Jeffrey E. Shield*[1]; [1]University of Nebraska, Mech. Engrg., N104 WSEC, Lincoln, NE 68588-0656 USA

Interfacial interactions in magnetic materials lead to many interesting effects, among them exchange-spring behavior. The exchange-spring interaction involves spins coupling across interfaces, resulting in remanence enhancement and improved energy densities. In order to exploit the interfacial effects in magnetic materials for applications, it is necessary to develop techniques and alloys that result in nanoscale structures, with grain sizes less than 40 nm. In addition, combinations of hard and soft magnetic phases are desirable for optimum magnetic properties. In this talk, various processing routes to a nanoscale structure will be discussed. These include atom-up approaches such as cluster deposition, and more tradition bulk routes such as melt spinning. In the cluster deposition approach, gas phase condensation results in the formation of 8 nm Fe clusters, which have been imbedded in a hard magnetic matrix. In melt-spinning, efforts have concentrated on understanding the nanostructural development and in controlling the grain size and distribution through alloying additions in both the Nd-Fe-B and Sm-Co systems. This talk will summarize efforts in these areas.

9:15 AM
Surface Nanocrystalline Metallic Materials Fabricated by Surface Severe Plastic Deformation: *Nobuhiro Tsuji*[1]; Masahide Sato[1]; Yoritoshi Minamino[1]; Yuichiro Koizumi[1]; [1]Osaka University, Dept. of Adaptive Machine Sys., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

Severe plastic deformation (SPD) above 4 of equivalent strain can produce ultrafine grained (UFG) metallic materials whose mean grain sizes are around 100 nm. In order to fabricate bulk UFG materials, however, special equipment and/or techniques as well as huge amount of plastic working energy are necessary. Meanwhile, the present authors have succeeded in fabricating surface nanocrystalline materials in bulky shape, where only subsurface layers have nanocrystalline structures, by a surface-SPD process using simple wire-brushing. Nanocrystals with mean diameters below 100 nm formed at surface layers of various kinds of Al alloys, Cu alloys and steels after the surface-SPD processing at ambient temperature. In case of commercial purity aluminum, the depth of the nanocrystalline layer was about 15 micro-meters. Interesting properties of the surface nanocrystalline materials will be also introduced. For example, the surface nanocrystalline aluminum performed significantly higher proof stress than the coarse grained material without nanocrystalline surface.

9:35 AM Cancelled
Development of Nanocrystalline Light Metals Via Mechanochemical Processing

9:55 AM Invited
Processing Bulk Structures from Nanopowders of Aluminum: *Jixiong Han*[1]; Martin J. Pluth[1]; Jai A. Sekhar[1]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Chem. & Matls. Engrg., 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 500 and 650°C for various times. Both hot and cold rolling of the pressed pellets was also utilized to assess workability and hardness after processing. Density was measured, and thin foils were prepared for TEM. Observations revealed that the oxide scale remained intact on sintering at 500°C, but showed evidence of breakage at higher temperatures, although in both cases a very interesting Al matrix-Al oxide nanocomposite resulted. TEM observations revealed that both constituents retained nanoscale dimensions, though there was evidence of growth of the Al grains compared with the initial particles. High densification, coupled with high hardness could be achieved. In addition, the sintered materials could be cold-rolled a s high as 60% reduction in thickness with additional densification and hardness increase. High-resolution SEM observations revealed nanoscale cracks in the rolled materials. These various results will be presented and discussed. In addition, attempts were made to produce extrusions from the nanopowders. The results of the microstructure, thermal stability and mechanical properties of these materials will also be presented. Support for this research from AFOSR under grant no. F49620-01-1-0127, Dr. Craig S. Hartley, Program Monitor, is deeply appreciated.

10:20 AM
Modern Lithographic Techniques for the Fabrication of Patterned Self-Assembled Monolayers on Metallic Surfaces: *Francesco Stellacci*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 13-5049, Cambridge, MA 02139 USA

One of the main challenge in nanotechnology is to generated surfaces with controlled morphology properties. Here we present two techniques based on scanning probe microscopes that allow for the patterning of functionalized molecules on metallic surfaces. The first technique is Dip Pen Nanolithography (DPN) that allows for the controlled deposition of molecules on surfaces. Patterns can be written with 20 nm resolution. We show that, in DPN, by varying the writing speed and the relative humidity it is possible to control the morphology of the written self-assembled monolayers (SAMs) for both hydrophobic and hydrophilic thiolated molecules on a gold surface. We have used this properties to control the self-assembly of nanoparticles on this surface or to generate efficient etching masks. The second technique is replacement lithography (RL) that allows for the patterning of < 4 nm features. Here, we demonstrate the ability of RL to pattern supramolecular fluorescent nanowires.

10:40 AM
Plasma Engineered Nanostructured Spherical Powders: *Tapas Laha*[1]; Brandon Potens[2]; Arvind Agarwal[3]; Sudipta Seal[4]; [1]Florida International University, Mech. & Matls. Engrg., 10555 W. Flagler St., CEAS 3362, Miami, FL 33174 USA; [2]Florida International University, Mech. & Matls. Engrg., Mech. & Matls. Engrg., 10555 W. Flagler St., Miami, FL 33174 USA; [3]Florida International University, 10555 W. Flagler St., CEAS 3464, Miami, FL 33174 USA; [4]University of Central Florida, Mech. Matls. & Aeros. Engrg., Engrg. Bldg. 1, Rm. 381, PO Box 162455, Orlando, FL 32816 USA

Nano-structured spherical powders of Al and Al2O3 were synthesized by plasma spraying irregular shaped micron sized powders. The sprayed powders were collected under extreme cooling condition of liquid nitrogen to ensure the retention of nanograins in the powders. Both the as-received powders and the plasma-engineered powders were observed in Scanning Electron Microscope to study the change in

chemical composition, morphology and size of the powders due to plasma processing. X-Ray Diffraction investigations were made to carry out the phase study and grain size measurement. The formation of nanograins in plasma-engineered powders was examined by performing Transmission Electron Microscopy.

11:00 AM Invited
Directed Assembly of Mesoarchitectures and Networks from 0-D and 1-D Nanounits: *G. Ramanath*[1]; [1]Rensselaer Polytechnic Institute, Dept. of Matls. Sci. & Engrg., Troy, NY 12180 USA

There is widespread interest in harnessing nanotubes and nanowires for applications such as device interconnection, switching, field emission, sensing, molecular sieving, and structural reinforcements for nanocomposites. In order to realize these exciting possibilities, it is essential to controllably create mesoscale architectures and networks from nanoscale units, placed in desired locations and orientations, by scaleable approaches. This talk will describe two powerful bottom-up strategies to assemble: a) mesonetworks of metallic nanowires from nanoparticles, and b) multidirectional mesoarchitectures of carbon nanotubes (CNTs). The first example will illustrate a room-temperature templateless wet-chemical method for producing metal nanowires from nanoparticles using biphasic liquid mixtures. Nanoparticles are forced to impinge and coalesce at high mobility liquid-liquid interfaces leading to the formation of polycrystalline nanowires. The role of surface processes and liquid-interface chemistries in the mo rphological evolution of the nanowires will be described based on electron microscopy, diffraction, and electron and optical spectroscopy measurements. The second example will describe a hybrid processing approach that combines substrate-selective CVD and lithographic chiseling of substrates to controllably place and grow CNTs at premeditated nucleation sites and orientations. The CNTs selectively grow on silica surfaces in exclusion to silicon, in an orientation parallel to the surface normal. By shaping the silica templates by lithography, we obtain a variety of 2D and 3D mesoscale relief, porous, architectures with complex shapes, configurations, and lengths. Salient features of the growth mechanism will be discussed. If time permits, a newly discovered strategy to site-selectively anchor nanoclusters to CNTs will also be presented.

11:25 AM
Hydrogen Storage Properties of Multiwall Carbon Nanotubes Grown in Oxygen Added PECVD: *Jai-Young Lee*[1]; Hyun-Seok Kim[1]; Ho Lee[1]; Jin-Ho Kim[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Engrg., 373-1, Guseong-dong, Yuseong-gu, Daejon 305-701 Korea

Hydrogen storage properties of the two kinds of multiwall carbon nanotubes (MWNTs) synthesized by microwave PECVD were evaluated. The first sample grown in CH4/H2 reaction gas was curly shaped nanotubes with blocked nanoholes and closed caps and the next sample grown in CH4/H2/O2 reaction gas had less defective structure with connected holes and open caps. The hydrogen storage properties of the carbon nanotubes with these closed and open structures were compared by thermal desorption technique. Though the MWNTs with blocked and closed structure had two different desorption ranges such as 290-330 and about 420 K and evolved about 0.64 and 0.03 wt% of hydrogen respectively, the MWNTs with open MWNTs evolved about 1.94 wt% of hydrogen at ambient temperature. And the hydrogen desorption activation energy was calculated. The obtained hydrogen desorption activation energy of MWNTs with closed and open structure at ambient temperature was -18.5 kJ/mol H2 and -16.5 kJ/mol H2, respectively. The activation energy of high temperature hydrogen desorption in MWNTs with closed structure was -124.4 kJ/mol H2. The hydrogen desorbed between 290 and 330 K was the hydrogen physi-sorbed in nano-hole and the hydrogen desorbed at about 420 K was that chemi-sorbed in defects of nanotubes.

The Didier de Fontaine Symposium on the Thermodynamics of Alloys: Joint Session with Computational Thermodynamics and Phase Transformations I

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA; Mark D. Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Gerbrand Ceder, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Wednesday AM Room: 216B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

8:30 AM Invited
The Ground State of Binary Compounds from First Principles: *Alex Zunger*[1]; [1]NREL, Basic Sci., 1617 Cole Blvd., Golden, CO 80401 USA

Finding the ground state structures of compounds has become a cornerstone problem in inorganic structural chemistry, metallurgy, and solid state physics. I will briefly review the way this problem was first approached in the early classical literature, and then describe the "modern", Mixed-Basis-Cluster-Expansion approach to it. In this approach, first-principles total energy methods of just a handful of compounds are maped into a generalized Ising-like expansion that accounts for atomic relaxation, long-range strain effects, and generic bonding effects. Human intervention is minimal, and no adjustable parameters exist. Didier's seminal contributions to this approach will be discussed.

9:00 AM Invited
Multi-Scale Thermodynamics Calculations from the First-Principles: *Tetsuo Mohri*[1]; Ying Chen[2]; Munekazu Ohno[1]; [1]Hokkaido University, Grad. Sch. of Engrg., Div. of Matls. Sci. & Engrg., Kita-13 Nishi-8, Kita-ku, Sapporo 060-8628 Japan; [2]Univeristy of Tokyo, Dept. of Quantum Engrg. & Sys. Sci., Hongo 7-3-1, Bunkyo-ku, Tokyo 113-8655 Japan

Based on FLAPW total energy calculations combined with Cluster Expansion Method and Cluster Variation Method, extensive studies on phase equilibria for Fe-Ni, Fe-Pd and Fe-Pt systems have been attempted. The transition temperatures of L10-disorder were well reproduced with high accuracy. In order to extend the present studies to predict microstructural evolution process, Phase Field Method has been employed. The preliminary calculations have been successfuly performed to reproduce the essential feature of three kinds of multiscale phenomena; 1. Atomistic ordering process, 2. Nucleation of Anti Phase Domain and its wetting process and 3. Growth and coalescence process of APD. In order to evaluate gradient energy coefficient term without resorting to empirical means, the possibility of first-principles calcualtion has been explored.

9:30 AM Invited
Kinetic Pathways for Solid State Transformations: *Georges Martin*[1]; [1]CEA, Cabinet du Haut Commissaire, 33 rue de la Fédération, Paris 75015 France

Provided that the enthalpy H({r}) of a set of atoms is known as a function of the position {r} of all the atoms, and that the enthalpy hyper-surface H({r}) exhibits well defined minima separated by barriers of some kT, the classical transition state theory permits to compute the transition probability per unit time, Wij, between two configurations, i and j of the set of atoms. Once the set of the Wij's is known for all possible configurations, Kinetic Monte Carlo techniques generate realistic kinetic pathways for solid state coherent unmixing and/or ordering. Based mainly on broken bounds models for H({r}), but also on cluster expansions, the activity in this field is flourishing. I'll focus on what has been understood recently on the effect of the diffusion mechanism on kinetic pathways, a question of importance for practical metallurgy: - vacancy diffusion mechanism, with conserved and non-conserved vacancies; - monomer versus n-mer solute mobil-

ity; - competition between thermally activated atomic jumps and ballistic jumps in the case of "driven alloys". The link with mean field approximations and phase field models will be briefly discussed at the end.

10:00 AM Break

10:10 AM Invited
Phase Field Method: Microstructure Evolutions and Plasticity: *Alphonse Finel*[1]; Yann M. Le Bouar[1]; Quentin Bronchart[1]; [1]ONERA, LEM (CNRS-ONERA), BP 72, Chatillon 92322 France

The Phase Field Method is extensively used to study the dynamics of microstructures inherited from phase transformations and it has recently been extended to the dynamics of dislocations, and thus to the domain of plastic deformation. We will present recent advances in these two fields. In particular, we will address the problem of fluctuations and time scale for microstructural evolution, as well as the problem of the short range elastic interactions between dislocations.

10:40 AM Invited
Site Preference in Frank-Kasper Intermetallic Compounds: *Marcel H.F. Sluiter*[1]; Alain Pasturel[2]; Yoshiyuki Kawazoe[1]; [1]Tohoku University, Inst. for Matls. Rsch., 2-1-1 Katahira, Aoba-ku, Sendai-shi, Miyagi-ken 980-8577 Japan; [2]CNRS, Laboratoire de Physique et Modelisation des Milieux Condenses, Maison des magistees BP 166, Grenoble-Cedex 09 38042 France

Site occupation in a variety of Frank-Kasper type intermetallic phases has been predicted using density functional electronic structure methods. For the rather limited number of cases where experimental verification is possible, generally good agreement is found. As a number of systems have been examined, the validity of simple rules of thumb can be examined. The applicability of rules based on atomic size and coordination number, on nearest neighbor like and unlike pair counts, and on approximate local point symmetry and valence electron per atom ratios have been evaluated. As a result an amalgam of rules of thumb can be put forward which generally explains the findings obtained so far. Such general rules are expected to be of utility in the thermodynamic modeling for phase diagram calculations.

11:10 AM
Formation Process of Anti-Phase Boundaries in Al5Ti3 Type Superstructure Studied by Monte Carlo Simulation: *Satoshi Hata*[1]; Takayoshi Nakano[2]; Kiyoshi Higuchi[1]; Yousuke Nagasawa[2]; Masaru Itakura[1]; Noriyuki Kuwano[3]; Yoshitsugu Tomokiyo[1]; Yukichi Umakoshi[2]; [1]Kyushu University, Dept. of Appl. Sci. for Elect. & Matls., 6-1 Kasugakouen, Kasuga, Fukuoka 816-8580 Japan; [2]Osaka University, Dept. of Matls. Sci. & Engrg. & Handai Frontier Rsch. Ctr., Grad. Sch. of Engrg., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan; [3]Kyushu University, Advd. Sci. & Tech. Ctr. for Coop. Rsch., 6-1 Kasugakouen, Kasuga, Fukuoka 816-8580 Japan

Al-rich TiAl forms metastable Al_5Ti_3 superstructure in the $L1_0$ matrix. It is expected that Al_5Ti_3 can introduce many types of anti-phase boundaries (APBs) because of its large unit cell. However, most of the APBs observed experimentally are of A-type that is composed of structural units of Al_5Ti_3. The formation process of APBs in Al_5Ti_3 was analysed by Monte Carlo simulation in a two-dimensional Ising lattice. For reproducing the Al_5Ti_3 type ordering in the (002) disordered matrix, effective pairwise interactions reported by Kulkarni were modified. In the early stage of ordering, segments of A-type APBs appear with a higher frequency than those formed by random-nucleation and subsequent bounding of Al_5Ti_3 microdomains. The A-type APBs grow with Al_5Ti_3 domains and sweep out the other types of APBs. Such a formation process is due to quite low energy of A-type APB that creates wrong atomic bonds only at large coordination distances.

The Role of Grain Boundaries in Material Design: Grain Boundary Character

Sponsored by: Materials Processing and Manufacturing Division, ASM/MSCTS-Texture & Anisotropy Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Brent L. Adams, Brigham Young University, Department of Mechanical Engineering, Provo, UT 84602-0001 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA

Wednesday AM Room: 218A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Brent L. Adams, Brigham Young University, Dept. of Mech. Engrg., Provo, UT 84602-0001 USA; Gregory Rohrer, Carnegie Mellon University, Dept. of Matls. Sci. & Engrg., Pittsburgh, PA 15123-3890 USA

8:30 AM Opening Comments

8:35 AM Invited
A New Approach to Grain Boundary Engineering in the 21st Century: *Tadao Watanabe*[1]; [1]Tohoku University, Nanomech., Aramaki-Aza-Aoba 01, Aoba-Ku, Sendai, Miyagi 980-8579 Japan

Grain boundaries can play crucial roles in designing high performance advanced structural and functional materials. They have a large variety of structure-dependent properties and geometrical configurations. A new principle of materials design and development, called Grain Boundary Design/Control or Engineering has been established based on systematic studies of the relationship between grain boundary structure and properties, and the control of grain boundary microstructures. It is strongly required that a large variety and a wide flexibility of grain boundaries should be effectively utilized in future materials design and development. The development of useful processing techniques for grain boundary engineering has been attempted in order to confer desirable property and high performance to a polycrystalline material in the last decade. This paper will present a prospect of grain boundary engineering in the 21st century, referring to a new approach to grain boundary engineering including nanocrystalline materials and the rejuvenation of damaged materials.

9:00 AM Invited
Evolution of Higher Order Correlations in Microstructures During Annealing: *Mukul Kumar*[1]; [1]Lawrence Livermore National Laboratory, 7000 East Ave., L-356, Livermore, CA 94550 USA

Sequential thermomechanical processing consisting of several cycles of moderate strain and high temperature annealing has been shown to systematically modify the topology of the microstructure. The optimization treatments performed on FCC metals and alloys with low stacking fault energies have resulted in microstructures with high fractions of S3n variants and other special boundaries. Concurrently, there are dramatic changes in the triple junction distributions owing to the higher order correlations imposed by crystallography. However, a fundamental question regarding the stability of such microstructures during high temperature excursions under service conditions is still open for debate. EBSD orientation mapping and transmission electron microscopy observations of the deformed and annealed states will be presented. These will be correlated with the changes in the microstructural topology such as the network of random grain boundaries. This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

9:25 AM Invited
Non-Destructive Characterization of Grain Boundaries in 3D: *Dorte Juul Jensen*[1]; Xiaowei Fu[1]; Soren Schmidt[1]; [1]Riso National Laboratory, Ctr. for Fundamental Rsch., Metal Structures in 4-D, Roskilde DK 4000 Denmark

A grain boundary is classified by the crystallographic misorientation across it (3 parameters) and by the grain boundary plane (2 parameters). Additional four parameters describe the precise atomic arrangement at the boundary. Experimentally it is straightforward to determine the misorientation, e.g. by EBSP, and these 3 parameters may be sufficient to understand given materials properties. However in other cases also the grain boundary planes have to be determined. In the present presentation, it is described how all 5 grain boundary parameters can be determined fast and non-destructively for large bulk speci-

mens by 3 Dimensional X-Ray Diffraction Microscopy. Results for Al samples are presented. The precision in the determination is explained and options for improvements are discussed.

9:50 AM Invited
Grain Boundary Plane Textures in Polycrystals: *Gregory S. Rohrer*[1]; David M. Saylor[2]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA; [2]National Institute of Standards and Technology, Matls. Sci. & Engrg. Lab., Gaithersburg, MD 20899 USA

To distinguish one type of grain boundary from another, the values of five independent parameters must be specified. Three parameters describe the lattice misorientation and two parameters describe the normal to the grain boundary plane. Our recent observations of five parameter grain boundary distributions in microstructures that result from extensive grain growth have led to the following conclusions. First, there is significant texture in the space of grain boundary plane orientations, even when there is no significant misorientation texture. Second, low energy interfaces are observed more frequently than higher energy interfaces. Furthermore, for high angle grain boundaries, the anisotropy associated with the grain boundary plane orientation is greater than that associated with the misorientation. The relative amounts of grain boundary anisotropy in several materials (magnesia, strontium titanate, titania, spinel, and aluminum) will be compared and the origin of the texture will be discussed.

10:15 AM Break

10:30 AM
Design of the Grain Boundary Character Distribution in Polycrystals: *Brent Larsen Adams*[1]; [1]Brigham Young University, Mech. Engrg., 435 CTB, Provo, UT 84602-4201 USA

Material properties that depend upon the grain boundary character distribution (GBCD) is the focus of this presentation. Methodology for incorporating the GBCD in the Fourier framework for microstructure design is described. A failure criterion is postulated that requires the fraction of susceptible grain boundaries to exceed the percolation threshold. This criterion is extended to the statistical ensemble, and its dependence upon critical crack length is examined. The relationship with other aspects of microstructure design is also described.

10:55 AM Invited
Length Scales in Grain Boundary Manifolds: *Elizabeth A. Holm*[1]; Erin McGarrity[2]; Jan H. Meinke[2]; Phillip M. Duxbury[2]; [1]Sandia National Laboratories, Dept. 1834, PO Box 5800, MS 1411, Albuquerque, NM 87185 USA; [2]Michigan State University, Dept. of Physics & Astron., Lansing, MI 48824-1116 USA

Although the tools of microstructural analysis - microscopy, image acquisition, computers, and computational science - have advanced, the measures of microstructure remain the traditional planar parameters of quantitative stereology, developed early in this century. While two-dimensional (2D) parameters like grain size, phase fraction, and particle aspect ratio are undeniably important, many properties are controlled by the characteristics of the three-dimensional (3D) grain boundary manifold. For example, the distribution of weak grain boundaries controls the location and roughness of the preferred fracture plane. Likewise, the connectivity of twin-type boundaries governs the corrosion resistance of grain boundary engineered materials. In this paper, we examine critical length scales in grain boundary manifolds, and we derive their relationships to interface properties such as energy, roughness, and connectivity. These new, experimentally accessible length scales provide a more precise tool for microstructural quantification as well as new insight into microstructure/property relationships.

11:20 AM
The Role of Grain Size and Grain Boundary Character on the Creep Behavior of INCONEL Alloy 718: *C. J. Boehlert*[1]; D. Dickmann[1]; S. Civelekoglu[1]; R. C. Gundakaram[1]; N. Eisinger[2]; [1]Alfred University, Sch. of Ceram. Engrg. & Matls. Sci., 2 Pine St., Alfred, NY 14802 USA; [2]Special Metals Corporation, Huntington, WV USA

Grain boundary engineering of metals and alloys is typically accomplished through changing the grain boundary character distribution (GBCD) by thermomechanical processing techniques. The resulting GBCD can have a significant impact on mechanical behavior especially for properties which are significantly influenced by intergranular deformation, such as creep. The GBCD of INCONEL alloy 718 has been evaluated and the results indicate that the grain boundary character is significantly dependent on processing and subsequent heat treatment, where grain size plays an important role. The grain size and GBCD also have a significant impact on the creep behavior, especially within the creep regimes associated with diffusion and grain boundary sliding. This research was supported by the NSF grant DMR-0134789.

Third International Symposium on Ultrafine Grained Materials: Mechanical Properties

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; Terence G. Langdon, University of Southern California, Departments of Aerospace & Mechanical Engineering and Materials Engineering, Los Angeles, CA 90089-1453 USA; Terry C. Lowe, Metallicum, Santa Fe, NM 87501 USA; S. Lee Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright Patterson AFB, OH 45433 USA; Dong H. Shin, Hanyang University, Department of Metallurgy and Material Science, Ansan, Kyunggi-Do 425-791 Korea; Ruslan Z. Valiev, Institute of Physics of Advanced Material, Ufa State Aviation Technology University, Ufa 450000 Russia

Wednesday AM Room: 207A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Michael J. Zehetbauer, University of Vienna, Inst. Matls. Physics, Wien 1090 Austria; Alexei Yu Vinogradov, Osaka City University, Dept. Intelligent Matls. Engrg., Osaka 855-8585 Japan; Carl C. Koch, North Carolina State University, Matls. Sci. & Engrg., Raleigh, NC 27695 USA

8:30 AM Invited
Effect of Processing Techniques on the Mechanical Properties of Nanocrystalline/Ultrafine Grained Metals: *Carl C. Koch*[1]; *Khaled M.S. Youssef*[1]; [1]North Carolina State University, Matls. Sci. & Engrg., 233 Riddick Bldg., 2401 Stinson Dr., Raleigh, NC 27695 USA

Elemental metals with nanoscale and/or ultrafine grain sizes have been prepared by ball milling and compaction of powders and by cold rolling of bulk sheets at liquid nitrogen temperature followed by controlled annealing treatments. The metals studied include an fcc structure metal, Cu, and bcc metals Ta and Fe. The structures/microstructures are characterized by XRD and TEM. Of major importance is the grain size distribution which is obtained using dark field TEM. The mechanical behavior of these materials is probed by microhardness, miniaturized disk bend tests (MDBT), automated ball indentation tests (ABI), and shear punch tests. The results of these experiments are compared with recent work in our laboratory on Zn and research reported in the literature, which suggests that a combination of nanoscale and submicron (or even micron) size grains provides the optimum values of strength and ductility.

8:50 AM Invited
Tough Ultrafine-Grained Metals at Cryogenic Temperatures: *Yinmin Wang*[1]; En (Evan) Ma[1]; Rulsan Z. Valiev[2]; Yuntian T. Zhu[3]; [1]Johns Hopkins University, Matls. Sci. & Engrg., Baltimore, MD 21218 USA; [2]Ufa State Aviation Technical University, Inst. of Physics of Advd. Matls., Ufa 450000 Russia; [3]Los Alamos National Laboratory, Matls. Sci. & Tech., Los Alamos, NM 87545 USA

Ultrafine-grained metals are a new class of materials that in general own a good combination of strength and ductility at room temperature. However, the tensile stress-strain curves of these materials show a small uniform tensile elongation that limits their practical utility. In this talk, our recent experimental findings regarding the tensile properties of ultrafine-grained metals at cryogenic temperatures will be presented. The data of ultrafine-grained copper (fcc, grain size in the range of 190-300 nm), titanium (hcp, 260 nm), and iron (bcc, 200 nm) indicate that a much higher yield strength and a larger uniform tensile elongation are obtained at low temperatures. The mechanisms leading to the coexistence of the high strength and ductility at cryogenic temperatures are discussed. In particular, different from conventional metals, ultrafine-grained fcc metals (Cu and Ni) exhibit obvious temperature dependence of the yield strength, and ultrafine-grained bcc metals (e.g., Fe) show pronounced ductility at 77 K.

9:10 AM Invited
Deformation Behaviors of Ultrafine-Grained Al Alloys Processed by Cryomilling Techniques: *F. A. Mohamed*[1]; B. Q. Han[2]; E. J. Lavernia[2]; [1]University of California, Chem. Engrg. Matls. Sci., Irvine, CA 92697 USA; [2]University of California, Chem. Engrg. Matls. Sci., Davis, CA 95616 USA

Most recently, ultrafine-grained and nanostructured aluminum alloys with grain sizes ranging from 100 nm to 500 nm have been successfully manufactured by consolidation of cryomilled aluminum powders. In the present study, uni-axial deformation behavior was used to investigate deformation mechanisms. Microstructure characteristics and X-ray diffraction patterns were also used in the present study. Characteristics of high strength and low work hardening were observed in deformation of cryomilled nanostructured aluminum alloys. The relationship among processing, microstructural characterization and mechanical deformation mechanisms was discussed.

9:30 AM
Cold-Deformed Cu-Ag Composite with Ultra-Fine Microstructures: *Ke Han*[1]; [1]National High Magnetic Field Laboratory, 1800 E. Paul Dirac Dr., Tallahassee, FL 32309 USA

Co-deformation of in-situ Cu-Ag and Cu-Nb composites produces high-strength-high-conductivity materials. The composites have various applications such as winding high field magnets. The Ag and Nb were chosen to add into Cu because they have different lattice parameter from Cu and have almost no solubility in Cu at room temperature so that additions of Ag or Nb introduce limited decreasing of the conductivity. Co-deformation provides the composites with a strength level as high as 1000MPa. This strength level is significantly higher than that predicted by a law-of-mixture and it is about 1/25 of the theoretical strength of Cu (25000MPa). Moreover, the stress-strain curves indicate internal stresses developed in the materials. This paper reports an investigation of the strengthening mechanisms and internal stresses in Cu-Ag and Cu-Nb composites.

9:45 AM
Compression Tests to Approximate the ECAP Deformation and Further Post-Processing of NanoSPD CP-Ti: *Leonhard Zeipper*[1]; Gerald Gemeinböck[1]; Michael Zehetbauer[2]; Georg Korb[1]; [1]ARC Seibersdorf Research GmbH, Matls. & Production Engrg., A-2444 Seibersdorf Austria; [2]University of Vienna, Inst. of Matls. Physics, Boltzmanngasse 5, A-1090 Vienna Austria

Ultrafine-grained and nanostructured CP-Ti exhibits extraordinary mechanical properties. A tremendous increase in strength and parallel in ductility can be achieved by an increasing number of Equal Channel Angular passes. The in situ stress-strain behaviour on the post-deformation by compression is studied using the Mecking-plot to illustrate different hardening stages. Different crack types nucleate during compression of different SPD pre-strained samples at room temperature and are discussed. Within the equal forming limit, the ECAP process is approximated by uniaxial compression tests at elevated temperatures. Furthermore the tensile behavour is reported and compared with the compressive counterpart. Links to technological post-deformation procedures like rolling and forging are given. Finally, the changing of deformation paths is seen to be a feature for further increasing the nanoSPD materials' properties.

10:00 AM
Effect of Grain Size from Millimeter to Nanometers on the Flow Stress of Low Stacking Fault Energy FCC Metals: Kang Jung[1]; *Hans Conrad*[1]; [1]North Carolina State University, Matls. Sci. & Engrg. Dept., Raleigh, NC 27695-7907 USA

The effect of grain size d on the flow stress of Cu, Ag, Au and Pd consists of three regimes: Regime I (d > ~10^-6m), 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 in Regime III. Dislocation cells occur in Regime I, but are essentially ascent in Regime II. Regime III is characterized by the absence of intergranular dislocations. The rate-controlling mechanism(s) operative in each regime are discussed.

10:15 AM
Fatigue Crack Growth in Ultrafine Grained Al-7.5Mg: *Peter S. Pao*[1]; Harry N. Jones[1]; Jerry C.R. Feng[1]; [1]Naval Research Laboratory, Code 6323, 4555 Overlook Ave. SW, Washington, DC 20375 USA

The fatigue crack growth rates of ultrafine grained (g.s. ~ 0.25 μm) Al-7.5Mg produced by SPD techniques were investigated and were compared to those of powder-metallurgy (P/M) Al-7Mg (g.s. ~ 2 μm) and ingot-metallurgy (I/M) Al-7Mg (g.s. ~ 100 μm). Ultrafine grained Al-7.5Mg was obtained by extruding nanocrystalline particulates, which were prepared by mechanically ball milling spray atomized Al-7.5Mg powders in liquid nitrogen. Fatigue crack growth rates of ultrafine grained Al-7.5Mg are significantly higher than those of P/M Al-7Mg, which, in turn, are significantly higher than those of I/M Al-7Mg. The fatigue crack growth threshold is the lowest in the ultrafine grained Al-7.5Mg, follows by P/M Al-7Mg, and is the highest in I/M Al-7Mg. The higher fatigue crack growth rates and lower thresholds in ultrafine grained Al-7.5Mg may be attributed to the much smoother fracture surface morphology and lower roughness induced crack closure.

10:30 AM Break

10:40 AM Invited
Deformation Behavior of Ultrafine-Grained Iron Processed by Equal-Channel-Angular Pressing: *E. J. Lavernia*[1]; B. Q. Han[1]; F. A. Mohamed[2]; [1]University of California, Chem. Engrg. & Matls. Sci., Davis, CA 95616 USA; [2]University of California, Chem. Engrg. & Matls. Sci., Irvine, CA 92697 USA

In the present study, the microstructural evolution during pressing and the deformation behavior of ultrafine-grained pure Fe processed via equal-channel angular pressing (ECAP) was investigated by means of transmission electron microscopy, tensile and compressive tests. Intensive dislocation-cell blocks as well as ultrafine grains were observed after severe plastic deformation. Because of the presence of ultrafine-grained microstructure and non-equilibrium grain boundaries, the materials display a distinct mechanical behavior from their counterpart unprocessed materials. Several mechanical issues including high strength, necking deformation and low ductility were discussed.

11:00 AM Invited
Plastic Flow and Mechanical Behavior of Some Mg- and Al-Alloys Deformed by Equal-Channel-Angular Pressing: *Peter K. Liaw*[1]; Grigoreta Mihaela Stoica[1]; L. J. Chen[2]; E. A. Payzant[3]; S. R. Agnew[4]; C. Xu[5]; T. G. Langdon[5]; D. E. Fielden[1]; [1]University of Tennessee, Matl. Sci. & Engrg., 323 Dougherty Bldg., Knoxville, TN 37996-2200 USA; [2]Shenyang University of Technology, Shenyang 110023 China; [3]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; [4]University of Virginia, Matl. Sci. Engrg., Charlottesville, VA 22904 USA; [5]University of Southern California, Dept. Aeros. & Mech. Engrg. & Matl. Sci., Los Angeles, CA 90089 USA

Equal-Channel-Angular Pressing (ECAP) is an efficient method of severe plastic deformation for use with metals and composites to produce novel mechanical properties. ECAP is examined as a deformation mode derived from orthogonal cutting. Some practical approaches to evaluate the induced strain during ECAP, including strain inhomogeneity, are discussed. The deformation characteristics of the magnesium alloy, ZK60, aluminum alloy, 6061Al, and aluminum composite, 6061Al/Al2O3, are reviewed. The results of grain refinement, as well as its influence on the material behavior under tensile and cyclic loading, are presented. After processing by ECAP, ZK 60 has a ductility that increases by 2-3 times, and the 6061Al/Al2O3 composite exhibits higher strength and lower ductility by comparison with the unpressed materials.

11:20 AM
Fatigue Properties of Ultra-Fine Grain Materials Fabricated by Severe Plastic Deformation - The Effect of Strain Path: *Alexei Yu. Vinogradov*[1]; [1]Osaka City University, Dept. of Intelligent Matls. Engrg., Sumiyoshi-ku, Sugimoto 3-3-138, Osaka 855-8585 Japan

The experimental results concerning the fatigue behavior of ultrafine grain materials fabricated by severe plastic deformation (SPD) are reviewed. The possibility to significantly enhance the fatigue performance of meals and alloys after grain reduction down to nano-scopic scale is demonstrated. A special attention is paid to the influence of strain path and the amount of strain imposed during manufacturing on fatigue life and to the role of alloying and precipitation hardening in fatigue of SPD metals. The key importance of grain boundaries in fatigue damage of SPD metals is argued. The main reason limiting the fatigue life under constant stress or strain cyclic loading is supposed to be related to structural instability resulting from severe plastic deformation. Possibilities to enhance the fatigue performance of SPD materials are explored via structural stabilization through post-processing heat treatment, alloying and precipitation hardening.

11:35 AM
Mechanical Behaviour of IF Steel Processed by Equal Channel Angular Pressing: *Joke De Messemaeker*[1]; Bert Verlinden[1]; Jan Van Humbeek[1]; [1]K.U. Leuven, Dept. of Metall. & Matls. Engrg., Kasteelpark Arenberg 44, Heverlee 3001 Belgium

The evolution of the yield stress and hardening rate measured in a compression test of IF steel processed by equal channel angular pressing (ECAP) at 200°C via route B_A, is followed up to a von Mises equivalent strain of 9. At the highest strain the dependence of the mechanical behaviour on strain path is established by comparison of the 4 classical routes A, C, B_A and B_C. The evolution and strain path dependence are linked to TEM observations of the microstructure, and the compression test data is compared with hardness measurements made on the plane of TEM observation. After annealing at 500°C for

3h the recovered microstructure, showing slight grain growth, was quantitatively analysed by EBSD. The change in yield stress and hardening rate with respect to the as-deformed samples is discussed.

11:50 AM Invited
Mechanical Properties of SPD Metals and Their Physical Backgrounds: *Michael Josef Zehetbauer*[1]; [1]University of Vienna, Inst. of Matls. Physics, Boltzmanngasse 5, Wien 1090 Austria

This contribution reviews the exceptional mechanical properties of SPD metals (strength, ductility, and fracture toughness) and discusses the physical processes behind. Most investigations have been done on strength, showing that the hydrostatic pressure of the SPD method governs the scale of grain structure and thus the resulting strength. Since the same is true for the deformation temperature, a model has been successfully suggested by the author which correlates the resulting grain size with the extent of dislocation annihilation. As for the ductility of SPD materials, hitherto only qualitative ideas exist which either consider the number of high angle boundaries, or the high density of deformation induced vacancies both increasing the probability for grain boundary sliding. For the fracture toughness, an ultrafine grained structure should be beneficial, too, because of an increase of total fracture energy, but this has not always been confirmed by experiment.

12:10 PM
Mechanical Property Characterization Using Shear - Punch Tests: *Ramesh Kumar Guduru*[1]; *Ronald Otto Scattergood*[1]; [1]North Carolina State University, Matls. Sci. & Engrg., PO Box 7907, Raleigh, NC 27695-7907 USA

The evaluation of mechanical properties from small specimens is critical when the availability of material is limited. Shear punch testing is one such miniaturized specimen test technique from which one can obtain mechanical properties like yield strength, ultimate strength and strain hardening exponents. A shear punch testing procedure was standardized in our laboratory and the mechanical properties for different materials; mild steel, pure Al, pure Zn, austenitic stainless steel, Al 6061 alloy and martensitic stainless steel were evaluated. A linear correlation for the yield and ultimate strength was established between data obtained from shear punch and uni-axial tensile tests. The dependency of the shear strength (yield and ultimate) was studied as a function of the sample thickness and die clearance for medium and high strength materials. The effect of die/punch clearance for a given thickness was also investigated. Results will be compared for conventional and nanocrystalline materials.

12:25 PM
Effect of Grain Size on Fatigue Properties for Electric Refined Iron: *Chitoshi Masuda*[1]; [1]Waseda University, Kagami-Memorial Lab. for Matls. Sci. & Tech., 2-8-26, Nishiwaseda, Sinjuku, Tokyo Japan

The fatigue strength, fatigue crack propagation properties, fatigue fracture mechanisms have been studied for many metallic materials. Especially for steels many effects have been discussed. Recently the steels having fine grain size near 1 mm have been developed and their tensile strengths have increased with decreasing the grain size. The tensile strengths have been linearly related with the inverse square root of grain sizes, that is, the Hall-Petch relationship for welded steels up to about the tensile strength of 800MPa. The fatigue strengths and fatigue fracture mechanism for welded steels having fine grain size have not so cleared. In this study, the fatigue strength and fatigue fracture mechanisms are fundamentally discussed with the cast steel using electric refined iron. The ingot was forged and rolled at a medium temperature. Finally the iron was drawn into 4mm in diameter at room temperature. The average grain size was about 3 mm. The fatigue test was performed by rotating bending machine at room temperature at the stress ratio of -1. Fatigue strength for electric refined cast iron was about 300 MPa at the number of cycles to failure of 107. For S25C carbon steel normalized and S35C carbon steels(12Heats) quenched and tempered at 600°C(11 Heats), the average fatigue strengths were about 243 and 385 MPa, respectively. The electric refined iron has medium fatigue strength between S25C and S35C carbon steels. The reason of increase of the fatigue strength for electric refined iron would be caused by the decrease of the grain size of material. The fatigue crack initiation mechanism for fine grain size steels have not yet been discussed. The fatigue crack initiates on the specimen surface by slip mechanism and propagates into the outermost one grain by mode II, III or their combined mode(Stage I type crack). After pass through the outermost grain the crack direction change into the normal direction of stress axis(Stage II type crack). If the grain size is fine, the Stage I type crack propagation mechanism initiated from the specimen surface is the same as for slip mechanism proposed by Laird, the Stage I type crack is quickly change its direction and the fatigue strength would not be expected high. But the fatigue strength for electric refined iron having fine grain size is very high. Therefore the another fatigue crack initiation mechanism would be operated.

5th Global Innovations Symposium: Trends in LIGA, Miniaturization, and Nano-Scale Materials, Devices and Technologies: Manufacturing and Evaluation of Layered Nano-Scale Materials

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Powder Materials Committee, MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS), MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), MPMD/EPD-Process Modeling Analysis & Control Committee, MPMD-Surface Engineering Committee, MPMD-Shaping and Forming Committee, MPMD-Solidification Committee

Program Organizers: John E. Smugeresky, Sandia National Laboratories, Department 8724, Livermore, CA 94551-0969 USA; Steven H. Goods, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Sean J. Hearne, Sandia National Laboratories, Albuquerque, NM 87185-1415 USA; Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA

Wednesday PM Room: 202B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Dave F. Bahr, Washington State University, Mech. & Matls. Engrg., Pullman, WA 99164-2920 USA; Steven H. Goods, Sandia National Laboratories, Dept. 8725, Livermore, CA 94551-0969 USA; Richard G. Hoagland, Los Alamos National Laboratory, Matls. Sci. & Tech. Div., Los Alamos, NM 87545 USA

2:00 PM
Nanoimprint Technology: A Low Cost, Mass Manufacture of Nanostructures: *Khershed P. Cooper*[1]; [1]Naval Research Laboratory/ Office of Naval Research, Matls. Sci. & Tech. Div., Code 6325, 4555 Overlook Ave. SW, Washington, DC 20375-5320 USA

Nanomanufacturing seeks to exploit new opportunities from the manipulation of physical, mechanical, chemical and biological processes at the nanoscale in order to produce new materials with unique properties and new devices with unique functionalities. One nanomanufacturing technology that is receiving attention is nanoimprinting. It involves using a rigid or flexible mold to imprint nano-scale forms into resist-coated materials. After reactive ion etching and lift-off, nanostructures are produced in silicon and other materials. The technique is now capable of producing simple structures, such as gratings and gates. Nanoimprinting is being promoted as a means to break the barrier that "traditional" lithography will face as the demand increases for smaller (<100nm) feature and device sizes. A variant is laser-assisted direct imprinting. It consists of directly imprinting nanostructures in rapidly-melted surfaces. This paper will review the requirements for nanomanufacturing, the current status of nanoimprinting, and materials-related issues facing techniques such as direct imprinting.

2:20 PM
Advances in Assembling Microsystems with Adhesives: *John A. Emerson*[1]; Neville R. Moody[2]; Rachel K. Guinta[1]; [1]Sandia National Laboratories, MS 0958, Albuquerque, NM 87185-0958 USA; [2]Sandia National Laboratories, MS 0969, Livermore, CA 94551-0969 USA

As electronic and optical microsystems reach the micro- and nano-scales, efficient assembly and packaging requires the use of adhesive bonds. A primary issue is that as bondline thickness decreases, knowledge of the stability and dewetting dynamics of thin adhesive films is important for obtaining robust, void-free adhesive bonds. While researchers have studied dewetting dynamics of thin films of model, non-polar polymers, little experimental work has been done regarding dewetting dynamics of thin adhesive films, which exhibit more complex short-range interactions. Also, the adhesion of such submicron thick bond lines is found to depend strongly on the strength of the interfacial bonding, which in turn, depends on the interfacial chemistry. In this presentation we will report on tests to date showing that the interfacial fracture toughness of thin epoxy films attached to aluminum substrates decreases with film thickness, reaching a lower limiting value of 1.5 Jm-2.

2:40 PM
White Beam Diffraction Analysis of Plastic Deformation in 2D Systems: *Rosa I. Barabash*[1]; Gene E. Ice[1]; Nobumichi Tamura[2]; Bryan C. Valek[3]; John C. Bravman[3]; Ralph Spolenak[4]; Jim Patel[2]; [1]Oak Ridge National Laboratory, Metals & Ceram., MS-6118, One Bethel Valley Rd., Oak Ridge, TN 37831-6118 USA; [2]Berkeley National Laboratory, Advd. Light Source, Cyclotron Rd., Berkeley, CA 94720 USA; [3]Stanford University, Matls. Sci. & Engrg., Stanford, CA 94305 USA; [4]Max Planck Institut fur Metallforschung, Heisenbergstrasse 3, Stuttgart D-7056 Germany

White beam X-ray microdiffraction is particularly well suited to the study of salient features of mechanical behavior when the characteristic size associated with microstructure and/or deformation approaches the nano-scale. The mechanical properties and deformation mechanisms of materials at very small length scales gain additional features comparing with bulk deformation microstructures. Surfaces and interfaces create additional constraints and change the conditions for dislocation formation and movement in 2D systems. A statistical description of diffraction from dislocations in 2D systems is presented. White beam diffraction analysis of several distinct examples of dislocation structures in 2D systems is discussed.

3:00 PM
Stress Evolution During Electrodeposition of Ni Thin Films: *Sean J. Hearne*[1]; Steve C. Seel[1]; Jerrold A. Floro[1]; Chris Dyck[1]; Wenjun Fan[2]; S. R.J. Brueck[2]; [1]Sandia National Laboratories, Nanostruct. & Semiconductor Physics Dept., PO Box 5800 MS 1415, Albuquerque, NM 87185-1415 USA; [2]University of New Mexico, Albuquerque, NM 87106 USA

The majority of thin films used in industrial applications grow via an islanding (Volmer-Weber) mode, where discrete islands grow until they coalesce into a continuous film. It is commonly accepted that a tensile stress is induced when these islands coalesce. However, prior to this work there had been no systematic demonstration of the functional stress generation behavior associated solely with the coalescence process. To address this Ni films were electroplated onto patterned substrates to obtain a direct comparison of the experimentally-measured tensile stress due to island coalescence with theoretical predictions. This allowed for the systematic variation of island size and geometry while avoiding stochastic island coalescence that have plagued previous measurements obtained over the last 30 years. In the presentation, we will compare our experimentally measured results with those predicted by a Hertzian contact model recently proposed by Freund and Chason1, and with finite element models. This work was partially supported by the DOE Office of Basic Energy Sciences and by the ARO/MURI in Deep Subwavelength Optical Nanolithography. Sandia is a multiprogram laboratory of the United States Department of Energy operated by Sandia Corporation, a Lockheed Martin Company, under contract DE-AC04-94AL85000. 1 - L.B. Freund, E. Chason, JAP 89, 4866 (2001).

3:20 PM
Low Stress, High Reflectivity Thin Films for MEMS Mirrors: *David P. Adams*[1]; [1]Sandia National Laboratories, Thin Film, Vacuum & Pkgg. Dept., PO Box 5800, Albuquerque, NM 87185 USA

The need for optical MEMs devices that handle powers approaching 1 Watt has motivated this research of high-reflectivity, low-stress thin films and their performance. While methods are established for depositing highly reflective multilayers for different wavelengths of interest, it remains challenging to apply similar films to MEMs devices that are ~2 microns thick and hundreds of microns in width. Towards this end, we discuss the properties of low residual stress layers that have been developed for MEMs applications. Films discussed in this talk include Au with various adhesion promotion layers, Si/SiN Bragg reflectors and additional high damage threshold materials. We discuss several issues including: the mechanical properties of sputter deposited layers, stress changes that occur over months and the relationship(s) to processing, application to MEMs devices and performance during laser irradiation. In conclusion, we demonstrate the ability to maintain lambda/40 flatness (lambda= 1319nm) for mirrors as large as 500 microns.

3:40 PM Break

4:00 PM
Deformation Recovery Processes of Nano Indents Made in Erbium Hydride Thin Films: *James P. Lucas*[1]; Neville R. Moody[2]; [1]Michigan State University, Chem. Engrg. & Matl. Sci., 3526 Engrg. Bldg, E. Lansing, MI 48824-1226 USA; [2]Sandia National Laboratories, Livermore, CA 94551 USA

Deformation recovery of small indents in thin erbium hydride (ErH) films made using the nano indenter was investigated by scanning probe microscopy (SPM). The thickness of the ErH films on sapphire was nominally 1200 angstroms. The degree of recovery was determined by measuring the depth change of the indent impression with time and temperature subsequent to initial indentation of the film. By relating the rate of deformation recovery via the change in surface curvature of the bottom of the indent along with the Gibbs-Thompson relation-

ship, surface diffusivity has been assessed in soft viscoelastic materials. A similar approach is taken in assessing deformation recovery processes in hard ErH films and will be presented.

4:20 PM
Improved Hydrogen Sensing Characteristics of Nanocrystalline Doped Tin Oxide Sensor at Lower Temperature: *Satyajit Shukla*[1]; Sudipta Seal[1]; Lawrence Ludwig[2]; Clyde Parish[2]; [1]University of Central Florida, Mech. Matls. Aeros. Engrg. (MMAE) & Advd. Matls. Procg. & Analysis Ctr. (AMPAC), Engrg. # 381, 4000 Central Florida Blvd., Orlando, FL 32816 USA; [2]National Aeronautics and Space Administration, John F. Kennedy Space Ctr., Kennedy Space Ctr., FL 32899 USA

Nanocrystalline tin oxide semiconductor thin film is coated on Pyrex glass (silica) substrate using the sol-gel dip-coating technique. The thin film is characterized using different analytical techniques such as scanning electron microscopy (SEM), x-ray photoelectron spectroscopy (XPS), atomic force microscopy (AFM), and high-resolution transmission electron microscopy (HRTEM). The HRTEM sample preparation is done using focused ion-beam (FIB) milling technique. Under given processing conditions, tin oxide thin film having thickness 100-150 nm and nanocrystallite size 6-8 nm is obtained. The nanocrystalline tin oxide thin film sensor is doped with 6.5 and 13 mol % indium oxide to enhance hydrogen sensitivity at lower operating temperatures. The effect of calcination and operating temperatures, chamber pressure, electrode design, and the exposure to ultraviolet radiation on hydrogen gas sensitivity, the response and the recovery time, is systematically investigated. Models have been developed to explain low temperature hydrogen gas sensitivity of doped nanocrystalline tin oxide sensor.

4:40 PM
Nanoscale Mechanical Characterization of Silver Nanowires and Cu2O Nanocubes: *Xiaodong Li*[1]; Hongsheng Gao[1]; Catherine J. Murphy[2]; Linfeng Gou[2]; K. K. Caswell[2]; [1]University of South Carolina, Dept. of Mech. Engrg., 300 Main St., Columbia, SC 29208 USA; [2]University of South Carolina, Dept. of Chem. & Biochmst., 631 Sumter St., Columbia, SC 29208 USA

The hardness and elastic modulus of silver nanowires and Cu2O nanocubes was measured using a nanoindenter in conjunction with an atomic force microscope (AFM). The indentation hardness and elastic modulus values were compared with the bulk materials. Nanoscaole deformation behavior was studied by in situ AFM imaging of the indents. An array of nanoscale indents was successfully made on the silver wires and Cu2O cubes by directly indenting them. The shape and size of the indents are controllable. The nanoindentation approach permits the direct machining of a single silver nanowire and a Cu2O nanocube without complications of conventional lithography.

5:00 PM
Carbon Nanotubes and Other Fullerene-Related Nanocrystals in the Environment: A TEM Study: *L. E. Murr*[1]; J. J. Bang[1]; E. V. Esquivel[1]; [1]University of Texas, Metallurgl. & Matls. Engrg., 500 W. Univ. Ave., El Paso, TX 79968-0520 USA

We have discovered that carbon nanotubes and other fullerene-related nanocrystals are pervasive in the atmosphere - both indoor and outdoor. In fact, these carbon nanostructures have been observed even in a 10,000 year-old ice core sample, which is an indication that they have existed naturally in antiquity. Controlled experiments with methane flames (methane burning with air) has allowed a great variety of carbon nanocrystals and tubes to be examined by collecting complex aggregates on transmission electron microscope (TEM) grids coated with a thin formvar/carbon support substrate films in a specially designed thermal precipitator. These studies serve as a frame of reference for the examination of hundreds of other nanoparticulate examples collected randomly in the environment and observed in the TEM. Carbon/graphitic nanotubes and nanocrystals are observed in graphite brake-lining debris and air sampling near interstate highways; and may account in part for an estimated 10 million tons of brake-lining debris created in the U.S. alone over the past decade. The health implications, especially those related to respiratory illnesses are of course currently unknown, but a whole new global perspective on atmospheric nanoparticles is becoming apparent in light of these TEM observations over the past 2 years.

Advanced Materials for Energy Conversion II: Metal Hydrides IV - Dynamics of Metal Hydrides, and Tritium Gettering

Sponsored by: Light Metals Division, LMD-Reactive Metals Committee
Program Organizers: Dhanesh Chandra, University of Nevada, Metallurgical & Materials Engineering, Reno, NV 89557 USA; Renato G. Bautista, University of Nevada, Metallurgical and Materials Engineering, Reno, NV 89557-0136 USA; Louis Schlapbach, EMPA Swiss Federal, Laboratory for Materials Testing and Research, Duebendorf CH-8600 Switzerland

Wednesday PM — Room: 203A
March 17, 2004 — Location: Charlotte Convention Center

Session Chairs: G. Louis Powell, BWXT Y-12, Y-12 National Security Complex, Oak Ridge, TN 37831-8096 USA; Arndt Remhof, Ruhr Universität Bochum, D-44780 Bochum, Germany; Jeff LaCombe, University of Nevada, Metallurgl. & Matls. Engrg., Reno, NV 89557 USA

2:00 PM Keynote
Visualization of the Refraction-Like Behavior of Diffusion Fronts in Solid State Diffusion: *Arndt Remhof*[1]; [1]Ruhr Universität Bochum, D-44780 Bochum Germany

The large mobility of hydrogen in metals make metal hydrides ideal systems for investigations of diffusion fronts and waves in solids. The diffusion coefficient, which determines the velocity of the diffusing hydrogen ions, can be varied over a wide dynamical range. In order to study the analogies (or dissimilarities) between hydrogen diffusion waves and photon diffusion in turbid media, heterostructures with locally varying diffusion constants have to be realized. With planar structures such as interfaces or "prisms" one can investigate the behaviour of hydrogen diffusion fronts when they encounter these structures. The applicability of Snell's law to this diffusive system will be discussed. The distribution of hydrogen is visualized by means of an Y switchable mirror layer. Our measurements are in good agreement with numerical simulations.

2:30 PM Plenary
Separation of Small Amounts of Tritium from Nitrogen and Argon Gas Streams: *Joseph Raymond Wermer*[1]; David W. Howard[2]; [1]Los Alamos National Laboratory, Tritium Sci. & Engrg. Grp., MS C348, PO Box 1663, Los Alamos, NM 87545 USA; [2]Westinghouse Savannah River Company, Hydrogen Tech. Sect., Bldg. 735-11A, Savannah River Site, Aiken, SC 29808 USA

Traditional methods of removing small quantities of tritium from nitrogen or argon streams have involved oxidizing the hydrogen isotopes to form water, absorption of the tritiated water on a solid absorbent (typically molecular sieve), and disposal of the absorbent as solid radioactive waste. The handling concerns with tritiated water (HTO) as well as the cost of recycle or disposal has prompted new technology development. Recent advances in tritium processing technology has focused on methods of removing very small amounts of tritium from nitrogen or argon gas streams which 1) have high efficiency, 2) do not produce oxides of hydrogen, and 3) can be easily regenerated. Both palladium diffusers and metal getter technologies, or a combination of both, have been employed to solve this problem. In the metal getter area, a large effort has been underway for some time to find suitable materials that are compatible with the carrier gas streams.

3:00 PM Keynote
Relations Between Characteristics of Oxidation Overlayers and Hydride Development on Polycrystalline Uranium Surfaces: *M. Brill*[1]; J. Bloch[1]; Y. Ben-Eliyahu[1]; D. Hamawi[1]; T. Livneh[1]; M. H. Mintz[2]; [1]Nuclear Research Center Negev, PO Box 9001, Beer Sheva 84190 Israel; [2]Ben Gurion University of the Negev, Dept. of Nucl. Engrg., PO Box 653, Beer Sheva 84105 Isreal

The initial stages of hydrides development on hydride-forming metal surfaces are significantly affected by the chemical-structural properties of the oxidation overlayers that usually coat those surfaces. In the present study, controlled oxidation procedures were utilized to grow such thin (100-200Å) overlayers on polycrystalline uranium. The hydrides precipitation and growth kinetics were continuously monitored on these samples utilizing Hot-Stage-Microscopy (HSM). Correlations between certain properties of oxide layers and the hydride development kinetics were evaluated. It turned out that high-

temperature, low pressure oxidation processes, yield significantly more protective layers than ambient oxidation. Utilizing X-Ray Diffraction (XRD) and Raman spectroscopy it is concluded that such high temperature oxidation produces surface dioxide that is less defected than native oxide formed on uranium under ambient conditions. For a given high temperature oxidation procedure, the protective ability of the oxide depends on the oxygen exposure time, passing through a maximum. The occurrence of such a maximum is accounted for by the texture-thickness varieties of the layers.

3:25 PM Keynote
Effects of Surface Conditions and Thermomechanical Processing on Uranium Hydride Kinetics: *David F. Teter*[1]; Robert J. Hanrahan[1]; [1]Los Alamos National Laboratories, MST6, Bikini Atoll Rd., MS G770, Los Alamos, NM 87545 USA

The nucleation kinetics of uranium hydride depend strongly on the surface condition of the metal. In most bulk hydriding kinetics measurements, the surface is either pre-hydrided or activated to remove the nucleation barrier so that repeatable reaction kinetics are measured. In the present study, the effect of the surface condition of the metal on the hydriding kinetics was examined. Oxidized uranium rods with varying thicknesses were exposed to hydrogen to study the nucleation of hydride pits. The time to nucleation was found to increase with increasing oxide thickness. Also the nucleation time was dramatically reduced when the sample was vacuum outgassed even though the oxide thickness was unchanged. These observations are consistent with diffusion of hydrogen through the oxide layer being the rate-limiting step for hydride nucleation. Results of the effects of flow stress on the hydriding kinetics of uranium wires will also be presented.

3:50 PM Break

4:05 PM Plenary
Reaction of Oxygen With Uranium Hydride: *George L. Powell*[1]; [1]BWXT-Y12, LLC, Tech. Dvlp., PO Box 2009, Bear Creek Rd., Oak Ridge, TN 37831-8096 USA

Kinetics of the O_2 with UH_3 sponsored by DOE's National Spent Nuclear Fuels Program through INEEL was measured to deduce a defendable model for this reaction. The sample was located in an infrared gas cell with a mass spectrometer. The U was reacted isobarically with H_2 to yield UH_3 that was further characterized by surface sorption and desorption of H_2. Subsequent experiments used O_2 expansion to effect the reaction. The production and sorption of H_2 on the UH_3 yielded the reaction kinetics at low O_2 pressures and balanced the chemical equation for the reaction. Infrared gas analysis excluded H_2O as a reaction product. Light emission synchronized with the O_2 gas transfer and subsequent mass spectrometric analyses showed the quenching of the reaction by H_2. *Managed by BWXT Y-12, LLC for the U.S. Department of energy under contract DE-AC05-00OR22800.

4:35 PM Invited
Thermal Desorption Measurements of Hydrogen on Uranium Metal Surfaces: *Quirinus G. Grindstaff*[1]; [1]BWXT-Y12, LLC, Tech. Dvlp., PO Box 2009, Bear Creek Rd., Oak Ridge, TN 37831-8096 USA

Environmental corrosion of uranium metal produces an oxide film in which the oxygen/water/hydrogen "equilibrium" within the film regulates its growth. At high temperatures, this reaction produces hydrogen that absorbs into the bulk metal affecting subsequent mechanical properties (ductility) upon cooling. At low temperatures and in closed systems, this residual hydrogen in the oxide film may result in the formation of uranium hydride pits, which subsequently may lead to external hydrogen embrittlement, or fires upon exposure to air. Thermal desorption experiments using mass spectroscopy to monitor the rate that hydrogen desorbs from uranium coupons during heating in vacuum is being pursued as a method of determining the nature and distribution of hydrogen in and on uranium. In particular, the thermal desorption rates are being used to discriminate between hydrogen distributed within the metal, hydrogen as uranium hydride on the metal surface, and as other hydrogen species in the uranium oxide film. *Managed by BWXT Y-12, LLC for the U.S. Department of energy under contract DE-AC05-00OR22800.

5:00 PM Invited
Reaction of Hydrogen With an Organic Hydrogen Getter: *George L. Powell*[1]; [1]BWXT-Y12, LLC, Tech. Dvlp., PO Box 2009, Bear Creek Rd., Oak Ridge, TN 37831-8096 USA

DEB (1,4 bis (phenyl ethynyl) benzene typically mixed with 25 wt.% C-1 wt.% Pd) are made into organic hydrogen getters in porous pellet form by Honeywell Federal Manufacturing and Technologies Kansas City Plant. The hydrogen gettering reactions are very effective, and irreversible since the hydrogen gas is converted into an organic hydrocarbon. A simple two-volume system was used to incrementally titrate DEB pellets resulted in a series of pressure drop curves, from which the reaction rate was determined as a function of H2 pressure, DEB reaction extent, and temperature. Experimental methods are described for obtaining the measurements along with data analysis for reducing the pressure decay curves to a four-dimensional reaction rate model. *Managed by BWXT Y-12, LLC for the U.S. Department of energy under contract DE-AC05-00OR22800.

Advanced Materials for Energy Conversion II: Magnetic Materials & Hydrogen Permeation

Sponsored by: Light Metals Division, LMD-Reactive Metals Committee
Program Organizers: Dhanesh Chandra, University of Nevada, Metallurgical & Materials Engineering, Reno, NV 89557 USA; Renato G. Bautista, University of Nevada, Metallurgical and Materials Engineering, Reno, NV 89557-0136 USA; Louis Schlapbach, EMPA Swiss Federal, Laboratory for Materials Testing and Research, Duebendorf CH-8600 Switzerland

Wednesday PM Room: 204
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Jay Keller, Sandia National Laboratory, Livermore, CA 94550 USA; Shin-ichi Orimo, Tohoku University, Inst. for Matls. Rsch, Sendai, Miyagi 980-8577 Japan; Renato G. Bautista, University of Nevada, Metallurgl. & Matls. Engrg., Reno, NV 89557-0136 USA

2:00 PM Keynote
Control of Magnetocaloric Effects by Hydrogen Absorption into La(FexSi1-x)13 Magnetic Refrigerants: *Kazuaki Fukamichi*[1]; Asaya Fujita[1]; [1]Tohoku University, Dept. of Matls. Sci., Grad. Sch. of Engrg., Aoba-yama 02, Sendai, Miyagi 980-8579 Japan

Instead of conventional gas refrigerations, we are yearning for the development of new-type high performance refrigerants because of energy efficiency and environmental safety. Recently, we have developed promising magnetic refrigerant compounds $La(Fe_xSi_{1-x})_{13}$ ($0.86 \leq ?? \leq 0.90$) which show the first-order of the itinerant-electron metamagnetic transition from the paramagnetic state to the ferromagnetic state in applied magnetic fields. These compounds exhibit large values of the isothermal magnetic entropy change ΔS_m and the adiabatic temperature change ΔT_{ad} in the vicinity of the Curie temperature $T_C \sim 190K$. Controlling T_C of $La(Fe_xSi_{1-x})_{13}H_y$ by absorbing hydrogen, we can control easily T_C and also the working temperature covering room temperature. Furthermore, thermal conductivity and diffusivity of the present compounds are excellent, comparable tho se of Gd metal reported as one of the candidates for magnetic refrigerants. Finally, it should be emphasized that the elements of the present compounds are very cheap economically and also completely harmless for human life.

2:30 PM Keynote
Advances in Magnetic Cooling: *Karl A. Gschneidner*[1]; Vitalij K. Pecharsky[1]; [1]Iowa State University, Ames Lab., Ames, IA 50011-3020 USA

Magnetic refrigeration can be cost effective and save considerable energy (20 to 30%) over conventional gas compression technology. It is also an environmentally friendly technology, eliminating ozone depleting chemicals (CFCs), green house gases, (HCFCs and HFCs) and hazardous chemicals (NH_3). In 1997 Astronautics Corp. of America and Ames Laboratory demonstrated that magnetic refrigeration is a viable technology for near room temperature applications, such as large scale building air conditioning, refrigeration/freezer food processing plants and supermarket chillers. Since then seven other magnetic refrigerators have been reported to be operational - a second Astronautics Corp. of America refrigerator, three by the Japanese, and one each in Canada, China and France. Most of the recent devices use permanent magnets as the magnetic field source, and thus are aimed at the consumer markets for household refrigerator/freezers and air conditioners, and automotive climate control. The future prospects of magnetic cooling are quite favorable.

2:55 PM
Rare-Earth Free Magnetostrictive Fe-Ga-X Alloys for Actuators and Sensors: Pinai Mungsantisuk[1]; Robert P. Corson[1]; *Sivaraman Guruswamy*[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

In our earlier work, we have shown that rare-earth free bcc Fe-Ga based alloys have an excellent combination of large low-field room temperature magnetostriction, good mechanical properties, and low hysteresis. These alloys are very attractive in numerous energy conversion device applications such as acoustic sensors and generators, linear motors and actuators. This paper examines the influence of partially substituting Ga in FeGa alloys with Al, Si, Ge, and Sn on the magnetostrictive behavior of FeGa based alloys. Magnetic, magnetostrictive, and elastic properties of the various ternary alloys are presented. It is shown that substitution of Ga with Al can be made in FeGa alloys in certain composition ranges without a reduction in magnetostriction, and the addition of Si, Ge, and Sn results in a decrease of magnetostriction. The FeGaAl ternary alloy maintains all the attractive features in the binary FeGa alloys. Work supported by NSF Grant #0241603.

3:20 PM Break

3:40 PM Invited
Hydrogen Permeation of Ternary Ti-Ni-Nb Alloys: *Kiyoshi Aoki*[1]; Kazuhiro Ishikawa[1]; Kunihiko Hashi[1]; Takeshi Matsuda[1]; [1]Kitami Institute of Technology, Matls. Sci., Koen-chyo, Kitami, Hokkaido 090-8507 Japan

Hydrogen permeation characteristics of ternary Ni-Ti-Nb alloys were investigated in the pressure range of 0.2-0.97 MPa on the upstream side and the temperature range of 523-673 K. Hydrogen permeability of the B2-NiTi phase is 10-10 [mol H2 m-2 s-1 Pa-0.5] figures and that of the Ni30Ti31Nb39 alloy is1.93x10-8 [mol H2 m-2 s-1 Pa-0.5] at 673 K. The Ni30Ti31Nb39 alloy consists of the primary phase, bcc-NbTi, and the eutectic NiTi-NbTi alloys. The NiTi phase plays a major role in prevention of hydrogen brittleness for the NbTi phase, and the NbTi phase contributes mainly to the hydrogen permeation of this alloy.

4:05 PM
Hydrogen Permeation in Nickel Based Alloys: *Joshua H. Lamb*[1]; Venugopal Arjunan[1]; Dhanesh Chandra[1]; [1]University of Nevada, Metall. & Matls. Sci., 1664 N. Virginia St., MS 388, Reno, NV 89557-0136 USA

The hydrogen permeation properties of the nikel based alloy C-22 were examined. The alloy C-22 has been chosen to play a critical role in the storage of high level nuclear waste at the Yucca Mountain High Level Waste Repository. The expected humid enviornment within the waste repository has the potential for creating a cathodic charge on the surface of materials to be used in the facility. The presence of a cathodic charge stimulates the ionization of hydrogen, which will then diffuse into the material. The level of suceptibility of C-22 to this phenomenon is determined using an electrochemical method. The level of suceptibility of C-22 to both hydrogen diffusion and hydrogen induced cracking is found.

4:25 PM
The Influence of Group IVA Elements on the Magnetostriction of Fe: *Robert P. Corson*[1]; Pinai Mungsantisuk[1]; Sivaraman Guruswamy[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

This paper examines the changes in magnetostriction of Fe when Group IVA elements (Si, Ge, and Sn) are added to Fe in the concentration range of 2 to 15%. Each of these elements has a filled or empty d-shell, and have similar ground state valence electron configuration (s2p2). However, the atomic size increases as one moves down the column. The alloys were prepared by directional solidification to obtain (100) texture. Magnetostrictive, x-ray and elastic properties of the alloys were measured. Correlations between changes in magnetostriction, magnetic properties, elastic properties, and lattice parameter are performed. The results are contrasted with earlier work on Ga and Al additions that result in large increases in the magnetostriction of iron. This research effort seeks to understand how the alloying additions modify the magnetostriction in Fe and develop the ability to engineer alloys for various actuation and sensing applications.

4:50 PM
Influence of Ordering on the Magnetostriction of Fe-(27.5- y) at.% Ga- y at.% Al Alloys: *Rebecca Charlotte Chandler*[1]; Sivaraman Guruswamy[1]; [1]University of Utah, Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

Fe-27.5 at. % Ga alloys can be heat treated to obtain ordered phases based on a DO3(ordered bcc), D019 (ordered hexagonal), and L12 (ordered fcc) structures. This work examines the heat treatment conditions required to obtain these different phases and how the substitution of Ga with Al influences these ordering reactions. An evaluation of how the different ordering treatments influence the magnetostriction in polycrystalline Fe-27.5 at.%Ga, Fe-13.75 at.% Ga-13.75 at.% Al, and Fe-20.625 at.% Ga -6.875 at.% Al alloys is made. The samples were annealed first in the disordered bcc (A2) phase region to obtain a disordered bcc solid solution, followed by ordering-heat treatment in the appropriate temperature region of stability of each of the ordered phases. X-ray diffraction was used to characterize the phases after heat treatments. Magnetostriction measurements were made at different prestress levels. Magnetic properties were measured using a VSM. Work supported by NSF-DMR Grant # 0241603.

Advances in Superplasticity and Superplastic Forming: Modeling of Superplastic Forming Processes and Materials

Sponsored by: Materials Processing and Manufacturing Division, Structural Materials Division, MPMD-Shaping and Forming Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Structural Materials Committee
Program Organizers: Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; P. A. Friedman, Ford Motor Company, Dearborn, MI 48124 USA; Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; P. E. Krajewski, General Motors R&D Center; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; J. G. Schroth, General Motors, R&D Center, Materials and Processes Laboratory, Warren, MI 48090-9055 USA

Wednesday PM, March 17, 2004 — Room: 201B — Location: Charlotte Convention Center

Session Chairs: James G. Schroth, General Motors, R&D Ctr., Matls. & Processes Lab., Warren, MI 48090-9055 USA; Peter A. Friedman, Ford Motor Company, Dearborn, MI 48124 USA

2:00 PM
Influence of Friction and Die Geometry on Simulation of Superplastic Forming of Al-Mg Alloys: *N. R. Harrison*[1]; S. G. Luckey[2]; P. A. Friedman[2]; Z. C. Xia[2]; [1]University of Michigan, Dept. of Mech. Engrg., 2350 Hayward St., Ann Arbor, MI 48109 USA; [2]Ford Motor Company, Scientific Rsch. Lab., 2101 Village Rd., MD 3135/SRL, Dearborn, MI 48124 USA

The ability to form complex shapes from aluminum sheet makes superplastic forming an attractive option in the automotive industry. Al-Mg alloys such as AA5083 show moderate superplastic characteristics while also having adequate post-formed properties. However, the additional cost associated with processing these materials to be suitable for superplastic forming has prevented their widespread use. Recent work has shown that it may be possible to superplastically form conventionally-processed Al-Mg alloys, such as AA5182, for relatively simple parts where high strains are not required. However, the lower strain-rate sensitivity associated with these conventionally-processed, lower-cost alloys can result in premature thinning and failure in forming operations. This paper compares the influence of friction and die geometry on the forming simulation of both alloys using the Abaqus standard finite element software. The 2-dimensional simulation of a long rectangular box where plane strain is assumed was performed using solid elements. The motivation for this work lies in the understanding of the thinning behavior of materials with different strain-rate sensitivities (m-value) through observation of depth of draw, forming time and pressure cycle.

2:20 PM
Development of MARC for Analysis of Quick Plastic Forming Processes: Krishna Murali[2]; *G. Paul Montgomery*[1]; James G. Schroth[3]; [1]General Motors, R&D Ctr., MC 480-106-359, 30500 Mound Rd., Warren, MI 49090-9055 USA; [2]General Motors, Warren, MI 48090-9055 USA; [3]General Motors, R&D, MC 490-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

MARC, an implicit finite element code developed by MARC Analysis Research Corporation and later acquired by MacNeal-Schwendler Corporation, has been used to simulate the Quick Plastic Forming (QPF) process. For accurate simulations, GM developed pressure control algorithms to maintain the desired strain rate, a job-termination criterion to stop a simulation at the correct stage, and constitutive equations to describe material behavior accurately. These tools have been used successfully in plane-strain, generalized-plane-strain, and 3D MARC analyses of QPF panels. The first application of 3D MARC analysis

was to the GM EV1 wheelhouse. Simulation was performed while the production tool was being machined. Changes recommended by the analysis were implemented in the tool and parts were successfully manufactured. Good correlation was obtained between predicted and measured thickness in the panels.

2:40 PM
Effect of the Constitutive Equation on MARC Analysis of Quick Plastic Forming: *G. Paul Montgomery*[1]; [1]General Motors, R&D Ctr., MC 480-106-359, 30500 Mound Rd., Warren, MI 48090-9055 USA

A constitutive equation that explicitly includes the dependence of stress on both strain and strain rate has been developed for aluminum alloy AA5083 by fitting tensile-test data. This paper uses the MARC finite element code to compare the results of Quick-Plastic-Forming (QPF) simulations that use this new equation with results of previous simulations which used constitutive equations that depend only on strain rate. Test cases include plane strain analysis of a section through the G90 side-frame outer (original design) and 3D analyses of a rectangular butter tray and the EV1 wheelhouse. This paper also investigates the effect of allowing a higher maximum pressure during the forming process.

3:00 PM Break

3:20 PM
Mechanical Behavior and Modeling of AA5083 at 450C: *Paul E. Krajewski*[1]; G. Paul Montgomery[1]; [1]General Motors, R&D Ctr., 30500 Mound Rd., MC 480-106-212, Warren, MI 48090 USA

The mechanical behavior of AA5083 at 450°C, which was determined from tensile tests, was characterized for strain rates ranging from 0.0005/s to 0.3/s. As strain rate increased, a transition occurred from classic power-law-type hardening to transient behavior where a pronounced yield point was followed by strain softening. The power-law-type hardening regime was accurately modeled using a physics-based model that contained quasi-static, thermally activated, and viscous drag components. The complete stress-strain curve as a function of strain rate was modeled using a three-part phenomenological equation that incorporated hardening, transient, and damage components.

3:40 PM
Forming Limit Diagram of a Superplastic Formable AA5083 Aluminum Alloy: *Mihai Vulcan*[1]; Klaus Siegert[1]; [1]University of Stuttgart, Inst. for Metal Forming Tech., Holzgartenstrasse 17, Stuttgart 70174 Germany

The theoretical and experimental work on superplastic materials at the Institute for Metal Forming Technology (IFU) of the University of Stuttgart, Germany is focused on aluminum alloys. Two analytical models have been used for bulging superplastic materials using circular and elliptical draw rings. The experiments have been carried out with an in-house built equipment in order to verify the theoretical models and to determine the forming limit diagram for a superplastic aluminum alloy AA5083 at a specific forming temperature and strain rate. The pressure-time paths determined from the analytical models were also verified by FEM process simulations. Finally, further FEM simulations have been done in order to determine the pressure-time path for the superplastic forming process of an automotive body part. The part, a license pocket plate of the rear deck lid, has been superplasticaly formed using the matrice forming technique.

4:00 PM
Integrated Approach for Optimization of Superplastic Forming: Pushkarraj V. Deshmukh[1]; Naveen V. Thuramalla[1]; Fadi K. Abu-Farha[1]; *Marwan K. Khraisheh*[1]; [1]University of Kentucky, Ctr. for Mfg., Mech. Engrg. Dept., 210 CRMS Bldg., Lexington, KY 40506-0108 USA

In this work, the Superplastic Forming Process (SPF) is optimized using microstructure-based constitutive models of deformation and failure. Superplastic deformation is modeled within the continuum theory of viscoplasticity with an anisotropic yield function and a microstructure-based overstress function. A multi-scale stability criterion taking into account both geometrical (macroscopic) and microstructural features, including grain growth and cavitation, is presented. These models are incorporated into a FE code and are used to generate optimum forming pressure profiles. It is shown that the FE results on forming time and thinning using the optimum pressure profile are better than those obtained using other forming conditions.

4:20 PM
Aspects of Element Formulation and Strain Rate Control in Numerical Modeling of Superplastic Forming: *S. G. Luckey*[1]; P. A. Friedman[1]; Z. C. Xia[1]; [1]Ford Motor Company, Scientific Rsch. Lab., 2101 Village Rd., MD 3135/SRL, Dearborn, MI 48124 USA

The ability to accurately and cost effectively simulate superplastic forming (SPF) processes is an essential step to achieving widespread use of this technology in the automotive industry. Practical and fundamental understanding of the capabilities and limitations of different finite element formulations in SPF is paramount to establishing simulation tools that are critical to die design and process development. In this paper, the results of a study comparing different element formulations in simulating superplastic forming with the ABAQUS finite element software are discussed. Simulations were performed with solid, shell and membrane elements to predict forming characteristics and pressure-time curves. Simulation of the 2-dimensional forming process with layered solid elements of a long rectangular cavity accurately predicted strain localization in the vicinity of the die entry radii. However, this thinning phenomenon was not captured in simulations with shell or membrane elements. Furthermore, FEA predictions of SPF pressure-time curves have been found to be greatly affected by the element type and the type of strain rate control methods instituted in the simulation. Two methods of strain rate control were applied in this study: an algorithm based on limiting the rate of deformation in the element with the largest strain rate and a second algorithm which limits the rate of deformation based on an average of the fastest straining elements. The resulting pressure-time curves for each of these formulations were compared with respect to each type of element. Application of the averaging scheme in controlling strain rate was found to reduce the differences between the predicted pressure-time curves for the different types of elements.

Aluminum Reduction Technology: Environmental

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Alton T. Tabereaux, Alcoa Inc., Process Technology, Muscle Shoals, AL 35661 USA

Wednesday PM Room: 213D
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Ken Martchek, Alcoa Inc., Wexford, PA 15090 USA

2:00 PM
Ventilation Rates of Smelter Buildings in Northern Climates: *Edgar Dernedde*[1]; [1]Kroll International, 1820 Rue Nancy, Brossard, Quebec J4Y 2M5 Canada

Modern smelter buildings are ventilated by natural ventilation, which makes good use of the large heat loss from aluminum reduction cells. Ventilation rates are usually high during the summer period. During winter, however, the ventilation rate is reduced in smelters located in northern climates, in order to maintain adequate working conditions inside the cell room. During the cooler seasons sidewall, basement and roof openings of a smelter building may be closed partially, which effectively reduces the ventilation rate. Airflows through cell rooms with 180 kA S/S prebake cells were estimated theoretically for ventilation conditions during all seasons of the year. The results were compared with measurements taken on scale models of a smelter building. The agreement was satisfactory.

2:25 PM
Improvement of Pot Gas Collecting Efficiency by Implementation of Impulse Duct System: *Michael Sahling*[1]; Elmar Sturm[1]; Geir Wedde[2]; [1]Hamburger Aluminium-Werk GmbH, Potrms., Dradenauer Hauptdeich 15, Hamburg 21111 Germany; [2]Alstom Power, Environment, Ole Deviks vei 10, Oslo 0666 Norway

At existing dry scrubbers the drop of differential pressure is given by design of bag filters and the pot ducting at a given gas volume. To meet new regulations for HF-emissions at increased production referring to pot gas capture efficiency the total gas volume of a plant has to be increased which result in higher duct losses. At the HAW smelter in Germany the new impulse duct principle has been installed in the ducting from the pots to the dry scrubber. The gas volume was increased by 20% by adding more filter and fan capacity. The additional pot duct losses could be fully compensated for by the impulse duct system and reveals as a good additional cost effective solution for retrofitting of dry scrubbers.

2:50 PM
Experiences With High Performance Dry and Wet Scrubbing Systems for Potlines: *Geir Wedde*[1]; Astrid Holmsen[1]; Terje Opsahl[1]; [1]Alstom Power Norway AS, Ole Deviks Vei 10, Oslo Norway

For recent smelter potline expansions in Norway, the ABART system for dry scrubbing of fluorides with downstream seawater scrubbers for SO2 removal, have been installed. This has been successfully implemented with low power consumption and extremely low emissions of both fluorides and SO2 as a result. This paper reviews recent experiences and performances with the ABART dry scrubbing technology as well as the seawater desulphurisation process.

3:15 PM
Alumina Structural Hydroxyl as a Continuous Source of HF: *Margaret M. Hyland*[1]; Barry J. Welch[2]; Edwin Patterson[1]; Toshifumi Ashida[3]; James B. Metson[2]; [1]University of Auckland, Chem. & Matls. Engrg., PB 92019, Auckland New Zealand; [2]University of Auckland, Light Metals Rsch. Ctr., PB 92019, Auckland New Zealand; [3]Kinki University, Dept. of Biotech. & Chmst., Sch. of Engrg. Japan

The link between moisture on alumina and HF generation in aluminium reduction cells has been long established. The assumption has usually been that the 'culprit' is the loosely bound adsorbed water, generating HF via bath hydrolysis as this surface water is flashed off during alumina feeding. Structural water, or more correctly, structural hydroxyl, also makes a significant contribution to HF generation. Laboratory experiments show that hydroxyl can dissolve in molten cryolitic electrolytes and gives rise to electrochemically generated HF. The electrochemically generated HF could be readily distinguished from HF generated via thermal hydrolysis. Experiments with aluminas of varying combinations of high and low surface adsorbed moisture and structural hydroxyl (as measured by their LOI (20-300) and LOI (300-1000), respectively) confirmed the importance of electrochemically generated HF from structural hydroxyl. While some of the structural hydroxide reacts rapidly at the time of feeding, it also contributes to the steady state HF emission. From plant studies it was estimated that up to 8 kg F/tonne Al was generated from structural hydroxyl, for aluminas containing 0.4 wt % LOI (300-1000) and assuming 3wt% alumina in the bath. Structural hydroxyl is found in transition alumina phases in smelter grade aluminas. Their presence ensures that even conservative smelter specifications of surface areas of 60-80 m2/g can be met. Paradoxically, this surface area is specified to ensure that the HF adsorption capacity of the alumina is sufficient for scrubber requirements, but for reasons of both surface and structural water incorporation, having a high surface area also means that the alumina will generate more HF. This reopens the debate on the merits of high surface area aluminas.

3:40 PM Break

3:50 PM
International Aluminium Institute Anode Effect Survey Results: Willy Bjerke[1]; Robert Chase[1]; Reginald Gibson[1]; *Jerry Marks*[1]; [1]International Aluminium Institute, New Zealand House, Haymarket, London SW1 Y 4TE UK

The International Aluminium Institute (IAI) conducts annual surveys of member companies on anode effect performance data. The anode effect data allows calculation of specific emissions of PFCs and also allows survey participants to benchmark anode effect performance with others operating with similar technology. IAI member companies now account for over seventy-five% of world primary aluminum production. The survey data collected include average anode effect frequency, average anode effect duration and anode effect overvoltage. The data show that in the twelve years covering the period 1990 to 2001 IAI members reduced CO2 equivalent emissions from 4.0 to 1.2 metric tons of carbon dioxide equivalents per metric ton of primary aluminium for the two combined PFCs, a reduction of 70%. This substantial reduction in specific emissions has resulted in one of the few examples of an industry where total emissions of a greenhouse gas have been reduced even as total production has increased.

4:15 PM
PFC Emission Measurements at DUBAL Potlines: *Ahmed Karim Gupta*[1]; Vijayakumar C. Pillai[1]; Jerry Marks[2]; [1]Dubai Aluminium Company Ltd., Environml. Control, Smelter Ops., Jebel Ali, PO Box 3627, Dubai United Arab Emirates; [2]J Marks & Associates, 312 NE Brockton Dr., Lee Summit, MO 64064 USA

Measurements of the perfluorocarbon compounds (PFC) tetrafluoromethane (CF4) and hexafluoroethane (C2F6) were carried out for Dubal's D18 (180kA bar broken CWPB) and CD20 (210kA PFPB) cell technologies using FTIR Spectrometry, according to the US EPA Draft Protocol for PFC Measurements from Primary Aluminium Production. CF4 was measured by collecting time average samples from the exhaust duct at the entrance to the dry scrubber. The ratio of C2F6 to CF4 was measured by extractive FTIR in a continuous and real time manner. The measurement results were compared with anode effect (AE) data to derive the Intergovernmental Panel on Climate Change (IPCC) Tier 3b coefficients. The coefficients are used in the IPCC Tier 3 method that is recommended for the most accurate inventory of PFCs from primary aluminium production. Dubal's ability to collect process data for both AE voltage and time on AE allowed the determination of both the Slope and Overvoltage equation coefficients. This paper presents the results of the PFC measurements and the calculated IPCC Tier 3b coefficients. The paper also compares the efficacy of the Slope method and the Overvoltage method for estimating PFC emissions. Finally results of measurements of PFC emissions from new cells following bath-up are presented. The results confirm the evolution of PFC during early high voltage cell operation and provide insight into the time and voltage dynamics of these emissions during new cell start up.

4:40 PM
In-Plant Performance Comparison of Fourier Transform and Photoacoustic Infra-Red PFC Monitors: *Neal R. Dando*[1]; Weizong Xu[1]; Luis Espinoza-Nava[1]; [1]Alcoa Technology, 100 Techl. Dr., Alcoa Ctr., PA 15069 USA

Several different infra-red (IR)-based technologies have been proposed for monitoring perfluorocarbon (PFC) emissions at aluminum smelters. Unfortunately comparative in-plant performance data is virtually non-existant for these technologies. This paper presents the results of several in-plant comparative performance evaluations of Fourier transform (FT) and photoacoustic (PA)-IR based PFC monitors and discusses the relative merits of each technology for executing capable in-plant PFC montoring campaigns.

Aluminum Reduction Technology: Materials and Fundamentals

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Tom Alcorn, Noranda Aluminum Inc., New Madrid, MO 63869 USA; Jay Bruggeman, Alcoa Inc., Alcoa Center, PA 15069 USA; Alton T. Tabereaux, Alcoa Inc., Process Technology, Muscle Shoals, AL 35661 USA

Wednesday PM Room: 213A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: John J.J. Chen, The University of Auckland, Chem. & Matls. Engrg., Auckland New Zealand

2:00 PM
20 Years of Bath Recycling Using Autogenous Mills: *Andre Pinoncely*[1]; [1]Solios Carbone, 32 Rue Fleury Neuvesel, Givors 69702 France

In 1984, Aluminium Becancour Inc. awarded FCB, the former company name of Solios, a contract for the supply of the first fully airswept autogenous grinding system dedicated to the cryolitic bath processing. Today 16 industrial references are in operation worldwide, and the 20th anniversary give us the opportunity to draw up the successive developments brought to the original design in order to comply with the ever-demanding requirements of bath processing: separate storage of bath qualities, full ingots feeding, and semi-hot bath processing. This paper will also focus on the latest very hot bath pre-cooling system and its contribution to reduce the smelter HF emissions.

2:25 PM
Impregnation of Cathode Blocks With Boron Oxide: *Rudolf Keller*[1]; Brian J. Barca[1]; David G. Gatty[1]; [1]EMEC Consultants, 4221 Roundtop Rd., Export, PA 15632 USA

It was shown that boron oxide in cathode blocks suppresses cyanide formation and promotes wetting of the surface by aluminum if the metal contains some titanium. EMEC Consultants developed a method to impregnate cathode blocks with boron oxide. In a custom-built autoclave, half-length blocks were exposed to a melt of boron oxide and some borax. Pressures of 160 psi were typically maintained at temperatures of about 800°C. Amorphous cathode blocks gained about 12 wt% in the average, graphitized blocks around 14 wt% with a minimum of 10 wt% being a targeted value. Some impregnated blocks were made available for exposure in commercial cells for 6 months, and a pot at Century Aluminum of West Virginia was fully equipped.

2:50 PM
Plant Experience With an Experimental Titanium Diboride Cell: *Marilou McClung*[1]; John Browning[1]; Scott Carte[2]; Craig Lightle[1]; Richard O. Love[1]; Ron Zerkle[3]; [1]Century Aluminum of West Virginia, Tech., PO Box 98, Ravenswood, WV 26164 USA; [2]Century Aluminum

of West Virginia, Potrm. Production, PO Box 98, Ravenswood, WV 26164 USA; [3]Century Aluminum of West Virginia, Pot Repair, PO Box 98, Ravenswood, WV 26164 USA

Century Aluminum of West Virginia, partnered with EMEC Consultants, SGL Carbon, Century Aluminum of Kentucky, and Golden Northwest Aluminum with support from the Department of Energy on a project to create a lower resistance at the carbon cathode-metal interface in the aluminum reduction cell. The approach was to provide a replenishing source of boron within the cathode to react with a titanium laced meal pad to generate a titanium diboride layer at the cathode. The target result is a surface layer of TiB2 that should create a reduction in energy consumption, an increase in production and potentially an increase in pot life. This project has had three phases. Phase two, completed in 2002, involved replacing three standard blocks with three blocks impregnated with boron oxide. In the third phase a full cell of impregnated blocks was installed. This paper documents the methods and results of the second phase of this test and nine months operational information on the phase three full pot and control pot.

3:15 PM
Experiences with Olivine-Based Refractories in Potlinings of Aluminium Electrolysis Cells: *Ole-Jacob Siljan*[1]; [1]Norsk Hydro ASA, Rsch. Ctr. HPI, N3907 Porsgrunn Norway

Olivine-based refractory materials have been tried for an extensive period of time as lining materials in aluminium reduction cells. Olivine provides a cost efficient and thermally stable lining for aluminium reduction cells. The lower chemical stability of pure olivine towards cryolitic melts can be overcome by adding smaller amounts of aluminosilicate minerals to the refractory product. The performed trials with this modified olivine-based brick material (ALUBRICK 2092) clearly indicate that operating results of cells lined with olivine-based bricks are at least comparable to the results of standard lined cells with fireclay materials. The data presented in the paper show that the chemical resistivity of ALUBRICK 2092 is at least as good as for standard fireclay bricks. Likewise, the data also indicate that the deterioration and the subsequent alteration of thermal properties of ALUBRICK 2092 results in potentially smaller changes to the cell heat balance than the corresponding deterioration of fireclay materials.

3:40 PM Break

3:50 PM
Wear of Silicon Nitride Bonded SiC Bricks in Aluminium Electrolysis Cells: *Flemming Bay Andersen*[1]; Guido Dorsam[1]; [1]Corus Research, Development & Technology, Ceram. Rsch. Ctr., PO Box 10000, IJmuiden 1970 CA The Netherlands

Samples from a sidewall of an aluminium electrolysis cell were submitted for study of the wear mechanism of the SiC-brick. Samples were studied using a combination of chemical analysis, microscopy, SEM-EDS and XRD. Unused bricks from 2 suppliers were used as reference material. The unused bricks are very similar to each other. Differences are found in the grain size of the SiC and in the binder (b-Si3N4 contents varies between bricks). All bricks have a coarse matrix and higher porosity in the core. The wear mechanism of the bricks seems a combination of attack by Na, HF, oxidation and moisture in the different zones. Below bath level first oxidation and then Na play a major role but bricks are here the least attacked. At bath level attack is severe but observations were complicated through a repair of what cause secondary reactions to occur. Above bath level the bricks disintegrate but reaction products are almost absent. Here we hypothesize that HF-vapor transformed the reaction products into volatile fluor ides and other gasses. This study shows that the corrosion tests currently used for Si3N4 bonded SiC-bricks, does not simulate the wear mechanism described in this report. In order to evaluate the performance of these bricks it is important to develop a new corrosion test. A proposal for such a new test is presented.

4:15 PM
Corrosion Tests and Electrical Resistance Measurement of SiC-Si3N4 Refractory Materials: *Bingliang Gao*[1]; Zhaowen Wang[1]; Zhuxian Qiu[1]; [1]Northeastern University, Sch. of Matls. & Metall., Mail Box 117, Shenyang, Liaoning 110004 China

Silicon nitride bonded silicon carbide materials have excellent properties, such as resistance to molten aluminum and cryolite and air oxidation in aluminum electrolysis cells, so they are used in some Chinese industrial cells as sidewall materials. In this paper, we tested SiC-Si3N4 materials made in China, in molten cryolite, liquid aluminum and high temperature air by various devices. In order to simulate the extremely corrosive conditions when no side-ledge is present in industrial aluminum cells, co-corrosion tests were carried out. The electrical resistance of SiC-Si3N4 materials attacked by aluminum electrolyte for 48 hours was measured from room temperature up to 950°. It is demonstrated that SiC refractory materials used for cell lining have better properties than carbon lining material.

Beyond Nickel-Base Superalloys: Other Systems and Physical Properties of Silicides

Sponsored by: Structural Materials Division, SMD-Corrosion and Environmental Effects Committee-(Jt. ASM-MSCTS), SMD-High Temperature Alloys Committee, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), SMD-Refractory Metals Committee
Program Organizers: Joachim H. Schneibel, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA; David A. Alven, Lockheed Martin - KAPL, Inc., Schenectady, NY 12301-1072 USA; David U. Furrer, Ladish Company, Cudahy, WI 53110 USA; Dallis A. Hardwick, Air Force Research Laboratory, AFTL/MLLM, Wright-Patterson AFB, OH 45433 USA; Martin Janousek, Plansee AG Technology Center, Reutte, Tyrol A-6600 France; Yoshinao Mishima, Tokyo Institute of Technology, Precision and Intelligence Laboratory, Yokohama, Kanagawa 226 Japan; John A. Shields, HC Stark, Cleveland, OH 44117 USA; Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA

Wednesday PM Room: 211B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Ridwan Sakidja, University of Wisconsin, Dept. of Matls. Sci. & Engrg., Madison, WI 53706 USA; Joachim H. Schneibel, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6115 USA

2:00 PM
Role of Different Alloying Elements in Modifying the Microstructures of Nb-Si-Ti-Al-Cr-X Alloys for High Temperature Aeroengine Applications: *Raghvendra Tewari*[1]; Hyojin Song[1]; Amit Chatterjee[2]; Vijay K. Vasudevan[1]; [1]University of Cincinnati, Dept. of Chem. & Matls. Engrg., Cincinnati, OH 45221-0012 USA; [2]Rolls-Royce Corporation, 2001 S. Tibbs Ave., Indianapolis, IN 46241 USA

Niobium silicides have drawn a considerable interest for possible applications at high temperatures. The binary niobium silicides, however, exhibit low room temperature ductility and poor oxidation at elevated temperatures. A multi element approach is being adopted to improve these properties. The present paper reports role of different alloying elements in the development of the microstructures under -as cast, -heat treated and -hot deformed conditions in the Nb-Si-Ti-Cr-Al-X alloys. The morphological distribution, structure and chemical composition of various phases under different treatments have been studied in detail. Our investigations have revealed a strong solute partitioning tendency of various elements, dissolution and re-precipitation of the phases during various heat treatments. The role of different alloying elements has been examined in influencing the properties of the alloys. These results reveal that additional strengthening of the matrix phase can be achieved through the precipitation of the different phases. In addition, materials have been thermomechanically processed and microstructural changes have been investigated.

2:15 PM
Structure and Thermal Expansion of Mo5Si3 and (Mo1.xVx)5Si3 Compounds: *Claudia J. Rawn*[1]; Joachim H. Schneibel[1]; Thomas R. Watkins[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., PO Box 2008, MS 6064, Oak Ridge, TN 37831-6064 USA

Tetragonal Mo5Si3 exhibits a coefficient of thermal expansion (CTE) in the c-direction that is more than twice as large as that in the a-direction. This high thermal expansion anisotropy causes microcracking and is therefore detrimental for components made from Mo5Si3. To decrease this pronounced thermal expansion anisotropy other elements such as Nb, Cr, V, and Ti have been introduced. High temperature x-ray powder diffraction has been used to study the changes in the CTE(c)/CTE(a) ratio. With the addition of V into the structure the anisotropy ratio reduces rapidly for small concentrations and levels out at approximately 1.25 for higher concentrations. With high quality diffraction data Rietveld refinements can give information on the site occupancies of the various elements in the structure. This type of information is used to explain how the thermal expansion anisotropy correlates with the atomic structure. Research sponsored by the Division of Materials Sciences, Office of Basic Energy Sciences; the Division of Materials Sciences and Engineering and by the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of

FreedomCAR and Vehicle Technologies, as part of the High Temperature Materials Laboratory User Program. ORNL is operated by UT-Battelle, LLC, for the U.S. DOE under contract DE-AC05-00OR22725.

2:30 PM

Processing, Microstructures and Mechanical Properties of Directionally Solidified V-V_3Si In Situ Composites: *H. Bei*[1]; E. P. George[2]; G. M. Pharr[1]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., 434 Doughtery Engrg., Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA

The intermetallic V_3Si has low density, high melting point and high strength at elevated temperatures. However, it suffers from intrinsic brittleness and low fracture toughness at ambient temperature. In this study, V-V_3Si eutectic alloys were directionally solidified to produce ductile phase toughened composites in a high temperature optical floating zone furnace. Depending on the solidification conditions several microstructures were observed, such as well-aligned lamellar, fibrous and cellular. The interphase spacings increase with decreasing solidification rates in agreement with the Jackson-Hunt theory. The mechanical properties of the individual phases were investigated by nanoindentation. It is found that the elastic modulus and nanoindentation hardness of V_3Si are 214 and 13.8 GPa, respectively, and those of the V solid solution 185 and 3.4 GPa, respectively. The high temperature strength was also examined by tensile testing at elevated temperature. Prelimary results show that the ductile to brittle transition temperature is about 800ºC for this composite, and its strength is significantly higher than those of conventional V solid solution alloys.

2:45 PM

Effects of Thermomechanical Processing on Texture Formation in Rolled Molybdenum Sheets: *Werner Skrotzki*[1]; Ingwar Hünsche[1]; Carl-Georg Oertel[1]; Wolfram Knabl[2]; [1]Technische Universität Dresden, Inst. für Strukturphysik, Dresden 01062 Germany; [2]Plansee AG, Technologiezentrum, Reutte 6600 Austria

The deformation of polycrystalline materials strongly depends on microstructure and texture. While grain size and Taylor factor determine the strength of the material grain shape and texture may lead to anisotropic flow. To improve the formability of molybdenum sheets the mechanisms of microstructure and texture formation during hot rolling and annealing have been investigated. The texture measurements were done by X-ray and electron backscatter diffraction. The microstructure was observed by orientation imaging microscopy and orientation contrast. The structural parameters of deformed and recrystallized molybdenum sheets were characterized in detail with respect to reduction in thickness, through-thickness texture gradient, annealing temperature and annealing time, i.e. recrystallization kinetics. The results may be used to optimize the deep-drawing properties of molybdenum sheets.

3:00 PM

Microstructure and Oxidation Studies in the Cr-Si-Al System: Zhicong Yuan[1]; *Panayiotis Tsakiropoulos*[1]; Guosheng Shao[1]; John Watts[1]; [1]University of Surrey, Sch. of Engrg., Mech. & Aeros. Engrg., Metall. Rsch. Grp., Guildford, Surrey GU2 7XH England

The Cr-Si-Al system has been chosen to study phase equilibria and microstructure development and oxidation behaviour in intermetallic based alloys, owing to the importance of this system in the development of new materials to replace the Ni base superalloys that are used currently in gas turbine engines. The alloys were prepared using clean melting and casting in water-cooled copper crucibles/moulds and were studied in the as cast and heat-treated conditions. In our study particular attention has been paid to the solidification microstructures and on phase equilibria involving the Cr-Si silicides and Cr-Al intermetallics and on the role of Al additions in controlling phase selection and oxidation behaviour. Selected alloys have also been evaluated for their oxidation behaviour using isothermal oxidation tests that cover the whole range from pest, to intermediate to high temperature oxidation. The results of microstructural characterization will be presented and discussed.

3:15 PM

Defect Structure and Low-Temperature Mechanical Properties of Cr_2Zr -Cr_2 Nb Pseudo-Binary Laves Phase Alloys: *Takayuki Takasugi*[1]; Yoshiyasu Nakagawa[1]; Yasuyuki Kaneno[1]; Hirofumi Inoue[1]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

The effect of composition on the Cr_2Zr -Cr_2Nb pseudo-binary alloys which are composed of single Laves phase and the duplex microstructure with Cr solid solution was investigated, focusing upon the defect structure and low-temperature mechanical properties. The defect structures were analyzed using XRD and density measurement while the low-temperature mechanical properties were evaluated based on Vickers hardness and the fracture toughness. The C15 phase field is continuous between Cr_2Zr -Cr_2Nb, with a maximum phase field width of about 15at.% solubility. The defect mechanism is governed by anti-site constitutional defects for alloys along the pseudo-binary line, but by the mixture of vacancy and anti-site defects for alloys along the constant Zr/Nb plethal section. The relationships between the defect structure and low-temperature mechanical response are discussed, in correlation with the retained high-temperature Laves phase and atomic size of constituent elements.

3:30 PM

Development of Chromium-Tungsten Alloys: *Omer N. Dogan*[1]; Jeffrey A. Hawk[1]; [1]US Department of Energy, Albany Rsch. Ctr., 1450 Queen Ave., SW, Albany, OR 97321 USA

Cr alloys containing 0-30 weight % W were investigated for their high temperature strength and oxidation resistance. These experimental alloys are intended for use in elevated temperature applications. Alloys were melted in a water-cooled, copper-hearth arc furnace. Microstructure of the alloys was studied using X-ray diffraction, scanning electron microscopy, and light microscopy. Meyer and Vickers hardness tests were utilized for measuring room temperature strength. A hot hardness tester with a spherical ruby indenter was used to study the strength of these materials between 800ºC and 1200ºC. A parabolic relationship was observed between load and indent size at all temperatures. On the other hand, decrease in hardness of the alloys with temperature was linear up to 1200ºC.

3:45 PM

Electrical and Thermal Transport Properties of Single Crystalline Mo_5X_3 (X=Si, B, C): *Taisuke Hayashi*[1]; Kazuhiro Ito[1]; HIroyuki Nakamura[1]; Masaharu Yamaguchi[1]; [1]Kyoto University, Matls. Sci. & Engrg., Sakyo-ku, Kyoto 606-8501 Japan

Mo_5X_3 (X=Si, B, C) intermetallic compounds such as Mo_5SiB_2 ($D8_l$), Mo_5Si_3 ($D8_m$) and Mo_5Si_3C ($D8_8$) have a great potential for ultra-high temperature applications, such as an oxidation protective coating and a heating element. We measured the thermal conductivity and electrical resistivity of the Mo_5X_3 single crystals. The thermal and electrical conductivity at room temperature for Mo_5SiB_2 are about 30 W/mK and $2.5x10^6$ Ω^{-1} m^{-1}. They are higher than those of Mo_5Si_3 and Mo_5Si_3C single crystals, but are much smaller than those of $MoSi_2$ and Mo. The resistivity of Mo_5SiB_2, Mo_5Si_3 and Mo_5Si_3C single crystals exhibited a negative curvature ($d^2\rho(T)/dT^2<0$), with a tendency towards saturation. In the Mo_5Si_3C with large ρ_0 due to impurity carbon atoms, resistivity saturation is pronounced. In contrast, a much higher temperature is required to reach saturation in the Mo_5SiB_2.

4:00 PM

Microstructure Evolution of $E2_1$-$(Co, Ni)_{?R}AlC$ Based Heat Resistant Alloys: *Shinya Teramoto*[1]; [1]Tokyo Institute of Technology, Matls. Sci. & Engrg., 4259 Nagatsuta, Midori-ku, Yokohama, Kanagawa-ken 226-8502 Japan

We are designing new class of (Co, Ni)-base heat resistant alloys strengthened by $E2_1$-$(Co, Ni)_3AlC$. The $E2_1$ type ordered crystal structure is almost the same as that of the $L1_2$ type excluding that a carbon atom occupies the octahedral interstice at the body-center. It's reported $E2_1$-$(Co, Ni)_3AlC$ forms continuous solid solution between $E2_1$-Co_3AlC in the Co-Al-C system and $L1_2$-Ni_3Al in the Ni-Al system. The addition of Ir or/and Pt is thought to effectively improve the elevated temperature strength to go beyond Ni and Co-base superalloys. Objective of the present work is to conquest proper microstructures by controlling alloy composition, alloy process and heat treatment. They include not only the cuboidal $E2_1$ precipitates in the α-(Co, Ni) matrix, but also discontinuous precipitation of $E2_1/\alpha$ and directional solidification of the eutectic $E2_1$///00 1/, and so forth. We have also investigated phase stability of $E2_1$ phase from the viewpoint of magnetic properties.

4:15 PM Concluding Remarks Joachim H. Schneibel

Bulk Metallic Glasses: Phase Transformation and Alloy Design

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

Wednesday PM Room: 209A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Michael K. Miller, Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6136 USA; Todd C. Hufnagel, Johns Hopkins University, Matls. Sci. & Engrg., Baltimore, MD 21218-2681 USA

2:00 PM Invited
Apt Characterization of the Decomposition of Bulk Metallic Glasses: *Michael K. Miller*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831-6136 USA

The early stages of decomposition and phase separation of bulk metallic glasses may be quantified by atom probe tomography to provide information on their stability. Atom probe tomography has been applied to several bulk metallic glasses to reveal changes in the atomic arrangement of atoms in the as-cast state and after annealing in the vicinity of the glass transition temperature. The composition and morphology of coexisting phases may be characterized from the earliest stages of decomposition to the final crystalline microstructure. Research at the SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

2:25 PM Invited
In-Situ Scattering Studies of Phase Transformation Behaviors in Bulk Metallic Glass: *Xun-Li Wang*[1]; C. T. Liu[2]; J. K. Zhao[1]; Weihua Wang[3]; [1]Oak Ridge National Laboratory, Spallation Neutron Source, 701 Scarboro Rd., Oak Ridge, TN 37831 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831 USA; [3]Chinese Academy of Sciences, Inst. of Physics, Beijing 100080 China

Phase transformation is a promising way for making nanostructured materials in large quantities. Phase transformations involving nanostructured materials usually occur under conditions far from equilibrium. Although thermodynamics ultimately determines the equilibrium phase for a given set of conditions, the answers as to whether and how the nanostructured phase is produced from the meta-stable precursor depend on the kinetic processes. Here, we describe some recent in-situ synchrotron scattering experiments aiming to understand the kinetics of phase transformation in bulk metallic glass. For Zr52.5Cu17.9Ni14.6Al10Ti5, simultaneous measurements of diffraction and small angle scattering data provided first direct demonstration of a phase separation prior to the amorphous-to-crystalline phase transformation. Our data support the view that crystalline nucleation is achieved via phase separation through a mechanism of short-range diffusion of small atoms (e.g., Ni) whereas the growth is determined by long-range d iffusion. For Nd60Al10Fe20Co10, a magnetic bulk amorphous alloy, we have identified two distinctively different decomposition processes from scaling of the in-situ small angle scattering data. A correlation is established between the decomposition kinetics and the magnetic properties of the alloy. Research supported by Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy under Contract DE-AC05-00OR22725 with UT-Battelle.

2:50 PM Cancelled
Exploration of Bulk Metallic Glasses Based on Ti-Ni-Si

3:15 PM
Development Strategy for Bulk Aluminum Glass: *Wynn Steven Sanders*[1]; [1]Air Force Research Laboratory, AFRL/MLLMD, 2230 Tenth St., Bldg. 655, Rm. 025, Wright-Patterson AFB, OH 45324 USA

This paper presents a strategy for the development of bulk aluminum metallic glass. Recent research has led to new insights into the criteria for bulk metallic glass formation. First, bulk metallic glasses obey a unique topological relationship as shown in atomic size distribution plots. Additionally, there are preferred radius ratios that are beneficial to efficient atomic packing. Finally, appropriate alloying element selection can depress the liquidus relative to the glass transition temperature. These revised criteria are being used to guide efforts to develop new bulk metallic glasses based on aluminum. Composition space of known ternary marginal glass formers is being systematically explored via the addition of quaternary and higher order alloying elements based upon topological and chemical considerations. This approach has resulted in an increase in the glass forming ability of marginal aluminum glasses. The specific approach applied in this exploration study will be described and results will be presented.

3:40 PM
Effect of Oxygen Impurities on Al-, Cu-, and Ti-Based Metallic Glass: *Wynn Steven Sanders*[1]; [1]Air Force Research Laboratory, AFRL/MLLMD, 2230 Tenth St., Wright-Patterson AFB, OH 45324 USA

Oxygen contamination has been shown to dramatically reduce the glass forming ability of alloys. Although the effect of oxygen impurities have been extensively studied in the Zr-based bulk metallic glass forming systems, little work has been done on marginal glasses. The magnitude of this effect becomes important as new glass-forming systems are investigated and produced. This effect is even more critical for glasses that require high cooling rates; oxygen impurities can prevent glass formation entirely. The levels of oxygen impurity levels have been varied in aluminum-, copper-, and titanium-based marginal glass formers and its effect has been characterized by differential scanning calorimetry and x-ray diffraction. Initial results indicate that these alloys exhibit varying amounts of sensitivity to oxygen. Results from this analysis will be presented.

4:05 PM
Compaction of Amorphous Aluminum Alloy Powder by Direct Extrusion and Equal Channel Angular Extrusion: *O. N. Senkov*[1]; S. V. Senkova[1]; D. B. Miracle[2]; [1]UES, Inc., Matls. & Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

$Al_{89}Gd_7Ni_3Fe_1$ alloy powder produced by gas atomization was consolidated using equal channel angular extrusion (ECAE) and direct extrusion. The powder particle size was below 40 μm (-325 mesh grade) and the powder was ~80% amorphous. Compaction was conducted in the temperature range of 200°C to 500°C. Densities above 98% of the theoretical density were achieved after compaction at temperatures below 250°C and 100% density was achieved above 420°C. In the temperature range of 250°C to 420°C very poor consolidation of the powder occurred due to formation of brittle intermetallic phases. Non-homogeneous deformation of the powder was detected during consolidation at low temperatures and the consolidated material had an amorphous structure with precipitations of crystalline phases. After compaction at 450°C and 500°C, the material was fully dense and had a nanocrystalline structure. Features of interactions of the powder particles with amorphous and crystalline phases during consolidation are outlined.

4:30 PM
Topological Criteria for Amorphization Based on Thermodynamic Approach: *Oleg N. Senkov*[1]; Daniel B. Miracle[2]; [1]UES, Inc., Matls. & Processes Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA

A thermodynamic model for amorphization is proposed that allows analysis of the effect of composition, atomic radii, and elastic constants of constitutive elements on amorphization. The model is based on comparison of the Gibbs free energy and entropy of a non-equilibrium crystalline solid solution and the undercooled liquid. According to this model, the amorphous state is thermodynamically stable (relative to the crystalline solid solution) only for certain alloy compositions. The larger is the atom size of the solvent element, the better is the condition for amorphization. To achieve global instability of the crystal lattice, the atomic radius ratio R of solute and solvent elements should fall in a particular range of R. This range becomes wider when the size of the solvent element increases. The glass transition temperature increases with the atom size of the solvent element and it has a maximum at a certain concentration of the solute.

4:55 PM
Study of Glass Forming Ability and its Correlation with Eutectic Alloys: *Yi Li*[1]; [1]National University of Singapore, Dept. of Matls. Sci., Lower Kent Ridge Rd., Singapore 119260 Singapore

Many bulk metallic glasses have been found in many alloy systems over the last ten years. Despite the fact that many parameters have been used to find the best glass forming alloys and some of them are very successful, the way to find the alloy composition with the opti-

mum glass forming ability within one alloy system is still not clear. We have analyzed the glass forming ability around eutectic composition in terms of the competitive growth/formation of primary dendrites, eutectic and glass. It is concluded that the glass forming ability of a eutectic alloy system depends on the type of the eutectics, i.e. symmetric or asymmetric eutectic coupled zone. For the alloy systems with symmetric eutectic coupled zone, the best glass forming alloys should be at or very close to the eutectic composition. For the alloys with asymmetric eutectic coupled zone, the best glass forming alloys should be at off-eutectic compositions, probably towards the side of the faceted phase with a high entropy in the phase diagram. We will show our latest results on the glass formation in Zr, Pd, La and Ni based alloy discovered using the above analysis method.

Cast Shop Technology: Grain Refining

Sponsored by: Light Metals Division, LMD-Aluminum Committee
Program Organizers: Corleen Chesonis, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Jean-Pierre Martin, Aluminum Technologies Centre, c/o Industrial Materials Institute, Boucherville, QC J4B 6Y4 Canada; Alton T. Tabereaux, Alcoa Inc., Process Technology, Muscle Shoals, AL 35661 USA

Wednesday PM — Room: 213B/C
March 17, 2004 — Location: Charlotte Convention Center

Session Chairs: David H. StJohn, CAST Cooperative Research Center, University of Queensland, Brisbane, Queensland 4068 Australia; A. Lindsay Greer, University of Cambridge, Dept. of Matls. Sci. & Metall., Cambridge CB2 3QZ UK

NOTE: Session begins at 2:25 PM

2:25 PM
Investigation of Grain Refinement Fading in Hypoeutectic Aluminium-Silicon Alloys: *Tomasz Stuczynski*[1]; Zbigniew Zamkotowicz[1]; Boguslaw Augustyn[1]; Marzena Lech-Grega[1]; Wladyslaw Wezyk[1]; [1]The Institute on Non-Ferrous Metals, Light Metals Div., Pilsudskiego 19, Skawina 32-050 Poland

The results of the investigations concerning the grain refinement process in hypoeutectic silumins (using master alloys AlTi5B1, AlTi1.7B1.4 and AlTiC as grain refiners) will be presented. Using master alloys AlTi1.7B1.4 and AlTi5B1 as grain refiners, the fading of grain refinement effect was observed. Carried out examinations indicated the effect of the grain refinement fading is caused by sedimentation of boron compounds clusters in molten silumins and this process depends, among others, on the quantity of the free titanium in tested alloys.

2:50 PM
Synthesis of Al-Ti-B Master Alloys by Different Techniques Using Ti and B Bearing Salts: M. A. Shaheen[2]; Ibrahim Hamed Aly[3]; Aly Bastaweesy[3]; *Abdel-Nasser M. Omran*[1]; [1]Aluminium Company of Egypt, R&D, Nag-Hammady Egypt; [2]Suez Canal University, Metallurgl. Dept., Fac. of Engrg., Suez Egypt; [3]Minia University, Chem. Engrg. Dept., Fac. of Engrg., El-Minia Egypt

An Al-Ti-B alloys containing up to 10% titanium and 5% boron has been obtained by reacting of B and Ti bearing salts with aluminium. The parameters affecting the reaction process were studied. The experiments were carried out in two techniques: first by using mixture of KBF_4 and K_2TiF_6 and second by using mixture of KBF_4, K_2TiF_6 and granular aluminium. The results obtained were indicated that, the first technique is the best one. The efficiency of the Ti, B recovery up to 99% and 94% respectively. Microstructure examination, X-ray diffraction tests were carried out on the produced alloys.

3:15 PM
Grain Refiner Fade in Aluminium Alloys: *Paul L. Schaffer*[1]; Jacob W. Zindel[2]; Arne K. Dahle[1]; [1]University of Queensland, Div. of Matls., Frank White Bldg. (Bldg. 43), Cooper Rd., St. Lucia, Queensland 4072 Australia; [2]Ford Motor Company, MD 3182 SRL, PO Box 2053, Dearborn, MI 48121 USA

Grain refinement is usually an integral part of melt treatment undertaken in order to consistently produce castings with premium mechanical properties. However, the successful grain refinement of foundry alloys compared to wrought alloys has proven difficult due to a higher alloy content and long holding times encountered in the foundry. Often a melt is held for long periods and the grain refinement efficiency decreases, or fades, with time. Depending on the severity of fade, significant variations in the grain size can occur, which in turn can affect the mechanical properties, porosity distribution and consistency of the casting. Settling of the grain refining particles is believed to make a significant contribution to the mechanism of fade. However, the rate of fade is much faster than predicted by Stokes law and this suggests that other factors such as particle agglomeration, formation of oxide layers and melt turbulence also contribute to the loss of grain refinement during casting. Dedicated laboratory t rials have been conducted to determine the contributions of the separate mechanisms to the overall fade observed.

3:40 PM Break

4:15 PM
Reducing the Cost of Grain Refiner Additions to DC Casting: *Mark A. Easton*[1]; David H. StJohn[2]; Elizabeth Sweet[3]; [1]Monash University, CRC for Cast Metals Mfg. (CAST), Sch. of Physics & Matls. Engrg., PO Box 69, Melbourne, Victoria 3800 Australia; [2]University of Queensland, CRC for Cast Metals Mfg. (CAST), Div. of Matls. Engrg., Brisbane, Queensland 4068 Australia; [3]Comalco Research and Technical Support, Thomastown, Victoria Australia

This paper describes implementation of technology developed at the CAST CRC for reducing the cost of grain refiners added in VDC casting of billet. Experimental work showed that cost savings could be made in some alloys by reducing the amount of boron-containing rod grain refiner addition, and compensating for this by the addition of titanium to the alloy to increase the value of the growth restriction factor. A grain refiner calculator has been developed so that the lowest cost addition can be determined for a particular alloy while still achieving the desired fine grain microstructure. Recently the results from this calculator have been applied at Comalco casthouses without an increase in hot cracking rates. The methodology of implementation and the potential benefits of this technology are described.

4:40 PM
Opticast: A Method for Optimized Aluminum Grain Refinement: Lennart Backerud[1]; Holm Boettcher[2]; John Courtenay[3]; *Rein Vainik*[1]; [1]Opticast AB, Junkergatan 13, SE-12653, Hagersten, Stockholm Sweden; [2]AMAG, PO Box 32, A-5282 Ranshofen Austria; [3]MQP Limited, 6 Hallcroft Way, Knowle, Solihull B93 9EW UK

By measuring the grain size in samples taken from the casting furnace, the necessary amount of grain refiner can be calculated for each single cast. The Opticast method involves rapid sample preparation and grain size analysis, which allows the method to be used on-line in all cast houses. The grain sizes measured are compared against calibration curves and the optimum amount of addition is calculated. The method can also be used for production of ingots in any specified grain size range, within narrow limits. In practical application in two cast houses, cost savings of up to 90% have been accomplished for certain alloys, while the mean savings for the whole production is at least 50%. Due to decreased grain refiner additions considerable quality improvement has also been achieved.

5:05 PM
Production of Al-B Master Alloys from Boron-Bearing Salts Using Different Techniques: Ibrahim Hamed Aly[3]; A. Bastaweesy[3]; M. A. Shaheen[2]; *Abdel-Nasser M. Omran*[1]; [1]Aluminium Company of Egypt, R&D, Nag-Hammady Egypt; [2]Suez Canal University, Metallurgl. Dept, Fac. of Engrg., Suez Egypt; [3]Minia University, Chem. Engrg. Dept., Fac. of Engrg., El-Minia Egypt

An Al-B alloys containing about 5% boron has been obtained by reacting of potassium fluoroborate (KBF_4) with aluminium. The parameters affecting the reaction process such as: temperature, time, mixing speed, potassium fluoroborate to aluminium weight ratio and particle size were studied. The experiments were carried out in three techniques: first by using KBF_4 only, second adding mixture of KBF_4 and granular aluminium 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.

CFD Modeling and Simulation of Engineering Processes: Process Modeling II

Sponsored by: Materials Processing & Manufacturing Division, ASM/MSCTS-Materials & Processing, MPMD/EPD-Process Modeling Analysis & Control Committee, MPMD-Solidification Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)

Program Organizers: Laurentiu Nastac, Concurrent Technologies Corporation, Pittsburgh, PA 15219-1819 USA; Shekhar Bhansali, University of South Florida, Electrical Engineering, Tampa, FL 33620 USA; Adrian Vasile Catalina, BAE Systems, SD46 NASA-MSFC, Huntsville, AL 35812 USA

Wednesday PM Room: 206A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Aniruddha Mukhopadhyay, Fluent Inc., Lebanon, NH 03766 USA; Michel Bellet, Ecole des Mines de Paris, Paris France; Alain Jardy, Ecole des Mines de Nancy, Nancy France

2:00 PM Opening Remarks - Aniruddha Mukhopadhyay

2:05 PM Invited
Simulation of Bubble Formation Based on the Lattice Boltzmann Method: *Johannes Steiner*[1]; Christian Redl[1]; Wilhelm Brandstaetter[1]; Alois Triessnig[2]; [1]Mining University Leoben, Christian-Doppler-Lab. for Applied Computational Thermofluiddynamics, Franz-Josef-Strasse 18, Leoben 8700 Austria; [2]RHI AG, Rsch., Magnesitstrasse 2, Leoben 8700 Austria

The results of numerical simulations of bubble detachment from an orifice in a horizontal plate submerged in liquid are presented. The simulations are performed with a two-dimensional Lattice Boltzmann (LB) scheme. In previous works the same problem has been investigated by two-phase simulations in which surface tension effects were considered by an interaction potential formulation between the phases. In contrast, in the present work a volume of fluid (VOF) model consisting of a carrier fluid and a passively advected scalar function for the calculation of gravitational and surface tension forces, respectively, is used. The aim of this study is to verify the theoretical and experimental predictions of the dependencies of the departure diameter on gravitational forces, surface forces and wettability effects. Nonlinear fits to the simulation data yield functional dependencies which are in good agreement with the predictions.

2:40 PM
Computer Simulation on Flow Phenomena in Pot of Continuous Galvanizing Line: *Bo-Young Hur*[1]; *Xiangying Zhu*[1]; Arai Hiroshi[1]; [1]Gyeongsang National University, Dept. of Metallurgl. & Matls. Engrg., Kajoadong 900#, Chinju 660-701 S. Korea

Dross flow behavior and aluminum mixing process are important phenomena in continuous hot dip galvanizing process, which influences the quality of coating layer and then influences the anti-corrosive performance. In this paper, these two phenomena have been investigated using computer simulation technique that is based on finite element method. In order to avoid the adhesion of dross, baffle is generally used to prevent the bottom dross re-flotation. A comparison study of dross flow behavior was performed between baffled case and unbaffled case using computer simulation. Aluminum mixing route was mainly followed the top surface, and accumulation of Al concentration occurred near the corner between strip and free top surface. This route would be reasonable if the affection of the streak line of zinc flow and the lower density of the zinc solution due to containing high level Al were considered.

3:05 PM
CFD Modeling of Sintering Phenomena During Iron Ore Sintering: *Pengfu Tan*[1]; Dieter Neuschutz[2]; [1]Portovesme Nonferrous Metallurgical Company, S. P. n. 2 - Carbonia/Portoscuso - km. 16,5, Portoscuso, CA I-09010 Italy; [2]Lehrstuhl fur Werkstoffchemie, Rheinisch-Westfalische Technische Hochschule Aachen, D-52056 Aachen Germany

A CFD model of nickel flash furnace has been developed. Some results are presented in this work.

3:30 PM
Modeling Dissolution in Aluminum Alloys: *Tracie Zoeller*[1]; T. H. Sanders[2]; G. Paul Neitzel[1]; [1]Georgia Institute of Technology, Sch. of Mech. Engrg., Atlanta, GA 30332-0405 USA; [2]Georgia Institute of Technology, Matls. Sci. & Engrg., Atlanta, GA 30332-0245 USA

The dissolution of particles in a matrix is an important process that occurs during heat treatment of many materials. The effect of the heat treatment, or homogenization step, on spherical precipitates in an aluminum matrix was considered in this study. Upon solidification, an aluminum alloy microstructure is highly segregated. The microstructure consists of cored dendrites with various soluble and insoluble phases present in the dendritic regions. Subsequent heat treatments are performed to homogenize the microstructure. The microstructure evolution after each processing step is dependent upon the previous microstructures. The variation in local chemical composition may promote or hinder precipitation of new phases. A large volume fraction of coarse insoluble phases can lead to the occurrence of recrystallized grains via particle stimulated nucleation, while inhomogeneous solute distribution can lead to the precipitation of an uneven distribution of dispersoid phases. Understanding the dissolution of these secondary phases is important in predicting microstructural evolution in the alloy. A simple model will be presented to describe this moving boundary problem. Local equilibrium conditions are assumed at the precipitate-matrix interface. Composition profiles and dissolution times will be compared to experimental data and other models found in the literature.

3:55 PM Break

4:15 PM
Computational Model to Predict the Degradation of Particulate Material During Pneumatic Conveying: *Mayur K. Patel*[1]; P. Chapelle[1]; N. Christakis[1]; M. Cross[1]; [1]University of Greenwich, Sch. of Computing & Math. Scis., Old Royal Naval College, 30 Park Row, Greenwich SE10 9LS London

Pneumatic conveying of granular materials is relied upon in many industrial situations, because of its simplicity/flexibility. However, attrition/degradation of particulate material conveyed is commonly observed, in dilute phase flow, which affects "product-quality" and causes difficulties in subsequent materials handling operations. It is well-known that degradation during dilute phase pneumatic conveying results mainly from high-velocity impacts of the particles with pipe-walls at bends. Available computational models concentrate on the detailed description of the flow of the solids/gas phases and ignore the damage imparted to the particle. The objective of the paper is to report the development of a Computational framework to describe/model flow of the gas-solids mixture and to predict particle degradation during dilute phase pneumatic conveying. The key feature of the model is the prediction of particle breakage, due to particle-wall impacts. Calculations of propensity of degradation are based on the use of parametrised impact data obtained via laboratory scale degradation tests. Results of the approach are presented and compared against experimental data.

4:40 PM
Numerical Study of Fluid Flow in an Agitated Quench Tank: *Mohammed Maniruzzaman*[1]; Mike Stratton[1]; Richard Sisson[1]; [1]Worcester Polytechnic Institute, Ctr. for Heat Treating Excellence, Mech. Engrg. Dept., 100 Inst. Rd., Worcester, MA 01609 USA

Distortion and cracking of a metallic part quenched in a liquid quenchant can be reduced greatly by improving quench uniformity. Agitation of the quenchant is one of the important factors for quench uniformity. Agitation greatly enhances heat extraction rate from the quenched part surface. The flow in a quench tank can be optimized using computational fluid dynamics (CFD) as a tool for quench tank/agitation system design. In this study, numerical experiments are performed to predict the optimum impeller and tank design for uniform agitation in a quenched tank. Experiments are performed to validate the predictions.

Computational Thermodynamics and Phase Transformations: Thermodynamics and Phase Transformations

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizer: Jeffrey J. Hoyt, Sandia National Laboratories, Materials & Process Modeling, Albuquerque, NM 87122 USA

Wednesday PM Room: 202A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

2:00 PM
Alloy Thermodynamics by the Screened Generalized Perturbation Method: *A. V. Ruban*[1]; S. I. Simak[2]; S. Shalcross[2]; H. L. Skriver[1]; [1]Technical University of Denmark., Physics Dept., Bldg. 307, Lyngby 2800 Denmark; [2]Uppsala University, Physics Dept., Uppsala SE-75121 Sweden

The screened generalized perturbation method (SGPM) is an ab initio technique for calculating Ising-type interactions in alloys. Although it is based on a number of approximations (the coherent potential approximation is one of the most serious), it appears to yield a quantitatively accurate description of the configurational alloy energetics in many different type of alloys. Besides, in contrast to the widely used structure inversion method it is extremely efficient and provides a transparent physical picture of the "chemical" ordering. We demonstrate this point by applying the SGPM to calculations of the ordering energies, short-range parameters, order-disorder transition temperatures, and alloy configurations on the surfaces in CuZn, CuAu and PtCo systems.

2:20 PM
Prediction and Measurement of the Phonon Entropy of Alloying in Dilute Substitutional Alloys: *Olivier Delaire*[1]; Tabitha Swan-Wood[1]; Brent Fultz[1]; [1]CALTECH, Matls. Sci., Keck MC 138-78, Pasadena, CA 91125 USA

In this study, we investigate the entropic effects associated with changes in the vibrational modes of crystals sustained upon dilute substitutional alloying. Using inelastic neutron scattering techniques, we have measured the change in the phonon entropy of vanadium associated with the alloying of a few percent impurities of Ni, Pd, Pt or Co. The phonon entropy change upon alloying in vanadium was shown to be large and negative. In the case of 6-7% Pt and Co impurities, it is equal in magnitude to the configurational entropy gain of the alloy. We also present experimental results on Mo-7%Co alloys. Using a computational model based on the classical Born-von Karman approach, we extracted interatomic force-constants from our experimental data, from which phonon dispersion curves and density of states (DOS) could be calculated. In an effort to trace the microscopic origin of the effect observed, we also performed ab-initio calculations of the lattice-dynamics of these alloys. We calculated phonon dispersions and DOS curves with pseudo-potential based plane-wave density functional theory techniques, using a supercell approach to model the random substitutional alloys. We present a comparison of our ab-initio dynamics predictions with the results of our experimental measurement.

2:40 PM
Evolution of Precipitates in Systems With Heterogeneous Nucleation Sites: *Ernst Kozeschnik*[1]; Franz Dieter Fischer[2]; Jiri Svoboda[3]; [1]Graz University of Technology, Inst. for Matls. Sci., Welding & Forming, Kopernikusgasse 24, Graz 8010 Austria; [2]University of Mining, Inst. of Mech., Franz-Josef Straße 18, Leoben 8700 Austria; [3]Academy of Science of the Czech Republic, Inst. of Physics of Matls., Zizkova 22, Brno 61662 Czech Republic

In many metallic systems, precipitates can nucleate on various heterogeneous nucleation sites, such as dislocations, grain-boundaries or sub-grain boundaries. For instance, in typical martensitic-ferritic CrMo-steels, M23C6 precipitates are formed on austenite grain boundaries during cooling from the liquid state. Later, after martensite transformation and during heat treatment, precipitates of the same type also nucleate on the sub-boundaries of the martensite lath-structure. The precipitates on the prior austenite grain boundary (PAGB) are usually larger than the precipitates formed later inside the transformed austenite grain. If the evolution of these two populations of precipitate is modelled under the assumption of a random distribution, the larger particles on the PAGD will coarsen on the expense of the particles on the sub-grain boundaries, and the latter population will dissolve completely in relatively short time. In reality, the two populations evolve in a more or less independent way with a weak interaction between the system of particles on the PAGB and the martensite substructure. A model taking these aspects into account has been developed and it will be discussed in detail together with some typical applications.

3:00 PM
Analytical Sharp-Interface Model for Massive Transformations in Substitutional Alloys: *Jiri Svoboda*[1]; Franz Dieter Fischer[2]; Ernst Gamsjäger[2]; [1]Institute of Physics of Materials, Zizkova 22, Brno 61662 Czech Republic; [2]Montanuniversität Leoben, Inst. of Mech., Franz-Josef-Strasse 18, Leoben 8700 Austria

In the initial period of the massive transformation thin concentration spikes are formed in front of the moving interface. After a transition the interface moves together with spikes in a stationary manner (with a constant velocity) as long as all parent phase is transformed. Very recently a new sharp-interface finite-interface-mobility model for diffusional phase transformations in substitutional alloys was developed by means of application of Onsager's thermodynamic extremal principle. Under the assumption of the constraint diffusional fluxes of substitutional components the model predicts the jumps across the migrating interface of all chemical potentials to be the same. These jump conditions together with the conservation laws at the migrating interface represent the proper number of contact conditions for the coupling of the interface migration and bulk diffusion in adjacent grains. Based on Fick laws the steady state concentration profiles were calculated analytically. The steady state solutions predic t no concentration profiles in the product phase and exponential spikes in the parent phase. The jumps of the concentrations (height of the spikes) as well as the driving force acting at the interface are calculated from the jump conditions for chemical potentials across the interface. The kinetics of the massive transformation is determined by the interface driving force and by the interface mobility. It can be shown that the dissipation due to diffusion in the spikes corresponds to the difference of the total driving force and the driving force acting on the interface. The analytical model predicts, that the massive transformation is controlled exclusively by the interface migration characterized by the interface mobility. Furthermore, the model shows that the half-thickness of spikes is proportional to the tracer diffusion coefficient of the component and inverse proportional to the interface velocity. The analytical model is used for simulations of the massive transformation kinetics and spike properties in Fe-Cr-Ni system. The results are compared with the new sharp-interface finite-interface-mobility model. The agreement is very good.

3:20 PM
Modeling the Kinetics of Bainite Formation in Low-Carbon TRIP Steels: *Fateh Fazeli*[1]; Matthias Militzer[1]; [1]University of British Columbia, The Ctr. for Metallurgl. Process Engrg., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Isothermal bainitic ferrite formation in the temperature range of 350 to 450°C for a model 0.18C-1.55Mn-1.7Si TRIP steels is investigated in terms of transformation kinetics and bainite morphology. Both single-phase austenite and austenite embedded in the ferrite matrix have been adopted as initial parent phase to study the role of nucleation condition and austenite grain size on the reaction kinetics. In addition, austenite-to-bainite transformation studies are extended to an Fe-0.6C-1.5Mn-1.5Si alloy with a nominal carbon content similar to that expected in the remaining austenite of a low-carbon TRIP steel at the start of the bainite formation. The experimental transformation results provide an appropriate database to examine the predictive capabilities of the existing modeling approaches for the bainite reaction, i.e. Johnson-Mehl-Avrami-Kolmogorov, diffusion and displacive methodologies. Employing these modeling philosophies to analyze the measured kinetics, a thorough comparison of the different models is provided and potential limitations of the existing models are delineated. The challenges will be discussed to replicate the bainite formation kinetics in TRIP steels from a fundamental point of view for a wide range of investigated temperatures where different bainite morphologies have been detected.

3:40 PM Break

3:50 PM
Modeling Martensitic Phase Transformations in the Presence of Plasticity: *Dnyanesh Nitin Pawaskar*[1]; [1]California Institute of Technology, Engrg. & Applied Sci., 1200 E. Calif. Blvd., MC 104-44, Pasadena, CA 91125 USA

An approach to producing hard, but tough, steel is through the judicious use of retained austenite. The optimization of properties in this approach requires an understanding of the amount of retained austenite, the amount of plastic deformation and the martensitic morphology. We describe a model that studies the martensitic phase transformation in the presence of plasticity. The model is implemented using the finite elements and captures the microstructure evolution and plasticity. Our results reveal the complex interaction between the driving force for transformation, the morphology and the plasticity.

4:10 PM
ITR: Computational Tools for Multicomponent Materials Design: *Zi-Kui Liu*[1]; Lonq-Qing Chen[1]; Qiang Du[2]; Padma Raghavan[3]; Jorge Sofo[4]; Stephen A. Langer[5]; Christopher C. Wolverton[6]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Steidle Bldg., Univ. Park, PA 16802 USA; [2]Pennsylvania State University, Dept. of Math., Univ. Park, PA 16802 USA; [3]Pennsylvania State University, Dept. of Computer Sci. & Engrg., Univ. Park, PA 16802 USA; [4]Pennsylvania State University, Dept. of Physics, Univ. Park, PA 16802 USA; [5]National Institute of Standards and Technology, Math. & Computational Scis. Div., 100 Bureau Dr., Stop 8910, MD 20899 Gaithersburg; [6]Ford Motor Company, Ford Rsch. Lab., MD3028/SRL, Dearborn, MI 48121-2053 USA

The medium size Information Technology Research (ITR) project funded by the National Science Foundation (NSF) in 2002 for five years will be presented. This collaborative research project involves two materials scientists (Zi-Kui Liu, Long-Qing Chen), a computer scientist (Padma Raghavan), a mathematician (Qiang Du), and three physicists (Stephen Langer, Christopher Wolverton, Jorge Sofo) from academia, industry and national laboratory. It is a synergistic effort that leverages the overlapping and complimentary expertise of the researchers in the areas of scalable parallel scientific computing, first-principles and atomistic calculations, computational thermodynamics, mesoscale microstructure evolution, and macroscopic mechanical property modeling. The research progress up to date will be reported in this presentation.

4:30 PM
Automating Multicomponent Materials Design: *Padma Raghavan*[1]; Keita Teranishi[1]; Zi-Kui Liu[2]; [1]Pennsylvania State University, Computer Sci. & Engrg., 308 Pond Lab., Univ. Park, PA 16802-6106 USA; [2]Pennsylvania State University, Matls. Sci. & Engrg., 0209 Steidle Bldg., Univ. Park, PA 16802 USA

We will report on the design of our client-server software architecture for determining the macroscopic properties of Al-Cu-Si-Mg systems. The server couples the following four main steps asynchronously through the use of intermediate databases: (1) computing ab-initio results, (2) CALPHAD data optimization to determine thermodynamic properties, (3) phase-field simulation to generate microstructures, and, (4) finite-element analysis using OOF to determine macrostructural properties. Our server is "grid-enabled" using Globus and can deploy compute-intensive simulations for any step on multiprocessors and networks of workstations; additionally, it can access and update intermediate databases which could also be distributed over a wide area network. To answer design questions posed by web-enabled clients, the server will instantiate specific simulations using a rule-based system. The latter allows the server to meaningfully explore the design space to determine feasible compositions of the four main steps and precomputed results in intermediate databases.

4:50 PM
Computational Design of an Experiment on Super-Rapid Resolidification of Bi Via Shock Wave Release: *Jeff D. Colvin*[1]; Alan Jankowski[1]; Wayne King[1]; Mukul Kumar[1]; Bryan Reed[1]; Babak Sadigh[1]; [1]Lawrence Livermore National Laboratory, Matls. Sci. & Tech. Div., PO Box 808, L-356, Livermore, CA 94550 USA

When a material transforms from one solid state to another through a melt-refreeze transition after undergoing shock compression, it is not known whether the material recrystallizes back into the initial microstructure. There are no data at all on recrystallized microstructures at very high cooling rates (> 1e+7 K/s). We are doing experiments using Bi, a metal that has an anomalous melt curve, meaning that it can resolidify at lower density than the liquid density. This implies that shock-melted liquid Bi will immediately resolidify upon release of the pressure, with an effective cooling rate up to 1e+10 K/s. We present details of the computational design of a laser-driven experiment to shock-melt Bi on the Hugoniot after preheating the sample to 400 K and driving a pressure pulse of 20-30 kbar into it using the technique of tamped ablation (Colvin et al., Phys.Plasmas 10/7, July 2003).

Cost-Affordable Titanium Symposium Dedicated to Prof. Harvey Flower: Low Cost Titanium

Sponsored by: Structural Materials Division, SMD-Titanium Committee
Program Organizers: M. Ashraf Imam, Naval Research Laboratory, Washington, DC 20375-5000 USA; Derek J. Fray, University of Cambridge, Department of Materials Science and Metallurgy, Cambridge CB2 3Q2 UK; F. H. (Sam) Froes, University of Idaho, Institute of Materials and Advanced Processes, Moscow, ID 83844-3026 USA

Wednesday PM Room: 206B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: James Sears, South Dakota School of Mines, Advd. Matls. Procg. Ctr., Rapid City, SD 57701 USA

2:00 PM
Chemical Reactions of $Ti_3Al(O)$, $Al_3Ti(O)$, and Al_2O_3 with NaOH in Aqueous Solution: *G. Adam*[1]; D. L. Zhang[1]; B. K. Nicholson[2]; [1]University of Waikato, Dept. of Matl. & Proc. Engrg., PB 3105, Hamilton New Zealand; [2]University of Waikato, Dept. of Chmst., PB 3105, Hamilton New Zealand

This study deals with the chemical reaction between $Ti_3Al(O)$, $Al_3Ti(O)$, and Al_2O_3 phases in the $Ti_3Al(O)/Al_2O_3$, or $Al_3Ti(O)/Ti_3Al(O)/Al_2O_3$ powders produced by high energy mechanical milling of Al, and TiO_2 followed by heat-treatment. Separating the Al_2O_3 phase out of the systems to give material with titanium-rich phases was the main goal behind this study. The reactions of $Ti_3Al(O)/Al_2O_3$, or $Al_3Ti(O)/Ti_3Al(O)/Al_2O_3$ wit aqueous solutions of NaOH were performed at elevated temperature to chemically leach the Al_2O_3. The reaction between $Ti_3Al(O)$ in $Ti_3Al(O)/Al_2O_3$ powder and NaOH solution proceed faster than the reaction between $Al_3Ti(O)$ in $Al_3Ti(O)/Ti_3Al(O)/Al_2O_3$ at the same condition. Under the same reaction conditions, the reaction between $Ti_3Al(O)$ and H_2O results in formation of amorphous TiO_2, while the reaction between $Al_3Ti(O)$ and H_2O results in formation of $TiO_{1.04}$.

2:30 PM
Influences of Alloy Chemistry and Microstructure on the Machinability of Titanium Alloys: *Yoji Kosaka*[1]; Stephen P. Fox[1]; [1]TIMET, Henderson Tech. Lab., PO Box 2128, Henderson, NV 89009 USA

In many applications, machining is a major contributor to the cost of parts. Titanium alloys are recognized to be difficult to machine materials due primarily to their low thermal conductivity, high chemical reactivity and low modulus of elasticity compared with steels. There have been a number of studies regarding the conditions and the characteristics in the machining of Ti-6Al-4V, which is the most widely used alpha/beta titanium alloy in aerospace and non-aerospace applications. It is generally understood that the strength is one of the factors that determines the machinability of titanium alloys. Microstructure is believed to influence the machinability of titanium alloys, although few studies have been reported on this subject. In the present work drill machinability was studied on various alpha/beta titanium alloys. The effects of alloying elements and the microstructure on the tool life of drills will be discussed.

3:00 PM Cancelled
Characterisation of Ex-Situ Ti-TiB2 MMC Parts Produced by Mechanical Milling

3:30 PM
Analysis-Based Design of Experiments for Improving Ingot Surface Finish During Plasma Arc Cold Hearth Melting: *Yuan Pang*[1]; Chengming Wang[2]; Frank Spadafora[3]; Kuang-O (Oscar) Yu[3]; Hao Dong[1]; Daniel L. Winterscheidt[4]; [1]Concurrent Technologies Corporation, Product & Proc. Analysis, 425 Sixth Ave., Regional Enterprise Tower, 28th Floor, Pittsburgh, PA 15219-1819 USA; [2]The Procter & Gamble Company, Sharon Woods Tech. Ctr., 11450 Grooms Rd., SWTC Box C-21, GR-CNE72, Cincinnati, OH 45242 USA; [3]RMI Titanium Company, R&D, 1000 Warren Ave., Niles, OH 44446-0269

USA; [4]Concurrent Technologies Corporation, Mfg. Programs, 100 CTC Dr., Johnstown, PA 15904-1935 USA

During casting of titanium ingots using plasma arc cold hearth melting (PAM), cold shuts can form on the surface. Cold shuts must be removed by machining to avoid cracks developing in billet conversion. Subsequent vacuum arc remelting is currently required to eliminate this defect. The present work uses analysis tools to guide the design of experiments for achieving single-melt casting. Computational results for casting of subscale ingots are presented to show the effects of process parameters on metal temperatures. The surface conditions of three ingots are compared to demonstrate the importance of heat distribution and the model capability. The best combination of torch parameters suggested by the analysis has resulted in the most improved surface finish, which is of sufficient quality that no machining is necessary. How this methodology can be scaled up to achieve balanced heating between the ingot center and edge in large-diameter ingots is also addressed.

4:00 PM

Thermodynamic Modeling of Ti-Al-V Alloys: *Vasisht Venkatesh*[1]; Fan Zhang[2]; [1]TIMET Corp, HTL, PO Box 2128, Henderson, NV 89009 USA; [2]CompuTherm, LLC, 437 S. Yellowstone Dr., Ste. 217, Madison, WI 53719 USA

Implementation of computational modeling tools to improve titanium processing, via extrinsic (e.g., temperature, pass reduction, etc.) and intrinsic (e.g., grain size, chemistry, etc.) parameter optimization, has resulted in significant cost savings through the elimination of shop/laboratory trials and tests. PANDAT, a thermodynamic modeling software developed by CompuTherm, LLC, is being utilized to design new alloys and control heat treatment processes based on alloy chemistry. A special thermodynamic database for titanium alloys was developed for the determination of beta transus, phase proportions, partitioning coefficients and phase boundaries. Model predictions of beta transus, phase fractions and element partitioning are compared to experimental results for a range of Ti-Al-V-Fe-O-C chemistries. In addition, the effect of these alloying elements on beta transus will also be discussed.

4:30 PM

A New Source of Affordable Titanium Alloy Powder: *A. B. Godfrey*[1]; S. R. Thompson[1]; C. M. Ward-Close[1]; [1]QinetiQ Ltd., Cody Tech. Park, Ively Rd., Farnborough, Hampshire UK

This paper will announce the establishment of a new process for the production of titanium alloy powder based on Electrolytic De-Oxidation (the FFC Cambridge Process). Details of the different titanium powder products will be given and scale-up of production with a new purpose built facility in the UK at QinetiQ, Farnborough will be described. The current state of titanium powder metallurgy technology will be reviewed and the potential of new high-quality titanium alloy powder to reduce the cost of finished components will be discussed.

5:00 PM

Improving Grindability and Wear Resistance of Titanium Alloys: *Toru Okabe*[1]; Masafumi Kikuchi[1]; Chikahiro Ohkubo[1]; Marie Koike[1]; Osamu Okuno[1]; Yutaka Oda[1]; [1]Baylor College of Dentistry, Dept. of Biomatls. Sci., 3302 Gaston Ave., Dallas, TX 75246 USA

Titanium is becoming an important material in dentistry, but not all of the properties of pure titanium lend themselves to dental applications. To optimize the properties of titanium for use in dentistry, alloying is necessary. This presentation will describe work on characterizing some titanium alloys for grindability, wear, and corrosion resistance, which are all important properties for dentistry. The metals tested included CP Ti, Ti-6Al-4V, Ti-15Mo-2.8Nb-0.25Si, Ti-13Zr-13Nb, Ti-15V-3Cr-3Sn-3Al, and experimental binary Ti-Cu alloys. Improvements in grindability and wear seemed to occur with the presence of the eutectoid in the alloy. The alloys tested had excellent corrosion characteristics in the oxidation potential range of the human mouth.

Dislocations: Dislocations in Complex Materials

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Materials Processing & Manufacturing Division, Structural Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)

Program Organizers: Elizabeth A. Holm, Sandia National Laboratories, Albuquerque, NM 87185-1411 USA; Richard A. LeSar, Los Alamos National Laboratory, Theoretical Division, Los Alamos, NM 87545 USA; Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

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March 17, 2004 — Location: Charlotte Convention Center

Session Chair: TBA

2:00 PM Invited

Connection Between Dislocation Fine Structure and the Creep Properties of Metallic Alloys: *Michael J. Mills*[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Engrg., Columbus, OH 43210 USA

The importance of dislocation fine structure (core structure, dissociation, etc.) in determining the yield behavior of BCC metals, and some intermetallic compounds such as Ni3Al, has been well established. By comparison, the crucial role that dislocation fine structure plays in determining the creep performance of materials has received relatively little attention. This presentation will highlight the recent development of dislocation models of creep deformation in several important alloy systems. These models have originated from a detailed knowledge of dislocation core structure and morphology based on extensive weak beam and high resolution TEM investigation. Two classes of behavior will be discussed. For cases in which dislocation cores are compact, as in a-Ti solid solution alloys and ordered g-TiAl, the appearance of jogged-screw dislocations is a ubiquitous microstructural feature following creep deformation. On the basis of these observations, the classic jogged-screw dislocation model for creep has been modified. This modified model appears to provide an excellent, quantitative prediction of the creep properties in these alloy systems, and provides important insight into important alloy-development issues such as the source of solute strengthening and finite-size effects. As a second example, the shearing of g' precipitates during high-temperature, low-stress creep of Ni-based superalloys will be discussed. A novel mechanism for this shearing process via the movement of dissociated a<100> dislocations has been identified. These dislocations move via a coupled-climb and glide process. Under the assumption that a<100> dislocation motion in the g' precipitates is the rate-limiting recovery process, a creep rate equation has been developed which provides promising agreement with measurements. The important role that computational modeling can play in further developing these ideas will also be highlighted.

2:35 PM Cancelled

Computer Simulation of Dislocation Dynamics in a Solid Solution

2:55 PM

Strengthening of Nb3Sn Composite: *Jingping Chen*[1]; Ke Han[1]; Peter Kalu[1]; [1]National High Magnet Field Laboratory, 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA

Fabrication of Nb3Sn type superconductor composites requires a final heat treatment at about 700°C to form an A15 phase embedded in an annealed Cu-Sn solid solution matrix. The high temperature annealing diminishes many strengthening mechanisms which relate to strain hardening by accumulation of dislocations and decreasing the size of the dislocation cells, and the A15-Nb3Sn is a brittle hard phase which renders further deformation strengthening of Cu-Sn difficult. The most feasible approaches for strengthening the composite are either solid solution or dispersion strengthening. This paper assesses and compares the efficiency of the solid solution and dispersion strengthening in superconductor composite by consideration of both the fabrication procedures and interaction between dislocation and particles. The size of the strengthening particles, which range from the solute of single atom to the second phase is used to link the solid solution and dispersion strengthening and to optimize the design of the superconductor composites.

3:15 PM
A Peierls Model of the Critical Stress to Transmit a Screw Dislocation from a Disordered to an Ordered Phase: *Yao Shen*[1]; Peter M. Anderson[1]; [1]Ohio State University, Matls. Sci. & Engrg. Dept., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

This presentation will describe the features and predictions of a 2D Peierls model of screw dislocation transmission from a disordered to an ordered phase. The incoming and outgoing slip planes, and also the interface, are described by a local shear stress-shear displacement relation that is derived from the gamma surface for that region. Consequently, the configuration of the dislocation core changes during the transmission process, including potential core spreading into the interface. An idealized geometry is considered in which the incoming and outgoing slip planes are oriented perpendicular to a coherent interface, so that no residual dislocation content remains in the interface following transmission. The effect of unstable stacking fault energies associated with the slip planes and interface, the mismatch in elastic moduli, and the antiphase boundary energy of the ordered phase are varied to understand their effect on the critical stress for dislocation transmission.

3:35 PM Break

3:50 PM Invited
Apt Characterization of Solute Segregation to Individual Dislocations: *Michael K. Miller*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Bldg. 4500S, MS 6136, Oak Ridge, TN 37831-6136 USA

The solute segregation to individual dislocations may be quantified by atom probe tomography. Dislocations may be observed in the field ion images by a change of the normal concentric atom terraces to spirals. Dislocations are detected in atom maps by enhanced levels of solute along linear features. The magnitude of the solute segregation may be quantified with the use of the maximum segregation envelope method. Examples will be presented of solute segregation to dislocations in nickel aluminides, neutron irradiated pressure vessel steels and a mechanically alloyed, oxide dispersion strengthened (MA/ODS) ferritic alloy. Research at the SHaRE User Center was sponsored by the Division of Materials Sciences and Engineering, U. S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the Office of Nuclear Regulatory Research, U. S. Nuclear Regulatory Commission under inter-agency agreement DOE 1886-N695-3W with the U. S. Department of Energy.

4:25 PM
Phase-Field Simulation of Spinodal Decomposition in a Constrained Film With Mobile Interfacial Dislocations: *Shen-Yang Hu*[1]; *Long-Qing Chen*[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Univ. Park, PA 16802 USA

Spinodal decomposition in binary alloy thin films with an arbitrary distribution of mobile interfacial dislocations, and subject to elastically substrate constraint is studied. Composition evolution and dislocation motion are described by Cahn-Hilliard and Ginzburg-Landau equations, respectively. Elastic solutions, derived for elastically anisotropic thin films with stress free surface and substrate constraint, are employed. Temporal evolution of Cahn-Hilliard equation under thin film boundary conditions (the fluxes normal to the surface and the interface are zero) is solved by a semi-implicit Fourier-spectral method. The effect of the mobility of dislocations, the types and distributions of dislocations on spinodal decomposition process and microstructures are studied.

4:45 PM
Towards a Full Analytic Treatment of the Hall-Petch Behavior in Multilayers: Putting the Pieces Together: *Lawrence H. Friedman*[1]; [1]Pennsylvania State University, Dept. of Engrg. Sci. & Mech., 212 Earth & Engrg. Sci. Bldg., Univ. Park, PA 16802 USA

Elastically inhomogeneous multilayer films are exploited for use as hard coatings. These films exhibit a strong dependence between the compositional wavelength and hardness. The hardness also depends on the granular texture within a layer. According to the model of A. Misra and H. Kung (Adv.Eng.Mat.3(4),2001,p217), the yielding mechanism of dislocation transmission across grains competes with the mechanism for dislocation transmission across layer interfaces. Here, both mechanisms of transmission are discussed in light of a semi-analytic model that is qualitatively and quantitatively distinct from the classical Hall-Petch Relation, but still rooted in dislocation pile-up theory. A full analytic treatment of the Hall-Petch-like size-effect in multilayers must account for anomalous stress-concentration at the layer interfaces, dislocation source characteristics, and a variable dislocation pinning stress. From scaling arguments, the criteria for such an equation are described, and an approximate analytic formula is suggested that meets these criteria.

5:05 PM
Formation of Misfit Dislocations in Nano-Scale Ni-Cu Bilayer Films: *David Mitlin*[1]; Amit Misra[1]; Michael Nastasi[1]; Velimir Radmilovic[2]; Richard Hoagland[1]; David J. Embury[3]; J. P. Hirth[1]; Terence E. Mitchell[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, 1 Cyclotron Rd., Berkeley, CA 94720 USA; [3]McMaster University, Dept. of Matls. Sci., Hamilton, Ontario L8S 4H3 Canada

We investigated the mechanism of interface dislocation formation in a 5.0 nm Ni film epitaxially deposited on 100 nm of (001) Cu. Threading dislocations that pre-exist in the Cu substrate extend into the coherent Ni overlayer during growth and propagate in the [110] and directions along the interface. These dislocations are perfect glide dislocations with mixed character and lying on the {111} Ni planes, and were by far the most numerous in the microstructure. Lomer edge dislocations lying on the (001) Ni-Cu interface were also detected, constituting approximately 5% of the total interface dislocation content. Closely spaced adjacent pairs of perfect glide dislocations having the same Burgers vector were commonly observed at the interface. This dislocation configuration, along with several others that were observed, is explained in terms of the ability of favorably oriented dislocations to cross-slip.

Educational Issues in Transport Phenomena in Materials Processing: Presentations and Panel Discussion

Sponsored by: Materials Processing & Manufacturing Division, TMS-Education Committee
Program Organizers: Matthew John M. Krane, Purdue University, Department of Materials Engineering, West Lafayette, IN 47907 USA; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Wednesday PM Room: 209B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Adam C. Powell, Massachusetts Institute of Technology, Matls. Sci. & Engrg., Cambridge, MA 02139-4307 USA; Matthew John M. Krane, Purdue University, Matls. Sci. & Engrg., W. Lafayette, IN 47907-2044 USA

2:00 PM Invited
Transport Education in the Materials Curriculum: Innovative Approaches, Exercises, and New Challenges: *Adam Clayton Powell*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 4-117, Cambridge, MA 02139-4307 USA

This talk will address three areas in teaching transport phenomena in the materials science and engineering curriculum as it is practiced currently and envisioned to change in the future at MIT. The first covers innovative approaches to teaching individual concepts and structuring a transport course to meet the needs of upper-level undergraduates in an existing curriculum. The second introduces a new internet resource with exercises which may be used for homework assignments or supplementary reading examples in this subject. The third discusses changes in course design to support new curriculum needs in materials processing, from computational modeling to micro- and macroeconomic implications.

2:30 PM
A Software Tool for Teaching Transport Phenomena: *J. Bernardo Hernández-Morales*[1]; Rafael Fernández-Flores[2]; J. Sergio Téllez-Martínez[1]; [1]Universidad Nacional Autónoma de México, Depto. de Ingeniería Metalúrgica, Facultad de Química, Edificio "D", Cd. Universitaria, México, D.F. 04510 Mexico; [2]Universidad Nacional Autónoma de México, Inst. de Matemáticas, Circuito Institutos s/n, Cd. Universitaria, México, D.F. 04510 Mexico

Students taking courses on transport phenomena usually solve a large number of problems but do not have the opportunity to explore each problem in detail, mainly due to time constraints. But solving a problem just to get a single answer does not give the students a feel for the impact of the difeerent variables on the final result. To help alleviate this problem we have designed a user-friendly software tool that allows the student to solve traditional problems but also to change any given variable or even to work with a range of values for the relevant variables and plot the results. The software was built using VisualBasic 6 but may be distributed as an stand-alone application. An

important feature of the software is its capability to handle different units for a given problem. However, the answer is always given in the SI system. The software deals with fluid flow, heat transfer and mass transfer problems.

2:50 PM
Introduction to Mathematical Modelling Through a Laboratory Exercise: *J. Bernardo Hernandez-Morales*[1]; J. Sergio Téllez-Martínez[1]; [1]Universidad Nacional Autónoma de México, Depto. de Ingeniería Metalúrgica, Fac. de Química, Edif. "D", Cd. Universitaria, México, D.F. 04510 Mexico

Transport phenomena are the basis of mathematical modelling of materials processing. However, there is usually little effort spent to make students aware of this fact in courses on transport phenomena. Through a laboratory exercise, the concepts of empirical and fundaments mathematical models are introduced stressing the shortcomings of the former one; it also serves as an introduction to the fundamental concepts of heat transfer. In this lab the newtonian cooling of a carbon steel bar is studied both experimentally and theoretically. Starting the heat transfer course with this exercise offers the students a problem that is easier to grasp than the traditional approach found in textbooks.

3:10 PM
Lecture Modules in Materials Science: An NSF Educational Activity at Rensselaer Polytechnic Institute: *Afina Lupulescu*[1]; Martin E. Glicksman[1]; [1]Rensselaer Polytechnic Institute, Dept. of Matls. Sci. & Engrg., 110 8th St., Troy, NY 12180 USA

Diffusion in solids, kinetics, solidification and crystal growth are broadly applicable subfields of materials. A 3-year NSF grant proposes developing electronic modules to parallel and amplify texts and notes already established for these quantitative courses at Rensselaer. The modules, designed for ease of adoption by others, consist of standalone PowerPoint files that succinctly present the content of each lecture topic, adding commentary, animation, and emphases that extend the limits of notes and texts. Given the prior success encountered with our modules for diffusion in solids, we extended the approach to include Kinetics, Solidification and Crystal Growth. Topical contents are selected on the basis of the books and course notes already in use to teach these courses. In addition, areas of conceptual, or mathematical difficulty encountered by our students and known to us will be explicated, and appropriate comments and examples added to alert students and instructors adopting our modules.

3:40 PM
Teaching and Learning Transport Phenomena and Analytical Methods in Materials Engineering to Graduate Students: Makhlouf M. Makhlouf[1]; *Richard D. Sisson*[1]; [1]Worcester Polytechnic Institute, Matls. Sci. & Engrg., 100 Inst. Rd., Worcester, MA 01609 USA

For more than 20 years, we have been teaching a one semester introductory graduate course in analytical methods for materials engineering to M.S. and Ph.D. students in Materials Science and Engineering. Chemical and mechanical engineering graduate students also take this course as an elective. The learning objectives for this course are: 1. Understand and be able to apply the fundamentals of heat transfer and solid state diffusion to materials processing and engineering problems. 2. Understand and be able to apply the fundamentals of mathematical modeling to analytical and numerical solutions to the heat equation and other problems. This course follows selected topics from the Bird, Stewart and Lightfoot approach to transport phenomena via the Geiger and Poirier text Transport Phenomena in Metallurgy. The materials processes used to illustrate the fundamentals and meet the learning objectives include: heat treating, carburization/decarburization of steel, quenching and cooling, phase transformations and hydrogen permeation and diffusion. In addition, examples from ceramics and coatings have frequently been included. In this presentation, we will discuss the various strategies that have been used to aid the student learning including laboratory experiments coupled with model simulations.

4:10 PM Break

4:30 PM Panel Discussion
This discussion will develop the themes introduced by the presentations, with a focus on changes in the way this topic will be taught in Materials Science and Engineering Departments in the future.

Electrochemical Measurements and Processing of Materials: Electrochemical Sensors and Measurements

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Aqueous Processing Committee, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, EPD-Waste Treatment & Minimization Committee
Program Organizers: Uday B. Pal, Boston University, Department of Manufacturing Engineering, Brookline, MA 02446 USA; Akram M. Alfantazi, University of British Columbia, Department of Metel & Materials Engineering, Vancouver, BC V6T 1Z4 Canada; Adam C. Powell, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139-4307 USA

Wednesday PM — Room: 212A
March 17, 2004 — Location: Charlotte Convention Center

Session Chairs: Daniel Josell, National Institute of Standards and Technology, Metall., Gaithersburg, MD 20899 USA; Xionggang Lu, Shanghai University, Sch. of Matls. Sci. & Engrg., Shanghai 200072 China

2:00 PM Invited
Interdiffusivity Measurements in Oxide Systems by Solid State Galvanic Cell Method: *Seshadri Seetharaman*[1]; Du Sichen[1]; Anders Jakobsson[1]; [1]KTH, Matls. Sci. & Engrg., Stockholm 10044 Sweden

A galvanic cell method incorporating ZrO_2-CaO solid electrolyte was successfully developed in the present laboratory. This technique has the advantage of monitoring of interdiffusion in oxide systems continuously. A typical cell in case of the study of the interdiffusivities in the system MO-NO can be written as (-) Pt, M (s), MO-NO (s) || ZrO_2-CaO || MO (s), M (s), Pt (+) where the oxide in the left-hand electrode is initially a mechanical mixture of the two oxides. As the interdiffusion proceeded, the EMF followed a typical S-type diffusion curve, starting from zero tending towards thermodynamic equilibrium. The results were treated by a model based on Dunwald-Wagner relationship in order to evaluate the diffusivities. The method was applied to NiO-MgO solid solutions as well as interdiffusion between NiO and CoO-doped MgO. The results compare well with the results of diffusion couple experiments as well as dynamic high temperature X-ray studies.

2:30 PM Invited
Development of Electrochemical Sensors and Their Application for Characterizing the Metal/Electrolyte System During Localized Corrosion: *Howard W. Pickering*[1]; Konrad G. Weil[1]; Ryan C. Wolfe[2]; Barbara A. Shaw[2]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 326 Steidle Bldg., Univ. Park, PA 16802 USA; [2]Pennsylvania State University, Engrg. Sci. & Mech., 212 Earth & Engrg. Scis., Univ. Park, PA 16802 USA

Electrochemical microprobes for in-situ monitoring of the electrochemical conditions inside recesses are needed to better understand how localized corrosion and other charge transfer processes occur in confined spaces. The electrode potential distribution can be routinely measured in cavities of > 100 μm opening dimension, but the ability to measure the concentrations of chemical species and their change with time in the presence of potential gradients is only recently becoming possible. Microprobes for pH and chloride ion will be described and results with these sensors will be presented and discussed for creviced iron samples in aqueous electrolytes. Available results reveal that these species change in concentration and distribution with time both during the induction period before crevice corrosion starts and during propagation of crevice corrosion. Preliminary data show that ionic concentrations peak at locations of highest metal dissolution rate on the crevice wall.

3:00 PM
Electrochemical Atomic Force Microscopic Studies on the Localized Corrosion in Sputtered Chromium Nitride Thin Film: *Jyh-Wei Lee*[1]; Peng-Tzu Chen[2]; Hung-Kai Chen[2]; Jenq Gong Duh[2]; [1]Tung Nan Institute of Technology, Dept. Mech. Engrg., #152, Sec. 3, Pei-Shen Rd., Shen-Ken, Taipei County 222 Taiwan; [2]National Tsing Hua University, Dept. Matls. Sci. & Engrg., #101, Sec. 2, Kuang-Fu Rd., Hsin Chu 300 Taiwan

Electrochemical atomic force microscopy (ECAFM) has become a useful tool to study the surface properties and reactions of corrosion down to atomic scale. With the aid of electrochemical control, detailed surface morphology changes can be in-situ observed. In this study, the sodium chloride aqueous solution was employed to be the corrosive media to investigate the corrosion behavior of a sputtered chromium nitride thin film on stainless steel substrate. In-situ time-lapse sequences of images were obtained and the real-time polarization curves were recorded using the ECAFM. Localized pittings were observed on the thin film surface. Traditional electrochemical tests were used as a comparison. Scanning electron microscopy and electron probe micro analyzer were also conducted to explore the chemical composition changes around corrosion pits. The correlation between surface defects and corrosion pits of the chromium nitride film were discussed.

3:30 PM Break

4:00 PM
Non-Destructive Microstructural Evaluation of Thermal Barrier Coatings by Electrochemical Impedance Spectroscopy: Srinivas Vishweswaraiah[1]; *Balaji Jayaraj*[1]; Tianbao Du[1]; Vimal Desai[1]; Yong-ho Sohn[1]; [1]University of Central Florida, Advd. Matl. Procg. & Analysis Ctr., PO Box 162455, 4000 Central Florida Blvd., Orlando, FL 32826 USA

Electrochemical impedance spectroscopy (EIS) is being developed as a non-destructive evaluation (NDE) technique of thermal barrier coatings (TBCs) for quality control and life-remain assessment. The durability and reliability of TBCs play an important role in the service reliability and durability of hot-section components in advanced turbine engines. In this investigation, EIS was employed to non-destructively examine TBCs, as a function of microstructure, chemistry and thermal exposure during isothermal and cyclic oxidation in air at 1121°C. TBCs examined in this study include electron beam physical vapor deposited (EB-PVD), air-plasma sprayed (APS) ZrO2-7wt.%Y2O3 (YSZ) and APS CaTiO3 (CTO) on NiCoCrAlY or (Ni,Pt) Al bondcoat. Impedance response of the specimens at room temperature was recorded at corrosion potential for TBCs using an electrolyte solution, and analyzed with an equivalent AC circuit based on the multi-layered micro-constituents of the TBCs. Specimens were then characterized by optical and e lectron microscopy. Relative changes in values of resistance and capacitance for constituent components in TBCs were correlated to the variation in the microstructure, chemistry and the degradation induced by thermal cycling.

4:30 PM
Response of Poly (3-Hexylthiophene) Thin Film Chemiresistor Sensor to Hydrazine Vapor: *Hong Yang*[1]; Bryan A. Chin[1]; [1]Auburn University, Matls. Engrg., 201 Ross Hall, Auburn, AL 36849 USA

Hydrazine is a widely used missile propellant that is highly toxic to humans even in low exposures. Therefore, on-line monitoring to rapidly identify an accidental release of hydrazine is required to protect personnel from dangerous exposures. In this research, a low cost, passive, and highly sensitive chemiresistor sensor for hydrazine vapor detection has been developed. The sensor is fabricated using standard microelectronic manufacturing techniques and is integrated with a hydrazine sensitive conducting polymer thin film. As the sensor is exposed to hydrazine vapor, a permanent reduction in the conductivity of the polymer film occurs. Preliminary results have shown the sensor has a rapid response time of less than a few seconds and a detection range from a few ppb to hundreds of ppm hydrazine. Experiments have been conducted to investigate the effects of temperature on the response of sensor. Possible theoretical models that describe the observed behavior are discussed and compared with the experimental results.

Failure of Structural Materials: Fatigue

Sponsored by: Structural Materials Division, SMD-Structural Materials Committee
Program Organizers: Michael E. Stevenson, Metals and Materials Engineering, Suwanee, GA 30024 USA; Mark L. Weaver, University of Alabama, Metallurgical and Materials Engineering, Tuscaloosa, AL 35487-0202 USA

Wednesday PM Room: 211A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Mark L. Weaver, University of Alabama, Metallurgl. & Matls. Engrg., Tuscaloosa, AL 35487-0202 USA; Michael E. Stevenson, Metals & Materials Engineers, Suwanee, GA 30093 USA

2:00 PM
Fatigue Issues in Structural Aircraft Components: *James F. Lane*[1]; [1]Applied Technical Services, Inc., Matls. Testing, 1190 Atlanta Industrial Dr., Marietta, GA 30066 USA

Fatigue is the most prominent failure mechanism associated with fixed-wing aircraft. Structural components are specifically at risk on aircraft, since their inability to perform adequately may result in catastrophic failure. Identification of the fracture mechanism and the cause are imperative for the mitigation of these failures. This presentation will identify some structural components that have experienced fatigue failures and identify the cause for the initiation of fatigue cracks.

2:30 PM
Crystallographic Facet Determination from Fatigue Fracture Surfaces: *Yun Jo Ro*[1]; Sean R. Agnew[1]; Richard P. Gangloff[1]; [1]University of Virginia, Matls. Sci. & Engrg., 116 Engineer's Way, Charlottesville, VA 22904-4745 USA

Fatigue crack growth in aerospace aluminum alloys is accelerated within a moist environment, as compared to vacuum. SEM fractography is employed to determine qualitative fatigue damage mechanisms for alloy C47A (Al-2.7Cu-1.7Li-0.7Zn-0.6Mn-0.3Mg) tested in vacuum, wet air and 3%NaCl. All samples exhibit a faceted appearance in a low stress intensity range (< 4MPa¡îm). Quantitative crystallographic facet orientations are determined by combining traditional stereology with Electron Back Scattered Diffraction (EBSD). In vacuum, the fracture surface is transgranular, strongly tortuous and faceted in the entire stress intensity range. Quantitative analysis reveals that 86% of facets orientations are within 10¯¬ of {111} in vacuum, suggesting a mechanism associated with intense slip localization due to with precipitate shearing. Cracking in the moist environments is transgranular and nearly mode I throughout the whole stress intensity range. Facet orientations cover the entire irreducible triangle between {001} and {011} with an absence of facets near {111}.

2:50 PM
Ultrasonic Effects on Fatigue Behavior of Small Cracks in Age-Hardened Al Alloys: *Qiang Chen*[1]; Norio Kawagoishi[1]; Qingyuan Wang[2]; Xishu Wang[3]; [1]Kagoshima University, Mech. Engrg. Dept., 1-21-40 Korimoto, Kagoshima, Kagoshima 890-0065 Japan; [2]Sichuan University, Dept. of Civil Engrg. & Mech., Chengdu, Sichuan 610065 China; [3]Tsinghua University, Dept. of Engrg. Mech., Beijing 100084 China

Fatigue strengths of two age-hardened Al alloys, 6061-T6 and 7075-T6, were investigated in the super high cycle region by using ultrasonic techniques. The main concern of the present study focused on the initiation and propagation of small cracks in the age-hardened Al Alloys subjected to ultrasonic fatigue at a frequency of ~19.5 kHz. The results were compared with those obtained under conventional rotating bending fatigue at a frequency of ~55 Hz in order to assess the influence of loading frequency on the fatigue properties of the Al alloys.

3:10 PM
Effect of Grain Boundaries on the Growth Behavior of Short Fatigue Cracks in a 2xxx Al-Alloy: *Jinxia Li*[1]; Tongguang Zhai[1]; Matthew Garratt[2]; Gary Bray[2]; [1]University of Kentucky, Chem. & Matls. Engrg, 177 Anderson Hall, Lexington, KY 40506 USA; [2]Alcoa Technical Center, 100 Tech. Dr., Alcoa Ctr., PA 15069 USA

A study on the fatigue properties and fracture characteristic of a high strength 2xxx Al alloy has been done. Specimens were cyclically deformed over a range of stress amplitudes with a self-aligning four point bending rig. It was found that short cracks propagated in a crystallographic mode and were deflected at each grain boundary they interacted with. Periodical steps were found which ran all the way

across each grain and varied from one grain to another in terms of the density on the fracture surface. The fracture stepping by the crack front at a grain boundary could be directly correlated with the twist angle of the crack face deflection across the grain boundary. The twist angle of the crack plane deflection across the grain boundary was the key factor that controlled the crack growth through the boundary. Denser fracture steps across one grain boundary indicated a larger twist angle of crack deflection at the grain boundary and gave rise to a lower crack growth rate. The work provided further evidence that supported the previously reported model that a grain boundary with a large twist component caused retardation of a short crack crossing the boundary in planar slip alloys.

3:30 PM
Fracture Mechanism of Al Alloys Under Ultrasonic Fatigue: *Qiang Chen*[1]; Norio Kawagoishi[1]; Qingyuan Wang[2]; [1]Kagoshima University, Mech. Engrg. Dept., 1-21-40 Korimoto, Kagoshima 890-0065 Japan; [2]Sichuan University, Dept. of Civil. Engrg. & Mech., Chengdu, Sichuan 610065 China

Ultrasonic fatigue tests were conducted for two age-hardened Al alloys, 6061-T6 and 7075-T6. The fracture surfaces of fatigued Al alloys were analyzed in detail under a scanining electron microscope and compared with those failed under conventional rotating bending fatigue. The objective of the present study is aimed at investigating the fracture mechanism that are involved in the ultrasonic fatigue of the age-hardened Al Alloys for better understanding the influence of loading frequency on the fatigue properties of the Al alloys. Special attentions were given to the formation and distribution of fatigue voids under ultrasonic loading as well as the effect of fatigue voids on the fatigue life.

3:50 PM Break

4:05 PM
Advanced Experimental and Modelling Assessment of Roughness-Induced Crack Closure Behaviour in Damage Tolerance Aluminium Alloys: *Kern Hauw Khor*[1]; Nicolas Kamp[1]; Harvinder Singh Ubhi[2]; Hiroyuki Toda[4]; Jean-Yves Buffiére[3]; Wolfgang Ludwig[3]; Ian Sinclair[1]; [1]University of Southampton, Engrg. Matls. Rsch. Grp., Sch. of Engrg. Scis., Highfield, Southampton, Hampshire SO17 1BJ UK; [2]QinetiQ Ltd., Cody Tech. Park, Ively Rd., Farnborough, Hampshire GU14 0LX UK; [3]GEMPPM INSA Lyon, 20 Ave. Albert Einstein, Lyon, Villeurbane Cedex 69621 France; [4]Toyohashi University of Technology, Dept. of Production Systems Engrg., Toyohashi, AICHI 441-8580 Japan

Fatigue crack closure, especially roughness-induced crack closure (RICC), has been recognized as a significant extrinsic factor in determining fatigue crack growth rates via the shielding of external cyclic loads to the crack-tip region. In this work, several 2024-type aluminium alloys, with different dispersoid contents and degrees of recrystallisation have been studied for their RICC micro-mechanistic influences on fatigue crack growth behaviour. Detailed assessment of texture variant distributions, grain boundary separations and three-dimensional crack surface topography was carried out using electron backscattering diffraction (EBSD) and surface profilometry. A novel analytical RICC model has been developed to predict crack closure levels, which is found to be comparable to the current experimental results. High resolution X-Ray microtomography was used in conjunction with a gallium grain boundary wetting technique to visualise and analyse the correlation between three-dimensional structure of the grains and fatigue crack behaviour.

4:25 PM
Effects of Local Grain Orientation on Fatigue Crack Nucleation and Growth in Multicrystalline FCC Metallic Materials: *Pedro D. Peralta*[1]; Krishnakumar Komandur[1]; Robert Dickerson[2]; [1]Arizona State University, Dept. of Mech. & Aeros. Engrg., Engrg. Ctr., G Wing, Rm. 346, Tempe, AZ 85287-6106 USA; [2]Los Alamos National Laboratory, Matls. Sci. & Tech., MST-8, MS G755, Los Alamos, NM 87545 USA

The effect of local crystallography was studied in multicrystalline Compact-Tension specimens of pure nickel and a cast Ni-based superalloy. Samples were characterized using Orientation Imaging Microscopy to obtain the crystallographic orientations of the grains ahead of a notch. A fatigue crack growth test was then performed to characterize the crack nucleation sites and initial path in relation to the grain orientations. Results showed that crack could either grow with small deviations through all the grains ahead of it or experience large deflections from modeI. This behavior is correlated to the crystallographic characteristics of the local crack environment and the stress state obtained using finite elements. Intergranular cracks were found to prefer high angle boundaries, whereas transgranular cracks had a tendency to nucleate and grow following slip lines of systems with high Schmid factors. In addition, crack path tortuosity was more pronounced in grains with loading axes close to <111>.

4:45 PM
Fractography of Two Solute-Strengthened Superalloys Haynes 230 and Hastelloy X High-Temperature Fatigue Failed in Air: *Y. L. Lu*[1]; P. K. Liaw[1]; G. Y. Wang[1]; L. J. Chen[1]; M. L. Benson[1]; S. A. Thompson[2]; J. W. Blust[2]; P. F. Browning[2]; A. K. Bhattacharya[2]; J. M. Aurrecoechea[2]; D. L. Klarstrom[3]; [1]University of Tennessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37996-2200 USA; [2]Solar Turbines, Inc., 2200 Pacific Hwy., PO Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 W. Park Ave., PO Box 9013, Kokomo, IN 46904-9013 USA

Low-cycle-fatigue and fatigue-crack-growth tests with different hold times were conducted on Haynesâ 230 and Hastelloyâ X at 816 and 927°C in air. The fractography was investigated. The fracture surfaces, especially at 927°C, were covered with oxides, which made the identification of the fracture mechanism difficult. Different cleaning techniques were tried to remove the oxides on the fracture surfaces. The cleaning results of different techniques were compared. The fracture mechanism was found to change from a transgranular mode to an intergranular one as the hold time or temperature increases. This work is supported by the Solar Turbines Inc., Haynes International, Inc., the Center for Materials Processing, the University of Tennessee, and the U. S. Department of Energy's Advanced Turbine System Program. We also acknowledge the financial support of the National Science Foundation, the Combined Research-Curriculum Development Program, under EEC-9527527 and EEC-0203415, and the Integrative Education and Research Training (IGERT) Program under DGE-9987548, and the International Materials Institutes (IMI) Program, under DMR-0231320, to the University of Tennessee, Knoxville, with Dr. D. Durham, Ms. M. Poats, Dr. W. Jennings, Dr. L. Goldberg, and Dr. C. Huber as contract monitors, respectively.

5:05 PM
Quantitative Analysis on Low Cycle Fatigue Damage: A Microstructural Model for the Prediction of Fatigue Life: *Hyun Jin Kim*[1]; Joon Sik Park[1]; Chong Soo Lee[1]; [1]Postech, Matl. Sci. & Engrg., San 31, Hyojadong, Nam-gu, Pohang, Kyungbuk 790-784 Korea

The present investigation is aimed to develop a model predicting the low cycle fatigue life of a material in relation to its microstructural variables. Since the grain size is important in determining the number and the size of persistent slip bands, which are considered as the major crack initiation sites, the low cycle fatigue life is expected to vary depending on the grain size. To achieve this goal, the concept of damage accumulation by multiple surface cracks has been adopted. Statistical analyses were carried out to investigate the relationship between the density of surface cracks and the low cycle fatigue damage. Also, one-dimensional percolation solution was used to describe the interactions between multiple surface cracks. To verify the suggested model, low cycle fatigue tests were conducted for the steels with the various grain sizes. The results showed good agreements between the experimental data and the predicted curve.

General Abstracts: Session VII

Sponsored by: TMS
Program Organizers: Adrian C. Deneys, Praxair, Inc., Tarrytown, NY 10591-6717 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Wednesday PM Room: 208B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Arne Dahle, University of Queensland, Div. of Matls. Engrg., Brisbane, QLD 4072 Australia

2:00 PM
Control of Grain Size and Age Hardening in AA2618 Forgings Processed by Rapid Infrared Radiant Heating: *Hui Lu*[1]; Puja B. Kadolkar[2]; Teiichi Ando[1]; Craig A. Blue[2]; Rob Mayer[3]; [1]Northeastern University, Mech., Industrial & Mfg. Engrg., 360 Huntington Ave., 334 Snell Engrg. Ctr., Boston, MA 02115 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831 USA; [3]The Queen City Forging Company, 235-B Tennyson St., Cincinnati, OH 45226 USA

Effects of rapid infrared (RI) radiant processing on the microstructure and mechanical properties of AA2618 aluminum forgings were investigated. Extruded bars were forged after RI-preheating to 425°C and solutionized at 530°C also in a RI furnace for different soaking times. Rapid preheating prior to forging produced uniform grain sizes (27-32 μm) in solutionized specimens while conventional furnace preheating produced coarser grain sizes (~40 μm) in the same 2618 alloy. Use of RI preheating for forging and subsequent solution heat treatment produced sufficient age hardening in 2618 forgings, which was verified by hardness and tensile tests. RI processing potentially leads to energy-efficient, low cost commercial production.

2:25 PM
Magnetostriction of Nanocrystalline Fe2Tb Alloy: *Pekka Reino Ruuskanen*[1]; [1]Technical Research Centre of Finland, Processes, PO Box 1607, FIN-33101, Tampere Finland

High magnetostrictive nanocrystalline Fe2Tb was prepared with a solid state mechanical alloying method. As starting materials elemental Fe and Tb were used. As-alloyed powder consists of amorphous and nanocrystalline phases. As-alloyed powder was cold compacted followed by annealing at 500°C. This temperature is 30°C above the crystallisation temperature of the as-alloyed powder. After annealing the microstructure of the material consists of a single phase nanocrystalline Fe2Tb the crystal size being 7-8 nm. The magnetostriction of the nanocrystalline material was measured with and without a biasing magnetic field. Using a biasing magnetic field a maximum magnetostriction l (Dl/l) = 1580*10-6 was measured. This value was measured without a prestress. The stress sensitivity of the magnetostriction of the nanocrystalline material was found to be small.

2:50 PM
A Model for Delayed Hydride Cracking in Zirconium Alloys: *Young S. Kim*[1]; Yeo B. Yoon[1]; Sung S. Kim[1]; Yong M. Cheong[1]; In S. Kim[2]; [1]Korea Atomic Energy Research Institute, Zirconium Team, PO Box 105, Yuseong, Daejeon 305-353 Korea; [2]Korea Advanced Institute of Science and Technology, Nucl. Engrg. Dept., Kuseong-dong, Daejeon 305-701 Korea

The objective of this study is to identify factors governing the crack growth velocity and threshold stress intensity factor during delayed hydride cracking (DHC) of zirconium alloys and to propose a DHC model that can satisfactorily explain all the unresolved issues related to DHC. To this end, DHC tests have been conducted at temperatures ranging from 100 to 300°C on Zr-2.5Nb alloys with different yield strengths and hydrogen concentrations of 12 to 100 ppm. With increased supersaturated hydrogen concentration, DHC velocity (DHCV) of the Zr-2.5Nb alloys has been increased to a constant and its threshold stress intensity factor, KIH has also been decreased. Thus, DHCV and K_{IH} can be nicely described as a function of the supersaturated hydrogen concentration over the terminal solid solubility for dissolution (TSSD) independent of temperatures. Another factor to control DHCV is the yield strength of the Zr-2.5Nb pressure tube: faster DHCV for the Zr-2.5Nb tubes with a higher yield strength. Consequently, we conclude that delayed hydride cracking of Zr-2.5Nb pressure tubes is governed by their yield strength and a difference in the hydrogen concentration in solution between the crack tip and the bulk regions. Based on these findings, we suggest a new DHC model: a driving force for DHC is a concentration difference of hydrogen between the bulk region and the crack tip incurred by the nucleation of reoriented hydrides only at the crack tip subjected to applied tensile stress while its velocity is governed by a hydrogen concentration gradient determined by a stress gradient formed ahead of the crack tip. This DHC model has been validated through a supplementary experiment demonstrating that little supersaturated hydrogen concentration in the bulk region of zirconium alloys suppresses DHC and by correlating the yield strength effect and the striation spacings.

3:15 PM
A Hot Fracture Criterion for Processing of High-Mg 5xxx Aluminum Alloys: *Paul T. Wang*[1]; [1]Alcoa Inc., Proc. Tech., 100 Tech. Dr., Alcoa Ctr., PA 15069 USA

An investigation of hot fracture behavior of high Magnesium 5xxx aluminum alloys is pursued under both tensile and shear tests at various temperature and strain rate conditions. The test results are used for the purpose of constructing a hot fracture limit diagram. The diagram is composed of hot ductility as a function of strain rate, temperature, and hydrostatic stress, and represents two fracture modes - ductile and hot short. To further confirm these two fracture modes, fracture surfaces were analyzed by optical metallography and FEG-SEM. This leads to the development of a multiple-mode fracture criteria suitable for use in thermomechanical processing of aluminum. Hot rolling of multiple pass situations in the frame of Arbitrary Lagrangian-Eulerian encompassed the fracture criterion are simulated and compared with lab rolling experimental results.

3:40 PM Break

3:50 PM
Ageing Hardening and Softening Behavior of the Mg-RE Alloys: *Y. B. Xu*[1]; J. H. Zhang[1]; [1]Shenyang National Laboratory for Materials Science, Inst. of Metal Rsch., Chinese Acad. of Scis., Shenyang 110016 China

Ageing hardening and softening behavior of the Mg-RE alloys has been investigated by TEM. The results show that the first hardening-peak occurs at 475K for about 2 hours, and then the hardness of the alloy decreases sharply. The second hardening-peak appears during the following ageing. After ageing at 523K for 600 hours, the alloy starts to be softening. Investigations by TEM reveal that the first hardening-peak is due to the occurrence of the dispersed precipitates with the sizes of 5nm in diameter, and the sharp fall of the hardness results from the dissolution of the MgY phases. â-precipitation leads to the second hardening of the alloys, and the alloy softening again is proposed to attribute to the growth of the â-precipitates.

4:15 PM Cancelled
The Way to Reduce the Cost of Titanium Alloys

4:40 PM
Microstructure and Creep Behavior of a Cast Mg-RE Alloy: *Guiying Sha*[1]; Yongbo Xu[1]; Enhou Han[1]; [1]Shenyang National Laboratory for Materials Science, Inst. of Metal Rsch., Chinese Acad. of Scis., Shenyang 110016 China

The compress creep tests of a cast Mg-RE alloy were carried out at constant stress on Mayes fatigue machine in air-creep behavior of the alloy has been investigated at different temperatures. Observations by SEM and TEM show that the creep resistance of the alloy is relatively high at 473K under stress ≤ 100MPa and the steady creep rate is lower than s-1. The eutecticßphase comprised Ce, La, Nd etc elements and dynamic precipitation during creep is proposed to be the principal strengthening mechanism of the alloy - whereas the main reason for the decrease of the creep resistance is attributed to be twins shear deformation and cracking of the eutecticßphases.

5:05 PM
Magnesium Semi-Fabricated Products at Industrial Scale: *Juan Gomez-Cordobes*[1]; [1]Rauch Melting Technique, Innovation, Fichtenweg 3, Gmunden, Upper Austria 4810 Austria

In the year 2001, the European production of aluminium semi-fabricated products (rolled, extruded and forged products) did triplicate the shipments from aluminium casting processes in any of its forms. Due to quality, price and availability issues, the market structure in the magnesium world is drastically different, and while casting shipments world wide surpassed 130.000 tons in 2002, wrought alloys applications are still almost non existent. Based on the learnings gathered in two parallel projects in operation since 2002, the following paper provides an insight into the manufacture of magnesium semi-fabricated products at industrial scale. The study concentrates on the production of the material needed for the subsequent forming processes. It is in this area where a newly developed liquid metal handling and feeding concept has allowed for the production at industrial scale of high quality bars, billets and thin sheet to be subsequently utilised in forging, extrusion and rolling processes.

General Abstracts: Session VIII

Sponsored by: TMS
Program Organizers: Adrian C. Deneys, Praxair, Inc., Tarrytown, NY 10591-6717 USA; John J. Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland 00160 New Zealand; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Wednesday PM Room: 207D
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Margaret Hyland, University of Auckland, Dept. of Chem. & Matls. Engrg., Auckland New Zealand

2:00 PM
Morphology of Resultant Surfaces from Electro Discharge Machining and Electro Discharge Sawing: *V. Narsimha Rao*[1]; N. N. Ramesh[2]; P. Laxminarayana[3]; [1]CITD, Balanagar, Hyderabad 500 037

India; [2]Osmania University, Mech. Engrg. Dept., Hyderabad 500 007 India; [3]Osmania University, Mech. Engrg. Dept., College of Tech., Hyderabad 500 007 India

Electrical discharge machining (EDM) employs high frequency sparks for machining difficult to machine metals. Its slow erosion rates renders it unsuitable for large and faster cuts like sawing. A modified process called Electro discharge sawing (EDS) has been developed which is a hybrid process. In place of dielectric an electrolyte, Sodium silicate plus water, is used to promote arcing in place of sparking, to produce a passivating film on work surface to prevent short circuits and to quench the eroded debris and carry them off the inter electrode gap. The electrode is a steel belt continuously moving through ceramic guides. The erosion rates are extremely high. Morphological studies of eroded surfaces under scanning electron microscope for mechanism of erosion indicate the EDM surfaces to be matty with overlapping spark craters whereas EDS surfaces shows pitting erosion from arcing and short circuiting pulses.

2:25 PM

Microstructure-Property Studies of Sputter Deposited Cu(1-x)Ta(x) Thin Films: *Shannon Marie Jackson*[1]; J. Michael Rigsbee[1]; [1]North Carolina State University, Dept. of Matls. Sci. & Engrg., 229 Riddick, Campus Box 7907, Raleigh, NC 27695-7907 USA

A series of $Cu_{1-x}Ta_x$ (x = 0.05 to 0.5) thin films have been produced by DC magnetron sputter deposition using co-deposition (alloy) and sequential (layered) deposition modes at room and elevated temperatures. The nanoscale microstructures of these non-equilibrium "alloy" films have been investigated chemically and structurally using x-ray diffraction, scanning electron microscopy and high resolution, z-contrast transmission electron microscopy. The effects of Ta composition, deposition temperature and deposition mode on microstructure will be discussed, along with nanoindentation studies of selected samples.

2:50 PM

Effect of Material Characteristics on the Surface Texture From Burnishing Processes: *Voleti Sri Ram Murti*[1]; *K. Sudhakar*[1]; M. Rama Linga Reddy[1]; Syed Abraruddin Hasan[1]; [1]Osmania University, Mech. Engrg. Dept., Hyderabad, Andhra Pradesh 500 007 India

The present paper seeks to study the significant influence of Material Characteristics on the final texture of burnishing surfaces generated by Cold working of roughness peaks by hard pressed ball or roller. The analytical approach includes not only the conventional roughness indices but also their spatial and shape characteristics. The stratification of the surface texture was analyzed through its structure function. Plastic flow from cold working of roughness peaks resulting in their flattening appears to be the dominant mechanism in ductile materials. However rupture of the peaks cannot be ruled out which is the dominant mechanism for brittle materials. In Powder Metallurgy materials the closure of voids results in lower plastic flow. The stratification is prominently illustrated and analyzed through the structure function which proves to have highest sensitivity to identify stratification characteristics.

3:15 PM

High Temperature Performance of Novel Ti-Al-Oxide Composite Coatings Produced by Plasma Spraying: *Jing Liang*[1]; Byung-Young Choi[2]; Kai Zhang[1]; Deliang Zhang[3]; Wei Gao[1]; [1]The University of Auckland, Dept. of Chem. & Matls. Engrg., PB 92019, 20 Symonds St., Auckland New Zealand; [2]Chonbuk National University, Korea, Sch. of Advd. Matls. Engrg., Rsch. Ctr. of Industrial Tech., Chonju 561-756, Chonbuk Korea; [3]University of Waikato, Dept. of Matls. & Procg. Engrg., Hamilton New Zealand

TiAl/Al2O3 composite powder was produced by mechanical alloying, sintering and grounding. Thermal plasma spraying was used to coat this powder onto Ti-6Al-4V alloy samples. The high temperature oxidation and hot corrosion behaviour of the coatings was studied in dry air and Na2SO4 + NaCl vapour, respectively. The results showed that Ti-Al-oxide coatings have a much improved oxidation and hot corrosion resistance compared to the substrate Ti-6Al-4V alloy. The oxide formed on the surface of the coatings after high temperature exposure is composed of a mixture of a large portion of Al2O3 and a small portion of TiO2. The oxide scales have excellent adhesion to the coatings, showing superior scale spallation resistance. The mechanisms with which the coatings improved the oxidation resistance were studied based on microstructural analysis. It is believed this development can provide a new generation of coatings to the Ti based alloys which will be able to raise the application temperature of Ti alloys from 650ºC to 800-900ºC.

3:40 PM Break

3:50 PM

Construction and Applications of Wear Maps for Ferrous Thermal Spray Coatings: *Afsaneh Edrisy*[1]; Tom Perry[2]; Yang-Tse Cheng[2]; Ahmet T. Alpas[1]; [1]University of Windsor, Mech., Auto. & Matls. Engrg., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]General Motors, R&D Ctr., Matls. & Proc. Lab, MC 480-106-224, 30500 Mound Rd., Warren, MI 48090-9055 USA

Ferrous coatings with nominal 1020 composition were produced using two types of thermal spray deposition processes namely PTWA, and HVOF on aluminum alloy substrates in order to assess their wear resistances. Micomechanisms that control wear resistances of the coatings included deformation of the steel splat tips and their fracture that resulted in high wear rates at high loads, and the formation of oxide layers on the contact surfaces. The formation and spallation of friction-induced oxides controlled wear rates at high sliding velocities. The composition and thickness of the oxides that formed on the surfaces depended on the production methods. The results were summarized in the form of Wear Mechanism Maps using normal load vs. sliding speed axes on which the dominant wear mechanisms in each wear regime were marked. The wear maps help illustrate the effects of composition and structure on wear behavior of thermal sprayed coatings for automotive engine applications.

4:15 PM

A Comparison of Surfactant Aggregation Transition Concentration Determined Using Electrochemical Quartz Crystal Microbalance Tests, Surface Tension Measurements, Corrosion Inhibition Tests, and Theoretical Methods: *Dong Youp Ryu*[1]; Michael L. Free[1]; [1]University of Utah, Dept. of Metallurgl. Engrg., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

The surfactant aggregation transition concentration (atc) is the concentration associated with the transition from surfactant monomers to micelles in solutions. The atc is also related to the transition from monolayer to multilayer coverage of surfactant molecules at interfaces. Usually these values are similar although they are usually not identical due to surface energy issues associated with interfacial adsorption. These values can be determined by various methods such as Electrochemical Quartz Crystal Microbalance (EQCM), surface tension, and corrosion inhibition tests as well as theoretical methods. This paper discusses various approaches for determining the atc values as well as the factors that affect atc values.

4:40 PM

Microstructural Refinement of Fe-40a%Al Alloy by Thermomechanical Processing: Tomasz Sleboda[3]; *Norman S. Stoloff*[1]; Jason Kane[1]; Roger N. Wright[1]; David J. Duquette[1]; Seetharama C. Deevi[2]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Engrg., 110 8th St., Troy, NY 12180 USA; [2]Philip Morris USA, Richmond, VA 23234 USA; [3]Rensselaer Polytechnic Institute, Matls. Sci. & Engrg., Troy, NY 12180 USA (on leave from the AGH University of Science and Technology, Metall. & Matls. Sci. Dept., Al. Mickiewicza 30, 30-059 Krakow, Poland)

This research is focused on thermomechanical grain refinement of a Fe-40at.%Al alloy. Hot isostatic pressing of as-received and ball milled powder at relatively low pressures resulted in fully densified products. Ball milling caused significant powder particle size reduction, resulting in a finer structure after HIPing. Fully densified, as-received as well as ball milled, FeAl powders were tested in compression on a Gleeble thermomechanical simulator in the temperature range 700°C to 1100°C. Flow stress curves and microstructural changes were investigated after Gleeble tests. Room temperature compression tests revealed that grain refinement strongly influenced the mechanical properties of the investigated material. A separate part of this investigation involved a study of the evolution of recovery, recrystallization and grain growth in cold-rolled Fe-40Al sheet. Anneals were carried out over a temperature range of 100-1200°C for various selected times. Structural changes were characterized by o ptical microscopy and changes in mechanical properties were characterized by microhardness testing. Research supported by Philip Morris USA.

High Risk Technologies in Metallurgy with Commercial Potential: Session II

Sponsored by: TMS, PGA-Public and Governmental Affairs Committee
Program Organizers: Jean-Louis Staudenmann, NIST, Advanced Technology Program, Division 473, Gaithersburg, MD 20899-4730 USA; Diran Apelian, Worcester Polytechnic Institute, Metal Processing Institute, Worcester, MA 01609-2280 USA

Wednesday PM Room: 210A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Diran Apelian, Worcester Polytechnic Institute, Metal Procg. Inst., Worcester, MA 01609-2280 USA; Jean-Louis Staudenmann, NIST, Advd. Tech. Prog., Gaithersburg, MD 20899-4730 USA

2:00 PM
Nucleated Casting for the Production of Large Superalloy Ingots: *William T. Carter*[1]; Robin M. Forbes Jones[2]; [1]General Electric Global Research Center, Ceram. & Metall. Tech., One Rsch. Cir., Bldg. K1, Rm. 279MB, Niskayuna, NY 12309 USA; [2]Allvac, An Allegheny Tech. Co., Monroe, NC USA

To reduce the cost of electricity generated by land based gas turbines, designers and manufacturers of turbines strive to improve the design of future machines by increasing the thermodynamic efficiency, electrical output and reliability. Doing so requires higher firing temperatures and larger turbines. This, in turn, increases the temperatures and stresses on critical components such as the turbine wheel. To accommodate these increased temperatures and stresses, turbine wheel designers have changed the materials of construction from iron based alloys to increasingly more complicated nickel based superalloys. For example in a recent design change, one manufacturer changed from Alloy 706 to Alloy 718, and for future turbines, is projecting further increases in turbine wheel size. The current manufacturing process for making superalloy wheels results in only one wheel from each cast ingot. It is unlikely that the projected future increases in turbine wheel size can be accommodated using the standard triple melt ingot casting technique, which is limited by metallurgical requirements to maintain properties and minimize segregation. New casting technology will be required. A review of several promising alternative technologies resulted in the a selection of Nucleated Casting as the one that offered the most promise for making the larger ingots. Nucleated Casting separates the melting process from the solidification process, providing additional degrees of freedom in process control. However, several new technologies must be developed, making it a high technical risk solution with a long time before commercialization. A long-term technology development strategy was identified in which a research partnership between a turbine manufacturer and a ingot supplier, reinforced by a US Government program, was an essential ingredient. To increase the chances of successful completion within the four-year timeframe, a detailed list of the technical risks was identified and the project was structured to mitigate each of the risks. The development plan entails concurrent subscale process development along with development of validated process models. A successful program will allow the use of validated models to extrapolate findings to a commercial scale suitable for use in the next generations of gas turbines.

2:25 PM
Application of Composite Materials and Design for Advanced Wind Turbine Rotor: *Steven R. Kopf*[1]; [1]AdvanTek International, LLC, 7 Creek Pkwy., Ste. 770, Boothwyn, PA 19061 USA

Instantaneous Power Control™ (IPC™) is a new and revolutionary concept in rotor design and power control for utility scale wind turbines that offers the potential of increasing the rotor diameter by 30% without an equivalent increase in the capital cost of the system. IPC™ integrates an active control system, featuring sensors, high bandwidth actuators, and a computer controller, into wind blade design, and is expected to bring down wind energy cost by capturing more power at lower wind speeds without overloading the gearbox and generators during high winds. However, in order to successfully demonstrate this advanced rotor so that it can be commercialized, several high-risk technological hurdles must be overcome. One of these risks is the optimal application of advanced composite materials to the IPC™ rotor and control system. The IPC™ blades are much longer and more slender than a state-of-the-art blade system. As a result, the blade root bending moment and fatigue loads are much higher. Material selection and design methodology are critical to insuring that the blade meets commercial cost targets. Another identified risk area is fact that the power performance of the IPC™ blades is extremely sensitive to both manufacturing tolerances and structural deflections during turbine operations. Small variations in certain critical blade geometries can negate the advantage gained by the increase in rotor diameter. The paper will present an overview of the methodology that is being used to overcome these high-risk technological hurdles to allow the successful demonstration and subsequent commercialization of the IPC™ rotor and control technology.

2:50 PM
Heat Treating Aluminum Alloys Utilizing Fluidized Bed Quenching: *Jay Keist*[1]; Chuck Bergman[1]; [1]Technomics, LLC, 17200 Medina Rd., Plymouth, MI 55447 USA

To achieve optimum mechanical properties after heat treating, rapid quenching is critical to obtain a supersaturated solution. To obtain rapid quenching, manufacturers often employ water as a quenchant. The drawback of water is that a vapor barrier initially forms during the quenching which leads to variations in quenching rates from one section to another within the part. The large variations in quenching rates often manifests as unacceptable part warpage and high residual stresses. An attractive alternative is quenching in a fluidized bed. A vapor barrier is not formed during fluidized bed quenching thus drastically reducing residual stresses and warpage. We analyzed the quenching curves and resulting mechanical properties for various castings of aluminum alloys 319, 354, and 356. Water and fluidized bed quenching was compared at various temperatures. Smaller parts (less than 10 lbs) exhibited similar mechanical properties between water quenched and fluidized bed quenched samples. Despite the significantly longer quenching times, larger parts quenched in fluidized beds exhibited only a small decrease in mechanical properties as compared to water quenched samples. Furthermore, quenching in fluidized sand at relatively high temperatures (300 to 400°F) still yielded acceptable properties. The ability to quench at aging temperatures may lead simplified heat treating processes by quenching in the aging bed directly after solution.

3:15 PM Break

3:30 PM
Triboelectric Purification, Rapid Optical Analyses and Metallurgical Characterization of Powdered Metals: *John M. Stencel*[1]; Tapiwa Z. Gurupira[1]; Charles L. Jones[1]; Lou Lherbier[2]; David Novotnak[2]; Gregory Del Corso[2]; [1]Tribo Flow Separations, 1525 Bull Lea Rd., Ste. 10, Lexington, KY USA; [2]Tribo Flow Separations, 600 Mayer St., Bridgeville, PA USA

The economic manufacturing of high alloy metal powders into full dense parts has been impaired for years by variations in powder cleanliness. Ceramic inclusions inherent in most gas-atomized, induction melted powders can seriously affect the quality of some consolidated parts. Current remedies include screening the powder to finer mesh cuts to eliminate large inclusions and/or conducting extensive testing of the consolidated material to ensure quality requirements prior to customer shipment. Besides being time consuming, metallurgical techniques like tensile, Charpy impact, ultrasonic, water elutriation, metallographic and EDM tests that are used to evaluate quality only examine a small faction of the material actually produced. The development of triboelectric purification (TEP™) is a promising manufacturing process that can remove significant amounts of ceramic impurities from metal powders. Its application could increase usable atomized yields by 10-20%, while at the same time reduce quality losses. TEP™ also eliminates the uncertainty associated with evaluating a small sample of material by various test procedures since all of the powder from an atomized lot is purified by the process. To prove the efficacy of TEP™ in a timely manner, material subjected to purification can be characterized by using a newly developed, automated optical microscopic analysis (OMA™) technique that obviates the need for using the costly and time-consuming metallurgical tests. Because statistically significant OMA™ data are attainable within a 24 hour time period, a rapid assessment of atomized powder purity is possible. Results obtained on high-speed steel and prosthetic implant alloy, gas atomized powders using TEP™ processing and OMA™ analyses will be described.

3:55 PM
Measurements, Standards, and Data: Overcoming Technical Barriers to the Reductions of the Time-to-Market Cycle for Advanced Metals: *Clare M. Allocca*[1]; [1]National Institute of Standards and Technology, Matls. Sci. & Engrg. Lab., 100 Bureau Dr., Stop 8500, Bldg. 223/B316, Gathersburg, MD 20899-8500 USA

Current business realities present continuing challenges for the commercial application of advanced metals. Customers demand the devel-

opment and adaptation of materials more rapidly and efficiently than ever before. The simultaneous need for the reduction of the time-to-market cycle and final cost drives the need for new approaches to materials development. These drivers have placed renewed emphasis on the role of measurement science and computational modeling as enablers of advanced metals development. This presentation will describe some of the ways in which the NIST Materials Science and Engineering Laboratory has responded to these needs: Standard tests, metrology and models that can enable the accurate determination of sheet metal mechanical response, which in turn reduce the die tryout time for die sets for automotive bodies; Measurements, models, data, and standards needed by the microelectronics industry to evaluate the national standard Pb-free alloy and processes needed to use the alloy in circuit board assembly; Combinatorial methods for the acceleration of materials discovery and the reduction of development time of engineered materials; GMR; Superalloys. These and other activities will be discussed. Methods for interactions with NIST will also be addressed NIST Combinatorial Methods Center, and facilities such as the NIST Center for Neutron Research.

Hume Rothery Symposium: Structure and Diffusional Growth Mechanisms of Irrational Interphase Boundaries: Session VI

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, EMPMD/SMD-Alloy Phases Committee, MPMD-Phase Transformation Committee-(Jt. ASM-MSCTS)
Program Organizer: H. I. Aaronson, Carnegie Mellon University, Department of Materials Science and Engineering, Pittsburgh, PA 15213 USA

Wednesday PM Room: 208A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: B. C. Muddle, Monash University, Sch. of Physics & Matls. Engrg., Victoria 3800 Australia

2:00 PM General Discussion

International Laterite Nickel Symposium - 2004: Process and Operational Lessons Learned - Part II

Sponsored by: Extraction & Processing Division, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Process Fundamentals Committee, EPD-Process Mineralogy Committee, EPD-Pyrometallurgy Committee, EPD-Waste Treatment & Minimization Committee
Program Organizer: William P. Imrie, Bechtel Corporation, Mining and Metals, Englewood, CO 80111 USA

Wednesday PM Room: 217B/C
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: John G. Schofield, Pyrometallurgical Consultant, Nanoose Bay, BC V9P 9G5 Canada; Steve C.C. Barnett, V-P HSEQC Stainless Steel Materials, BHP-Billiton, London SW1V 1BH UK

2:00 PM
The Steps Required to Meet Production Targets at PT INCO: A New Innovative Business Strategy: *Chris Doyle*[1]; Management & Staff[1]; Management & Staff[2]; [1]PT INCO, Sorowako, Sulawesi Selatan 91984 Indonesia; [2]Inco Technical Services Ltd., 2060 Flavelle Blvd., Sheridan Park, Mississauga, Ontario L5K 1Z9 Canada

From 1997-2000, PT INCO undertook the Fourth Line Expansion Project (FLEX) to increase annual production rates from 90-100 to 150 Mlbs Ni/year. In 2003, PT INCO nickel production underwent a major sustainable step jump that finally exceeded the targeted rate. This paper presents the changes that led to this achievement. The focus of mining changed. Improving Ni grade was important but only if electric furnace feed met target chemistry so that on-specification slag could be produced. The intensity of diamond core drilling for exploration was dramatically upgraded, mine planning strategy was totally re-vamped and an ore chemistry quality assurance team was formed. In the processing plant, measurement systems were upgraded for better control. Teamwork between sections vastly improved. The final step jump in production occurred when the fifth hydro-electric power generator was added to the furnace grid to allow the power-starved furnaces to cruise in operation at 70+ MW.

2:30 PM
Falcondo's Revisited: *Edwin de Jesús Deveaux*[1]; Francisco Geraldo Longo[1]; [1]Falconbridge Dominicana, Mining & Dvlp., Box 1343, Santo Domingo Dominican Republic

After 32 years of operations beneficiating lateritic nickel ore in a fully integrated pyrometallurgical complex, Falconbridge Dominicana (FALCONDO), situated at Bonao and La Vega Provinces, Dominican Republic, has become profitable with one of the lowest nickel grade content processing plants in the world, and literally without any by-product (granulated slag, will be utilized in the future as a post-processing subproduct). To December 2002, mineral reserves were 64.1 million tonnes containing laterite grading as low as 1.15 % Ni when full dilution is applied. At a cut-off grade of 1.20% Ni, the reserves will provide 15-20 years of production. The mineralized thickness over the seven active deposits averages 6 m. Development of very selective and unique mining practices, together with improvements in the process plant, are key to achieving the production goal (30 kt Ni/yr) and to meet the quality standards for the ferronickel produced.

3:00 PM
Falconbridge Dominicana Reduction Shaft Furnace Advanced Control Development: *Lionel Ryan*[1]; J. Frias[2]; P. Rodriguez[2]; A. Morrow[3]; M. Boland[3]; David Sandoz[4]; [1]Noranda Inc./Falconbridge Ltd., Metallurgl. Tech. Grp., Falconbridge Tech. Ctr., Falconbridge, Ontario P0M 1S0 Canada; [2]Noranda Inc./Falconbridge Ltd., Falconbridge Dominicana C. Por A., PO Box 1343, Santo Domingo Dominican Republic; [3]Invensys (Foxboro), 33 Commercial St. C42-2E, Foxboro, MA 02035 USA; [4]Perceptive Engineering, Brindley House, 4 Bridgewater Ct., Barsbank Ln., Lymm WA13 0ER Cheshire

In 1996, the Dominican Republic operation of Falconbridge (Falcondo) developed a 5-year Process Control Plan in conjunction with the corporate Process Control group located in Sudbury, Ontario, Canada. This plan was further refined into an analysis of the areas of the highest potential economic return for the company with the reduction shaft furnace operation being the most promising. Historically, the reduction plant has been the bottleneck of ferronickel production. Since 1996 there has been a history of control development with the shaft furnaces, with a current status of 11 of the 12 furnaces running advanced model-based multi-variable predictive control. The combination of Advanced Process Control (APC), improved control system performance, and improved process information management has resulted in an increased throughput of 6% per shaft furnace and a reduction in operating costs of 4.5 US cents/lb of Ni. This paper reviews some of the major milestones that have been achieved in this development.

3:30 PM Break

3:50 PM
A Historical Overview of the Cerro Matoso Operation 1980 to 2003: *Julian Robert Kift*[1]; [1]Cerro Matoso S. A., Tecnología, Calle 114 No. 9-01. Torre A, Piso 5. Oficina 509, Bogotá, DC Colombia

The Cerro Matoso S.A. mine and smelter, in the Cordoba lowlands of northwestern Colombia, consist of a dedicated laterite nickel mine, ore blending, drying and an RKEF process to produce high-quality ferronickel. At a project investment of $400M, first production of ferronickel was in September 1982, and in its inaugural year CMSA produced 6,150 tons of contained Ni from a mined ore grade of 3.2% Ni. Initial operational difficulties resulted in a number of furnace run-outs, which impacted ramp up to design ore throughput of 128 dry t/h. Since then CMSA has steadily incorporated process improvements and initiatives to take the single line operation to an ore throughput of 175 dry t/h. A second 175 dry t/h line was added in 2001 (investment of US$ 300M) to consolidate CMSA as one of the world's lowest cost Ni producers with an installed annual production capacity of over 50,000 tons Ni. The most recent addition is commissioning of Phase 1 of an ore-upgrading project to counteract the effect of declining ore grades.

4:20 PM
Recent Improvement of Reduction Rotary Kilns at Hyuga Smelting Co., Ltd.: K. Moriyama[1]; M. Yamagiwa[1]; A. Kaikake[1]; *H. Takano*[1]; [1]Hyuga Smelting Co., Ltd., Production Sect., Funaba-cho 5, Hyuga City, Miyazaki Pref 883-8585 Japan

Operation improvement efforts at Hyuga Smelting Co., Ltd. have focused on calcining capacity in the reduction rotary kilns ("the kilns"), whose limited feed rate capacity had capped production at around 18,000t-Ni/Y since 1995. To improve the kilns' feed rate capacity of

80 wet t/hr, attempts were made to increase the gas flow in the kilns by increasing the capacity of the off-gas fans. However, as the increased gas flow resulted in only limited improvement of the kilns' feed rate, a scoop feeder was installed on each kiln to add coal to the middle of each kiln's interior to gain a higher efficiency of thermal supply and conversion. With these gas flow and coal charge addition operations, the kilns' feed rate capacity increased up to 120 wet t/hr, with burner fuel efficiency increasing by approx.10% (approx. 5% in total with the additional coal included). As a result, production was raised to a level of 22,000t-Ni/Y.

Lead-Free Solders and Processing Issues Relevant to Microelectronic Packaging: Microstructural Characterization and Evolution

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Electronic Packaging and Interconnection Materials Committee

Program Organizers: Laura J. Turbini, University of Toronto, Center for Microelectronic Assembly & Packaging, Toronto, ON M5S 3E4 Canada; Srinivas Chada, Jabil Circuit, Inc., FAR Lab/ Advanced Manufacturing Technology, St. Petersburg, FL 33716 USA; Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; Kwang-Lung Lin, National Cheng Kung University, Department of Materials Science and Engineering, Tainan 70101 Taiwan; Michael R. Notis, Lehigh University, Department of Materials Science and Engineering, Bethlehem, PA 18015 USA; Jin Yu, Korea Advanced Institute of Science and Technology, Center for Electronic Packaging Materials, Department of Materials Science & Engineering, Daejeon 305-701 Korea

Wednesday PM Room: 219B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Michael R. Notis, Lehigh University, Dept. of Matls. Sci. & Engrg., Bethlehem, PA 18015 USA; Eric J. Cotts, Binghamton University, Physics & Matls. Sci., Binghamton, NY 13902-6000 USA

2:00 PM Invited
The Microstructure of Pb Free Solder Joints: *Eric J. Cotts*[1]; Lawrence P. Lehman[1]; Lubov Zavalij[1]; Robert Kinyanjui[1]; [1]Binghamton University, Physics & Matls. Sci., Sci. 2, Vestal Pkwy. E., PO Box 6000, Binghamton, NY 13902-6000 USA

The microstructural evolution of the widely used lead free solder, Sn95.5Ag3.5Cu1.0 and a variety of close compositions, Sn100-y-xAgxCuy, where $0 < x < 5$ and $0 < y < 3$ was examined after reflow, or after reflow on either Cu or Ni/Au metallizations. Heat treatment was conducted in a Differential Scanning Calorimeter (DSC) providing good control of the thermal profile and a quantitative measure of any thermal reactions during the process, including solidification. The effect of peak reflow temperature, cooling rate (0.01 to 3 oC/s), solder composition and metallization on the microstructure of the Pb free solders was examined. The microstructure was viewed with optical microscopy using cross polarizers and by scanning electron microscopy, and the distribution of species within the joint was measured. Grain size and orientations were studied, and related to possible mechanisms of solidification. Support from the National Science Foundation, DM0218129 is gratefully acknowledged.

2:25 PM
Microstructure-Based 2D and 3D Modeling of the Deformation of Pb-Free Solders: Rajen S. Sidhu[1]; Xin Deng[1]; *Nikhilesh Chawla*[1]; [1]Arizona State University, Dept. of Chem. & Matls. Engrg., Tempe, AZ 85287 USA

It is well known that microstructure, as controlled by variations in cooling rate or thermal aging, directly influences the mechanical behavior of Pb-free solders. In this talk, we report on two-dimensional (2D) and three-dimensional (3D) microstructure-based simulations of the deformation in Pb-free solders and solder joints. Representative 2D scanning electron micrographs were used as a basis for the 2D analysis. For the 3D analysis, a serial sectioning approach was used to obtain several 2D sections along the thickness of the solder, followed by 3D reconstruction of the microstructure. The reconstructed 3D volume was then used as an input for modeling using finite element analysis. Several microstructural features of Pb-free solders were modeled. These included: (a) the influence of Ag_3Sn morphology and distribution on the monotonic behavior of Sn-Ag solder and (b) the influence of Cu_6Sn_5 intermetallic thickness and morphology on the shear behavior of aged Sn-Ag solder/Cu joints. The effect of these microstructure variables on deformation and damage in Pb-free solders, and the implications for microstructure design of solders with improved mechanical properties will be discussed.

2:45 PM
Effects of Ga Additions on the Microstructure and Properties of Sn-Zn Solder Alloys: *Jenn-Ming Song*[1]; Nai-Shuo Liu[1]; Kwang-Lung Lin[1]; [1]National Cheng Kung University, Dept. of Matls. Sci. & Engrg., Tainan 701 Taiwan

The effects of Ga content on the microstructure, thermal behavior and tensile properties of Sn-Zn eutectic alloy were examined in this study. Results show that Ga was dissolved in both Sn and Zn phases. This gave rise to irregular eutectic structure with misaligned, less distributed massive Zn-rich phase, relatively low melting point, and solid solution strengthening effect. Due to the inhomogeneous dissolution feature of Ga in Sn matrix, Sn-Zn-Ga alloys exhibit a broad melting range and an alternate normal-irregular eutectic structure. Notably, the addition of Ga into the Sn-Zn alloy will improve the tensile strength without reducing the ductility when the Ga content ranges from 0.05 wt% to 1wt%.

3:05 PM
In-Situ Synchrotron X-Ray Diffraction During Melting and Solidification of a Lead-Free Solder Paste: Nick Hoo[2]; Gavin Jackson[3]; Mike Hendriksen[4]; Hua Lu[1]; Rajkumar Durairaj[5]; *Chris Bailey*[1]; Ndy Ekere[5]; Jonathon Wright[6]; [1]University of Greenwich, Computing & Math. Scis., Old Royal Naval College, Greenwich, London SE10 9LS UK; [2]Tin Technology, Unit 3, Curo Park, Frogmore St., St. Albans, Hertfordshire AL2 2NN UK; [3]Henkel Loctite Adhesives, Multicore Solders, Kelsey House, Wood Ln. End, Hemel Hempstead, Hertfordshire HP2 4R UK; [4]Celestica Limited, West Ave., Kidsgrove, Stoke on Trent, Staffordshire ST7 1TL UK; [5]University of Greenwich, Elect. Mfg. Engrg. Rsch. Grp., Sch. of Engrg., Chatham Maritime, Chatham, Kent ME4 4TB UK; [6]European Synchrotron Radiation Facility, Rue Jules Horowitz, Grenoble 38043 France

The intense flux of high-energy X-rays provided by synchrotron radiation sources allows transmission diffraction experiments to be performed. This can be used to gain non-destructive phase and strain data from the bulk of solid materials. The ability to focus the beam of x-rays into a narrow beam or spot of monochromatic x-rays allows 2d and even 3d mapping to be performed. As with all metals and alloys, the properties of a solder alloy are largely dependent on microstructure. The basis of the microstructure is evolved during a reflow operation from the heating and cooling cycle imposed upon the solder paste. This paper will show results from experiments undertaken at the European Synchrotron Radiation Facility. In these experiments, lead-free solder paste was placed onto a printed circuit board and then reflowed to form a solder joint. Time-resolved x-ray diffraction data were collected both from the bulk solder and interfacial regions during the melting and solidification processes. The paper will discuss the formation of microstructure for different pad finishes and cooling rates.

3:25 PM
The Effect of Electroplating Parameters on the Compositions and Morphologies of Sn-Ag Bumps: Jong-Yeon Kim[1]; Jin Yu[1]; *Jae-Ho Lee*[2]; [1]Korea Advanced Institute of Science and Technology, Ctr. for Elect. Pkgg. Matls., Dept. of Matls. Sci. & Engrg., 373-1 Guseong-dong, Yuseong-gu, Daejeon 305-701 Korea; [2]Hongik University, Dept. of Metallurgl. Engrg. & Matls. Sci., 72-1 Sangsu-dong, Mapo-gu, Seoul 121-791 Korea

Eutetic Sn-Ag solder bumps formed by electroplating method have been studied for the flip chip interconnection. The various conditions of electroplating bath were tested to obtain eutectic composition of Sn-Ag alloy. The effect of Ag ion concentration in solution on the morphologies and compositions of the solder bump was analyzed. Pulse plating method was introduced to acquire a level surface and fine microstructure of the solder bump. The surface morphologies and composition of the solder bump were changed with the duty cycle applied for the plating. Surface roughness of the solder bump was enhanced by applying surface active additives in the electroplating bath.

3:45 PM Break

3:55 PM Invited
The Microstructure of Sn in Near Eutectic Sn-Ag-Cu Alloy Solder and its Role in Thermomechanical Fatigue: *Donald W. Henderson*[1]; James J. Woods[1]; Timothy A. Gosselin[1]; Jay Bartelo[1]; Son Tran[1]; David E. King[1]; T. M. Korhonen[2]; M. A. Korhonen[2]; L. P.

Lehman[3]; E. J. Cotts[3]; Sung K. Kang[4]; Paul Lauro[4]; Ismail C. Noyan[4]; Da-Yaun Shih[4]; Charles Goldsmith[5]; Karl J. Puttlitz[5]; [1]IBM Corporation, 1701 North St., Endicott, NY 13760 USA; [2]Cornell University, Dept. of Matls. Sci. & Engrg., Rm. T328 Bard Hall, Ithaca, NY 14853 USA; [3]Binghamton University, Physics Dept., Matl. Sci. Prog., Binghamton, NY 13902-6000 USA; [4]IBM Corporation, T. J. Watson Rsch. Ctr., Yorktown Heights, NY 10598 USA; [5]IBM Corporation, Hopewell Junction, NY 12533 USA

During the solidification of solder joints comprised of near eutectic Sn-Ag-Cu alloys, the Sn phase grows rapidly with a dendritic growth morphology. Notwithstanding the complicated Sn growth topology, the Sn phase demonstrates single crystallographic orientations over large regions. Typical solder Ball Grid Array (BGA) joints, 900μ in diameter, are comprised of only 1 to 10 different Sn crystallographic domains. When such solder joints are submitted to cyclic thermomechanical strains, the solder joint fatigue process is characterized by the recrystallization of the Sn phase in the higher deformation regions with the production of a much smaller grain size. Grain boundary sliding is enabled in these recrystallized regions and leads to extensive grain boundary damage, resulting in fatigue crack initiation and growth along the recrystallized Sn grain boundaries.

4:20 PM
Quantitative Metallography of β-Sn Dendrites in Sn-3.8Ag-0.7Cu Solder Via Electron Backscatter Diffraction: Daniel Emelander[1]; Jason Jeannette[1]; Aaron LaLonde[1]; Carolyn Larson[1]; Ward Rietz[1]; *Douglas J. Swenson*[1]; Donald W. Henderson[2]; [1]Michigan Technological University, Dept. of Matls. Sci. & Engrg., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]IBM Microelectronics, Dept. U13, Bldg. 022-2, Rm. H007, 1701 North St., Endicott, NY 13761 USA

Near ternary eutectic Sn-Ag-Cu (SAC) alloys are leading candidates for a new microelectronics industry standard lead-free solder. In the solid state, these solders consist of β-Sn, Ag_3Sn and Cu_6Sn_5, where β-Sn comprises the vast majority of microconstituent volume fraction. The β-Sn phase adopts a dendritic morphology, and in a typical micrograph a solder joint appears to contain a large number of fine β-Sn dendrites. In this study, electron backscatter diffraction (EBSD) has been utilized to map the crystallographic orientations of the β-Sn dendrites in more than forty Sn-3.8 wt.%Ag-0.7wt.%Cu solder balls that were cooled at rates ranging from 0.35-3.0°C/s. It was found that at all cooling rates, there are in fact very few (5 ±2) crystallographically independent β-Sn dendrites per solder ball cross section. This suggests that a typical solder joint contains perhaps 10 β-Sn "grains", and as such may not be considered to be "polycrystalline" for purposes of mechanical properties modeling. Possible reasons for the existence of so few β-Sn dendrites are presented, and the utility of polarized light microscopy for identifying independent β-Sn dendrites is also discussed.

4:40 PM
Dissolutive Wetting in the Bi-Sn System: *Liang Yin*[1]; Timothy J. Singler[1]; Dorel Homentcovschi[1]; [1]SUNY-Binghamton, Dept. of Mech. Engrg., Binghamton, NY 13902-6000 USA

Development of a candidate Pb-free solder alloy requires a fundamental understanding of the alloy's wetting performance. When there is finite solubility of the solid substrate in the liquid alloy, significant dissolution of the substrate can occur. Isothermal sessile drop experiments of seven Bi-Sn alloys wetting Bi substrates were performed in a gaseous reducing atmosphere at 250°C. Meta-stable contact angles and dissolution depths were characterized as functions of alloy composition. Spreading kinetics and dynamic contact angles were also investigated. The coupling of dissolution and wetting are discussed. A partial analytical model is presented that predicts the liquid-solid interface evolution.

5:00 PM
Generation of Low Melting Point Solder and Thermal-Fuse Alloy Wires by Continuous Casting: *Zeinab A. Daya*[1]; Felix Hong[1]; Hiroshi Soda[1]; Zhiuri Wang[1]; Alexander McLean[1]; Genjiro Motoyasu[2]; [1]University of Toronto, Matls. Sci. & Engrg., 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]Chiba Institute of Technology, Mechanical Science, 2 Tsudanuma, Narashino, Chiba-ken 275-0016 Japan

Because of the brittle nature of bismuth and the strong tendency for segregation, solder and thermal-fuse alloys with high bismuth concentration are not easily produced in the form of small wires by ingot casting and extrusion methods. In this work, an innovative heated-mold continuous casting process, known as the Ohno Continuous Casting (OCC) process has been applied to generate Bi-Sn alloy wires of several compositions. The microstructure was examined and tensile tests performed for wires cast at various speeds. The deformation behavior was investigated by observing the surface structure of wires at various tensile deformation stages, providing insight into the structure-property relationship in low melting point alloys.

Magnesium Technology 2004: Fundamental Research

Sponsored by: Light Metals Division, LMD-Magnesium Committee
Program Organizer: Alan A. Luo, General Motors, Materials and Processes Laboratory, Warren, MI 48090-9055 USA

Wednesday PM — Room: 203B
March 17, 2004 — Location: Charlotte Convention Center

Session Chairs: John Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Zi-Kui Liu, Pennsylvania State University, University Park, PA 16082-5006 USA

2:00 PM
Diffusion Couple Study of the Mg-Al System: *Matt Benzio*[1]; Carl Brubaker[1]; Zi-Kui Liu[1]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Engrg., Steidle Bldg., Univ. Park, PA 16802 USA

The Mg-Al system was studied using the diffusion couple technique. The diffusion couples were characterized using light microscopy, electron probe microanalysis, and Vickers hardness. The growth constants, interdiffusion coefficients, activation energies and interdiffusion prefactors for the three intermediate compounds were estimated.

2:20 PM
Ignition Resistance of Various Magnesium Alloys: *Jean-Jacques Blandin*[1]; Eric Grosjean[2]; Michel Suery[1]; [1]INP Grenoble, Génie Physique et Mécanique des Matériaux (GPM2), ENSPG, BP 46, Saint-Martin d'Hères 38402 France; [2]EADS, Ctr. Commun de Recherches, 12 rue Pasteur, BP 76, (now at AIRBUS), Suresnes 92152 France

The fear of ignition is still today a factor which limits the use of magnesium alloys. The aim of this work is to investigate the ignition resistance of pure magnesium and of two magnesium alloys (AZ91 and WE43) in as-cast conditions. For pure magnesium, continuous heating tests show that ignition generally starts at a temperature lower than the melting point whereas for the AZ 91 alloy, the ignition temperature is between the solidus and the liquidus temperatures, suggesting that liquid phase is needed to initiate burning. For the WE 43 alloy, no ignition is detected up to temperature significantly higher than the liquidus and consequently, this alloy can be considered as an ignition-proof magnesium alloy. The various ignition resistances of the studied materials are discussed in terms of differences in composition and oxidation processes.

2:40 PM
Influence of Zinc on the Solubility of Zirconium in Magnesium and the Subsequent Grain Refinement by Zirconium: *Zoë C.G. Hildebrand*[1]; Ma Qian[1]; David H. StJohn[1]; Malcolm T. Frost[2]; [1]University of Queensland, Div. of Matls., Sch. of Engrg., St Lucia, Brisbane, Queensland 4072 Australia; [2]Australian Magnesium Corporation Limited, PO Box 1364, Milton, Brisbane, Queensland 4064 Australia

Most zirconium-containing commercial magnesium alloys also contain zinc, which varies from ~ 0.5% to ~ 6.5% depending upon the given alloy system. Early work has suggested that the presence of zinc in the range from 3% to 5% could significantly increase the solubility of zirconium in magnesium, therefore enhancing the grain refinement of the final alloy by zirconium. This study investigates the influence of zinc in the range from 0% to 8% on the solubility of zirconium in pure magnesium at three different temperatures 680, 730 and 780°C. The effect of zinc alone as well as the combined effect of zinc and zirconium on the grain refinement of pure magnesium at each of the three temperatures is also investigated. Recommendations are made for the levels of zirconium addition to zinc-containing magnesium alloys as a function of zinc content based on the results obtained from this investigation.

3:00 PM
Metal-Mold Heat Transfer and Solidification of Magnesium Alloys in Belt Casting Processes: *J. S. Kim*[1]; M. Isac[1]; R. I.L. Guthrie[1]; [1]McGill University, McGill Metals Procg. Ctr., Montreal, PQ Canada

A high speed strip casting simulator has been designed to simulate the casting of magnesium sheet alloys (AM50 and AZ91) on a single belt horizontal caster. Using Inverse Heat Transfer Analysis, temperature-time data from thermocouples inserted in the copper or steel bar

substrates were used to deduce instantaneous heat fluxes between the solidifying strip and the moving mold substrates. Maximum, or peak, heat fluxes were registered downstream of the impact region for metal delivery onto the moving chill mold; for a copper substrate moving at 0.7m/s, peak heat fluxes were recorded after 0.3 seconds of contact, whereas those for a yttria stabilized zirconia coated steel substrate were registered following 2.5 seconds of metal-mold contact. The microstructures of the thin strips (3-4mm thick, 1m long, and 40mm wide) were characterized in terms of SDAS (Secondary Dendrite Arm Spacings) as well as cooling rates. It was found that the major thermal resistance to heat flow from the strip to the substrate resided in an interfacial layer separating the strip from the substrate. This interfacial layer presumably corresponded to the entrainment of a thin gas film, which was manifested in the texture of the bottom surface which was seen to contain numerous small air pockets. Methods to resolve these issues, and to improve thermal contact, are discussed. Results to date suggest that the peaks in the maximum heat fluxes corresponded to maxima in the release of the alloy's latent heat of crystallization during their transformation from the liquid to solid crystalline state.

3:20 PM
Microstructure Controlled Magnesium Alloys Via Cyclically Repeated Plastic Working: *Katsuyoshi Kondoh*[1]; Tachai Luangvaranunt[2]; Ritsuko Tsuzuki[1]; Shigeharu Kamado[3]; [1]The University of Tokyo, RCAST, 4-6-1, Komaba, Meguro-ku, Tokyo 153-8904 Japan; [2]Chulalongkorn University, Phyathai Rd. Proatumwan, Bangkok 10330 Thailand; [3]Nagaoka University of Technology, 1603-1, Kamitokioka, Nagaoka, Niigata, Japan

Solid-state processing to fabricate high-strengthened magnesium alloys via cyclically repeated plastic working (RPW) and hot extrusion has been developed, in employing magnesium alloy coarse powder as input raw materials. The RPW process, consisting of the compaction and backward extrusion by inserting upper punches alternatively at room temperature, assists the refinement of the magnesium matrix grains by large plastic deformation. In increasing the number of cycles in the RPW, the refined grain size, d is remarkably reduced; for example, d=3-5im of the hot extruded magnesium alloy after RPW process with 200 cycles when using the AZ31 alloy raw powder with that of 120-150im. The RPWed hot extruded alloys show a good correspondence to Hall-Petch's relationship. The ultimate tensile strength of the AZ31 extruded alloy via RPW process with 100 cycles is 330-350MPa, and is much higher than that of the conventional hot extruded alloy without RPW.

3:40 PM Break

3:50 PM
Fatigue Behavior of Thixomolded® Magnesium AZ91D Using Ultrasonic Techniques: *A. R. Moore*[1]; C. J. Torbet[1]; A. Shyam[1]; J. W. Jones[1]; D. M. Walukas[2]; R. F. Decker[2]; [1]University of Michigan, Matls. Sci. & Engrg., Ann Arbor, MI 48109 USA; [2]Thixomat, Inc., Ann Arbor, MI 48108 USA

The fatigue behavior of the Thixomolded® magnesium alloy AZ91D has been examined via ultrasonic fatigue testing techniques at frequencies of approximately 20 kHz and for lifetimes as long as 10^9 cycles. An apparent endurance limit of approximately 65-70 MPa is observed. Comparison with the fatigue behavior of AZ91 produced by conventional die casting indicates that the Thixomolded® material has an endurance limit substantially higher than that of die cast material. The superior fatigue behavior of Thixomolded® AZ91D is attributed primarily to reduced porosity associated with the thixotropic processing technique. Fractographic analyses indicate that fatigue cracks leading to failure initiate at internal porosity in approximately 75% of tests and at the surface in the remainder of tests. Further fractographic studies illustrate that resulting fatigue lifetimes are greater for associated smaller fracture origination sites, in general. In addition, cumulative life distribution plots are employed in order to analyze apparent dual failure modes.

4:10 PM
Effect of Uniaxial Strain on the Surface Roughness of Pure Mg: *Mark R. Stoudt*[1]; Abraham Munitz[1]; S. W. Banovic[1]; Richard J. Fields[1]; [1]National Institute of Standards and Technology, Gaithersburg, MD 20899 USA

Pure Mg samples were deformed in uniaxial tension and the concomitant surface topographies were characterized with both scanning laser confocal microscopy (SCLM) and scanning electron microscopy (SEM) techniques. Electron channel patterning analyses were also performed to determine grain orientation. Initial results indicate two principal deformation mechanisms contributing to the development of surface morphology: (i) grain-to-grain interactions and subsequent out-of-plane rotations that produce surface features with low-frequency character (i.e., spanning several hundred microns), and (ii) slip band development within individual grains that produced surface with high frequency character (i.e., spanning one hundred to one thousand nanometers). In addition, slip band evolution was observed to be strongly dependent on grain boundary orientation (Schmid factor) in this materials. The results of this study will be presented and discussed with respect to current theories of plastic deformation and surface roughening.

4:30 PM
Fatigue of Die-Cast Magnesium Alloys: Carsten Potzies[1]; *Karl Ulrich Kainer*[1]; [1]GKSS-Research Center, Ctr. for Magnesium Tech., Max-Planck-Str. 1, Geesthacht 21502 Germany

The magnesium alloy AZ91, which is used in different automotive components, exhibits an excellent castability and is therefore usually fabricated by high pressure die casting. Unfortunately, it reveals only a poor creep resistance, while creep resistant magnesium alloys show a low castability. Alternative magnesium alloys have been developed, which show an acceptable creep resistance as well as a good castability. One of these alloys is a modified magnesium alloy based on AZ. Due to the high casting speeds in high pressure die casting, the melt flow is non-laminar and air can be entrapped causing porosity when the melt solidifies. Porosity highly influences the mechanical properties, reducing significantly the values of elongation, tensile strength and especially fatigue strength. Separately cast test bars of AZ91 and the modified AZ have been examined by rotation beam testing. S-N-curves were displayed to determine and to compare the fatigue lifes and fatigue limits.

Materials Analysis: Understanding the Columbia Disaster

Sponsored by: TMS
Program Organizers: Richard W. Russell, United Space Alliance, Materials & Processes Engineering, Kennedy Space Ctr., FL USA

Wednesday PM Room: Ballroom B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Richard W. Russell, United Space Alliance, Matls. & Processes Engrg., Kennedy Space Ctr., FL USA

2:00 PM Invited
The Materials and Processes Team Role in Columbia's Recovery, Recontruction and Analysis: *Rick Russell*[1]; [1]United Space Alliance, Kennedy Space Ctr., FL USA

An overview of the Columbia investigation, starting from recovery of debris items through the reconstruction and analysis at Kennedy Space Center will be presented. The Materials and Processes (M&P) team's role, including cleaning, field assessment, non-destructive inspection, sampling and analysis, and failure analysis will be discussed.

2:30 PM Invited
Fracture of Aluminum Structural Materials: *Robert Piascik*[1]; *Stephen Smith*[1]; [1]NASA Langley Research Center, Hampton, VA USA

During the Columbia reconstruction effort, a delamination fracture mode was observed on much of the orbiter aluminum structure debris. The delamination fracture is characterized by a fiber-like fracture mode similar to that observed in polymer matrix composite fracture. During the Columbia reconstruction effort, the unusual fracture mode was descriptively termed as a "broom straw looking failure". A detailed metallographic study was conducted to understand this first-of-a-kind fracture mode. The aim of this study was to understand this failure mode relative to the high-speed break-up of the orbiter during re-entry.

3:00 PM Invited
Failure Analysis of A286 Carrier Panel Fasteners: *Tom Collins*[1]; *Rick Russell*[2]; [1]Boeing, Huntington Beach, CA USA; [2]United Space Alliance, Kennedy Space Ctr., FL USA

During field assessment it was note that several A286 carrier panel fasteners had what appeared to be a brittle fracture mode. Results of failure analysis, including chemical, fractographic, metallographic and exemplar testing will be presented.

3:30 PM Break

3:50 PM Invited
Analytical Tools and Techniques: *Steve McDanels*[1]; [1]NASA, Kennedy Space Ctr., FL USA

Several methods of chemical analysis were performed in order to determine the most useful in understanding the slag's depositional characteristics. Techniques included: Scanning Electron Microscopy/ Energy Dispersive Spectroscopy (SEM/EDS), Electron Spectroscopy for Chemical Analysis (ESCA), X-Ray Diffraction (XRD), and Electron MicroProbe Analysis (EMPA). The relative merits and drawbacks of each technique will be explored.

4:20 PM Invited
Wing Leading Edge Debris Analysis: *Sandeep Shah*[1]; *Greg Jerman*[1]; [1]NASA Marshall Space Flight Center, Huntsville, AL USA

Having selected the RCC wing leading edge debris sample analysis techniques, guided by radiography, focused sampling with RCC intact was done for analysis to identify their content, layering, and if possible, hardware of origin. Analysis and Interpretation of this data across the wing leading edge was hoped to answer the high level questions of where did the breach occur? What was the sequence of melting? and What was the plasma flow direction? In sampling, two samples of each feature were taken and emphasis was placed on reproducibility and repeatability. The techniques used were metallography for cross-section, SEM for imaging and x-ray mapping, electron microprobe analysis for pinpoint accurate chemical analysis, x-ray diffraction to identify compounds. The data obtained was able to provide a picture of what happened, consistent with the leading failure scenario based on visual observations.

4:50 PM Invited
Failure Analysis Integration: The Materials and Processes Team's Role in the Overall Investigation and Conclusions: *Brian Mayeaux*[1]; *Julie Kramer-White*[1]; *Rick Russell*[2]; [1]NASA Johnson Space Center, Houston, TX USA; [2]United Space Alliance, Kennedy Space Ctr., FL USA

The Materials and Processes team's observations and results are compared and integrated with the findings and conclusions of the reconstruction team, and melded into the overall investigation.

Materials by Design: Atoms to Applications: Design for Mechanical Functionality II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA; Krishnan K. Sankaran, The Boeing Company, Phantom Works, St. Louis, MO 63166-0516 USA

Wednesday PM Room: 210B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: M. K. Sunkara, University of Louisville, Louisville, KY 40292-0001 USA

2:00 PM
Characteristic Dimensions in the Fracture and Fatigue of Biomaterials: *R. O. Ritchie*[1]; [1]Lawrence Berkeley National Laboratory, Matls. Scis. Div., Berkeley, CA 94720 USA

This presentation focuses on the fatigue and fracture toughness properties of human bone and dentin (the major constituent in teeth), in simulated physiological environments, and on how these properties depend upon the hierarchical nature of the nano/microstructure of these mineralized tissues with characteristic microstructural length scales which extend from nanometers to hundreds of micrometers. Although there is substantial clinical interest in their fracture resistance, little mechanistic information is available on how these hard mineralized tissues derive their toughness and how they are specifically affected by cyclic fatigue. Although several toughening mechanisms have been proposed, rarely has their contribution been characterized quantitatively or their origin determined in terms of the salient features of the microstructure. In the present talk, in vitro experiments are described that establish that fracture in dentin and bone is locally strain-controlled. Further, it is shown that toughening is a marked function of orientation and is developed through a variety of extrinsic mechanisms, including crack bridging (from collagen fibrils and uncracked ligaments), crack deflection and diffuse microcracking. These mechanisms provide the means to describe the macroscopic mechanical properties of these biological materials in terms of the nano/microscale features of their structure

2:30 PM
Cellular Control of Toughening Mechanisms in Bone: Bioengineering for Materials Design: *Deepak Vashishth*[1]; [1]Rensselaer Polytechnic Institute, Dept. of Biomed. Engrg., 110 8th St., Jonsson Engrg. Ctr., Rm. 7046, Troy, NY 12180 USA

Bone is a brittle microcracking composite that forms microcracks at several levels of organization during day to day loading. Using a combination of engineering and biological techniques, experimental evidence is presented here to demonstrate how bone utilizes its material and biological characteristics to produce and remove microcracks in order to optimize toughness while remaining physiologically viable. Specifically: (a) Fracture mechanics-based crack growth resistance tests and scanning electron microscopy are utilized to identify the mechanism by which microcrack formation during crack propagation contributes to bone's resistance against fracture or fracture toughness. (b) Histological examination of microdamage and cellular network in aging human bone and statistical techniques are utilized to identify the mechanism by which damaged bone is targeted and eventually replaced by new bone.

3:00 PM
Damage Evolution in Hot Deformation: Nano-Micro- Mesoscale Growth: *Amit Ghosh*[1]; [1]University of Michigan, Matls. Sci. & Engrg., 2300 Hayward St., Ann Arbor, MI 48109-2136 USA

Engineering alloys exhibit formation of internal voids during large plastic deformation at elevated temperature, a matter of concern in metal forming processes. Typically such void formation occurs at and near grain boundaries, due to grain boundary sliding effects, albeit small at times, which provide an accommodating mechanism for deformation. Thermomechanical processing and casting often leave fine scale damage in materials, particularly at hard second phase particles and grain boundaries. These damages can grow during subsequent deformation primarily by a strain-controlled process. While it is often assumed that recrystallization and homogenization treatments can heal damage and rid material of preexisting damage, research has shown that this notion is only approximately true. "Apparently healed" voids may only represent a weak interface, or nanometer scale voids. As these nanovoids provide free surfaces, they experience deviatoric stresses during deformation and grow by dislocation accumulation at their tips. Gradual, statistical opening of the nanovoids give rise to a "continuous nucleation" phenomenon. In this talk, growth of such voids and the role of healing processes on subsequent damage evolution will be discussed.

3:30 PM Break

3:45 PM
Modeling and Experiments of the Deformation of Ti-Based Alloys at Two Different Length Scales: *Clyde L. Briant*[1]; [1]Brown University, Engrg., Providence, RI 02912 USA

The two features that most often control the properties of materials are the chemistry of the alloy and the microstructural constituents. Both must be considered carefully if one is going to design the overall properties of a material. The primary example that will be used for this talk are titanium-based materials. First we will consider only monolithic materials, both commercial purity titanium and Ti-6Al-4V, and show the importance of small concentrations of interstitial impurities on the mechanical properties. These changes must be a result of changes in chemical bonding within the alloy and must be modeled with these techniques. We will also present a study of Ti-6Al-4V/TiC composites. It will be shown that the presence of the TiC changes the deformation mode that occurs under some conditions, but once again the strength of the material is controlled by the matrix. Examples of methods to model these microstructures will also be presented and the more general utility of these microstructure-focused methods of modeling will be demonstrated.

4:15 PM
Accomplishing Structural Alloy Design Goals Within the Limitations of Bulk Processes: *David M. Bowden*[1]; Thomas J. Watson[2]; [1]The Boeing Company, MC S245-1003, PO Box 516, St. Louis, MO 63166 USA; [2]Pratt & Whitney, E. Hartford, CT USA

Control of microstructure evolution at the atomic or short-range level to achieve breakthrough material properties is a topic of considerable interest. Computational materials science is emerging as a critical tool to support this level of materials design. And, a variety of methods are being investigated to produce these nanostructured metals, and the advantages of achieving fine microstructures have been clearly demonstrated. However, in order to transition this technology into real products, fine microstructures must be achievable in bulk material forms in an efficient and affordable manner. In this paper, we will discuss the linkage between the scale of design and the scale of

production critical to developing nanostructured metals for real applications. This analysis will include a discussion of particular structural applications and the requirements for those applications, both in terms of mechanical properties and cost, and examine the variety of processing approaches available to meet those requirements.

4:45 PM
Materials by Design: Naval Aircraft: *William E. Frazier*[1]; [1]Naval Air Systems Command, 48066 Shaw Rd., Patuxent River, MD 20657 USA

Navy aircraft and weapon systems operate in the world's harshest environments. Navy assets are subjected to extremes in weather conditions, loads (6 times that of land based aircraft), and electromagnetic environments (240v/m). Consequently, virtually every materials technology used on navy aircraft is engineered and tailored for its intended application. This paper discusses the navy's current approach in rapidly developing, qualifying, and transitioning new materials technologies. Aerospace materials programs on engineered materials, accelerated insertion, and collaborative, web-based knowledge management are discussed.

5:15 PM Concluding Remarks K. Rajan and K. K. Sankaran

Materials Education to Revitalize the Workforce: Session II

Sponsored by: TMS, Public & Governmental Affairs Committee, TMS-Education Committee
Program Organizers: Reza Abbaschian, University of Florida, College of Engineering, Gainesville, FL 32611-6400 USA; Iver E. Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA

Wednesday PM Room: 217D
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

2:00 PM Invited
Meeting the Needs of a Changing Student Population: *Rosemary Haggett*[1]; [1]National Science Foundation, Washington, DC USA

What can we do to ensure that we have a workforce with the knowledge of science, mathematics and technology needed in our contemporary workplace and community members capable of exercising responsible citizenship in an increasingly technological society? Although our population continues to grow, partly through immigration and partly through birth, the segments of the population that are expanding are less likely to complete a bachelor's degree or an advanced degree and are less likely to be offered educational opportunities by their employers. Furthermore, patterns of college attendance are changing. Although more and more students are enrolled in postsecondary programs, the educational environment is increasing in complexity. Young people and adults have many options for pursuing a degree or for enhancing their employability and opportunity for advancement through credentialing models offered by both traditional educational institutions and new for-profit providers. How can we effectively meet the needs of an increasingly diverse student population?

2:30 PM
Materials Science and Engineering Education – Its Continuing Evolution: *Carl C. Koch*[1]; J. Michael Rigsbee[1]; [1]North Carolina State University, Dept. of Matls. Sci. Engrg., Raleigh, NC 27695 USA

This talk will give a brief history of the coming of materials science and engineering and its present evolution. The program area of materials science developed in the late 1950's as interdisciplinary research in large industrial laboratories such as G.E. and Bell Laboratories. Under the leadership of Morris Fine, the first graduate and undergraduate programs were instituted at Northwestern University in the 1958-1959 period. Materials science and engineering had its roots in physical metallurgy and parts of solid state physics and chemistry. In turn physical metallurgy had grown out of mining and mineral processing programs. The various factors which have influenced its evolution will be considered. These include technological, societal, and accreditation constraints. The present and future trends in the direction of "soft" materials science and engineering will be discussed.

3:00 PM
The MSE PITCH Program for Graduate Recruiting: *Kevin S. Jones*[1]; Martha McDonald[1]; [1]University of Florida, Dept. of Matls. Sci. & Engrg., Gainesville, FL 32611 USA

The recruiting of domestic graduate students into Materials Science and Engineering is an ongoing problem nationwide. The Department of Materials Science and Engineering at the University of Florida has developed a new approach to recruiting domestic graduate students. This approach is called the PITCH (Publicize, Identify, Time, Convince, and Host) program. This process is very similar to the methods used to recruit highly sought after student athletes. Using this approach UF-MSE managed the successful recruitment of 75 graduate students over the past two years. The PITCH approach has resulted in over 85% of our last two recruiting classes entering to pursue a PhD. This same group averaged 70% US students and over 20% of the recruits were minority students. In addition the percentage of female students has increase to over 40% in the latest class. This talk will share the critical elements of our recruiting efforts that can assist other engineering schools with improving the quality and diversity of their programs.

3:30 PM Break

3:50 PM
Towards a New Undergraduate Curriculum in Materials Science and Engineering: Caroline A. Ross[1]; Samuel M. Allen[1]; Subra Suresh[1]; *Donald R. Sadoway*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Engrg., 77 Mass. Ave., Rm. 8-203, Cambridge, MA 02139-4307 USA

At MIT, wholesale revision of the undergraduate degree program in MSE is underway with the intention of devising a course of study whose aim is to educate specialists in the development and use of materials in technology. The new curriculum comprises core technical knowledge, professional development, and a capstone activity. Pedagogical considerations include integration of subject matter between subjects, reinforcement of theory through applications, and presentation of material on a need-to-know basis, i.e., in time blocks of several weeks as opposed to full semesters. In the junior and senior years students can tailor their course of study by choosing from a large number of restricted electives, each running approximately four weeks, building upon the core and moving towards the frontiers of the field. Professional development is to be embedded in the curriculum in accordance with ABET specifications. Launch of the new program is slated for fall 2003.

4:20 PM
Lifelong Learning: The Role of Professional Societies: *Michael J. Kenney*[1]; [1]ASM International

Professional societies must support and supplement the educational efforts of traditional educational institutions. Pre-college education has become rote in many aspects and standards are mandating a very homogeneous curriculum. College educational programs have diversified to such an extent that graduates lack some of the fundamental knowledge needed to succeed. It is incumbent on professional societies to reach out to both pre-college and college students and to provide development opportunities to working professionals. Without efforts in all these areas, the need for professional societies may disappear completely.

Materials Processing Fundamentals: Powders, Composites, Coatings and Measurements

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control 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: 212B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

2:00 PM
Dense Sintering of Long and Stepped Sample Through Traveling Zone Heating by Means of Electric Power Direct Supplying: *Shuji Tada*[1]; Zheng Ming Sun[1]; Hitoshi Hashimoto[1]; Toshihiko Abe[1]; [1]National Institute of Advanced Industrial Science and Technol-

ogy, Inst. for Structural & Engrg. Matls., 4-2-1 Nigatake, Miyaginoku, Sendai, Miyagi 983-8551 Japan

A traveling zone heating technique was examined in order to produce long rods or stepped components with high density by means of pressurized pulse discharge sintering. Local heating was enabled by electric power supplied perpendicular to the loading axis. One-direction continuous sintering process was successfully achieved assisted by the unique electrodes which were able to slide along the side wall of the cylinder in the direction of the loading axis while remaining in continuous contact with it. Four successive local heatings over a range 30 mm wide while moving the heating zone distances of 20 mm led to the production of a 55 mm long aluminum rod with a relative density of 99.7%. Also, a 30 mm long round bar with two steps was successfully sintered through this method owing to reducing the stress within thinner part while a thick portion was heated.

2:30 PM

A Study of Particle Size and Velocities Distributions in Centrifugal Atomisation Using Mathematical Regression Techniques: *P. Tsakiropoulos*[1]; Yawei Wang[1]; [1]University of Surrey, Sch. of Engrg., Mech. & Aeros. Engrg., Metall. Rsch. Grp., Guildford, Surrey GU2 7XH UK

A study of the centrifugal atomisation of liquids using a rotating disk was presented at the 2003 annual meeting for the case of the liquid impacting the centre of the rotating disk during atomisation. This year our paper would discuss the atomisation of liquids that impact the disk at off centre positions. The paper would present results on the distribution of particle sizes and velocities in centrifugal atomisation in a purpose-built atomisation chamber. The data was collected using Laser Phase Doppler Anemometry. Mathematical regression analyses were developed for the distributions of particle sizes and velocities for the different operating conditions using the non-dimensional technique of similarity theory. The regressed functions were applied to predict the effect of disk rotating speed, flow rate and disk diameter on the spatial distributions of particle size and velocities for alloy melts. The results of experiments and regression analyses will be presented and discussed.

2:50 PM

Study on the Preparation of Foam Aluminum by Powder Metallurgy Method: *L. Wei*[1]; G. C. Yao[1]; X. M. Zhang; [1]Northeastern University, Sch. of Matl. & Metall., Liaoning, Shenyang 110004 China

The experiments about process of preparation foam aluminum by powder metallurgy method was carried out in laboratory. The effect of the pressing parameters for making aluminium powder flans, foaming temperature, adding amount of foaming agent and foaming time on the density and porosity and pore morphology of foam aluminum were studied. The aluminium flans were protected in molten salt for isolating from the air. It was found that the hydrogen pressure in the bubble always enlarged along with the increase of time in first stage and make the bubble growing, when the hydrogen pressure was up to a determinate value, the bubble broken and then the hydrogen pressure reduced and expanding drive lack and led to shrinkage. On condition of 300 MPa making flans pressure, 675-680° foaming temperature, foaming time 7 min, adding 1.0%-1.5%TiH2, the foam aluminum specimens with even hole and high porosity were gained. The formation mechanism of forming air bubble was discussed in this work.

3:10 PM

Study on Grain Composition of the Biscuit for Preparing Inert Anode Based on NiFe2O4 Spinel: *Y. H. Liu*[1]; G. C. Yao[1]; J. H. Xi[1]; J. H. Zhang[1]; [1]Northeastern University, Matl. & Metall., Liaoning, Shenyang 110004 China

In order to obtain the NiFe2O4 spinel based inert anode samples possessing higher volume density and lower porosity, the grain graduation of NiFe2O4 spinel biscuit was studied on the base of the ball arrangement theory by means of vibration entity experiments. Four different main grains were decided according to the different size of products. The packed density was measured to define the optimal grain graduation. The mass ratio and the diameter ratio of main grains to filling grains was find out above all. On the base of it, different quantity of fine powder was added to the sample for the sake of obtaining optimal grain composition. The results validated that the actual stack form of the main grain fit the ball arrangement theory very well, and tend to be in the form of the body centered cubic or hexagonal close-packed, moreover the packed density was the greatest when the ratio of the coarse grains is 42%, medium grains was 18% and fine powder was 40%.

3:30 PM Break

4:00 PM

Experimental and Computational Investigations of the Bonding Layer in the CVD Coated WC+Co Cutting Tools: *Zhi-Jie Liu*[1]; Charles McNerny[2]; Pankaj Mehrotra[2]; Aharon Inspektor[2]; *Zi-Kui Liu*[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 107 Steidle Bldg., Univ. Park, PA 16802 USA; [2]Kennametal, Inc., Corp. Tech., Latrobe, PA 15650 USA

The effects of processing parameters on the bonding layer structure between tungsten carbide substrate and Al2O3 coating were investigated experimentally and computationally. Bonding layers, considerably thicker than the layers in commercial products, were deposited under industrial production conditions. Different from the belief in the literature that the bonding layer is a cubic Ti(C,N,O) phase, titanium oxides were found. Energy dispersive spectroscopy and X-ray diffraction measurements were used to determine the compositions and structures of the bonding layer and the Al2O3 layer. It was found that the a-Al2O3 phase formed on the oxide bonding layer, and the k-Al2O3 phase formed without the bonding layer. Thermodynamic calculations were conducted to explore the effects of processing parameters on the formation of various oxides. It was concluded that the decomposition efficiency of the gas precursors plays a critical role in the formation of oxides.

4:30 PM

The Interface Reaction in Preparation of Aluminum Composite Strengthened by Cenosphere Fly Ash: *L. L. Wu*[1]; G. C. Yao[1]; Y. H. Liu[1]; [1]Northeastern University, Sch. of Matl. & Metall., Liaoning, Shenyang 110004 China

The major constituents of cenosphere fly ash are various oxides such as Al2O3, SiO2, Fe2O3 and their qualities fraction sums surpass 85%. XRD analysis indicates that cenosphere fly ash present in complex glass and ceramic forms. This research adopts liquid stir casting to prepare aluminium composite strengthened by cenosphere fly ash. Cenosphere fly ash preheated at 500° for half an hour was mixed with aluminium at 780° and the composite was prepared. The solidification should be as rapid as possible to prevent floatation of fly ash particles to the top part of the castings. SEM and EDX analysis were carried on to observe the composite. SEM micrograph shows that there was no fly ash particle fall off from section plane of the specimen in the abrading, and the ash particles affixed fast with the aluminium body, and they dispersed uniformity. EDX results show that there were atomic Al, Mg, Fe, Si, C, in the interface of the fly ash particle and aluminium body, it demonstrated that Fe2O3 and SiO2 react with aluminum and produce Fe and Si, MgO react with Al2O3 forming MgO·Al2O3 spinel. These reactions were analysed by thermodynamics calculation also.

4:50 PM

Evaluation of the Initial Residual Stress Distribution and Multiple Pass Grinding Techniques on Final Residual Stress Distribution in D2 Steel: *Olga Petrovna Karabelchtchikova*[1]; Iris V. Rivero[1]; [1]Texas Tech University, Dept. of Industrial Engrg., Box 43061, Lubbock, TX 79409-3061 USA

The purpose of this investigation is to correlate the residual stress behavior of D2 steel when subjected to two- and four-passes resulting in equivalent depth during gentle and conventional grinding operations. Single-, double- and triple-tempering were performed on D2 steel samples followed by x-ray diffraction and microstructural material characterization. Hardness, cold work and surface residual stress measurements were collected at this stage of the study. Subsequently the samples were subjected to gentle and conventional grinding conditions to re-evaluate material's properties and surface integrity. Each grinding pass was assessed for its influence on every subsequent pass in terms of residual stresses. Due to non-linearity of the residual stress superposition, the distribution pattern between the samples undergone 2- and 4-pass grinding was shown to be considerably different. Overall, the study identified the optimal combination of treatments, used in D2 thread-rolling die manufacturing, for prolong service life and satisfactory performance.

5:10 PM

Characterization of Thermal Lags and Resistances in a Heat-Flux DSC: *Gregory E. Osborne*[1]; Jay I. Frankel[1]; Adrian S. Sabau[2]; Wallace D. Porter[2]; [1]University of Tennessee, Mech., Aeros. & Biomed. Engrg. Dept., 414 Dougherty Engrg. Bldg., Knoxville, TN 37996-2210 USA; [2]Oak Ridge National Laboratory, 1 Bethel Valley Rd., Oak Ridge, TN 37831-6083 USA

Differential Scanning Calorimetry (DSC) is often used to characterize thermophysical properties associated with phase transformation of metals and alloys. In such devices, however, the practical design of the instrument does not allow for direct temperature mea-

surements of the sample material. As a result, the contact conductances and radiative interactions among system components yield thermal lags between the collected temperature data and sample temperature. Therefore, a direct association of the recorded thermocouple readings to the sample site may produce erroneous results. In order to account for these heat transfer mechanisms, a new parameter estimation method has been developed utilizing a lumped heat transfer model for the key DSC components. Preliminary results using a benchmark numerical problem have been obtained which show accurate recovery of the system parameters and demonstrate the robustness of the new method. Based on these encouraging numerical results, the method is presently being applied to real experimental situations.

Metals for the Future: Functional Materials

Sponsored by: TMS,
Program Organizers: Manfred Wuttig, University of Maryland, Department of Materials & Nuclear Engineering, College Park, MD 20742-2115 USA; Sreeramamurthy Ankem, University of Maryland, Department of Material & Nuclear Engineering, College Park, MD 20742-2115 USA

Wednesday PM Room: 215
March 17, 2004 Location: Charlotte Convention Center

Session Chair: M. Wuttig, University of Maryland, Dept. of Matls. & Nuclear Engrg., College Park, MD 20742-2115 USA

2:00 PM Opening Remarks by M. Wuttig

2:10 PM Invited
Magnetic Domain Microstructures in Ferromagnetic Shape Memory Alloys: Sai Prasanth Venkateswaran[1]; *Marc De Graef*[1]; [1]Carnegie Mellon University, Matls. Sci. & Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

Ferromagnetic shape memory alloys, such as Ni_2MnGa and Co_2NiGa, have received considerable attention in recent years. These alloys undergo both a paramagnetic to ferromagnetic transition and a martensitic phase transition to a tetragonal structure with a c/a ratio of around 0.96. There is a complex interplay between structural domains (twins) and magnetic domains, and we will present quantitative Lorentz transmission electron microscopy observations of the domain structure in both austenitic and martensitic states. The Lorentz images are analyzed using the Transport-of-Intensity formalism, which allows us to extract quantitative information about the domain structure at the nanometer length scale. We will also present evidence for the existence of a magnetically modulated pre-transformation state, known as "magnetic tweed".

2:40 PM Invited
Self-Assembled Near-Zero-Thickness Nanolayers for Nanodevice Metallization: Interfacial Adhesion and Chemical Isolation: *G. Ramanath*[1]; P. G. Ganesan[1]; M. J. Frederick[1]; [1]Rensselaer Polytechnic Institute, Dept. of Matls. Sci. & Engrg., Troy, NY 12180 USA

Tailoring near-zero-thickness layers that enhance interfacial adhesion, but inhibit interdiffusion and phase formation, is a critical challenge for future metallization for integrated circuits and MEMS. For instance, <5-nm-thick barriers that can conformally coat sub-100-nm features with aspect-ratios >5:1 are needed to fully realize the potential of Cu metallization. Meeting such exacting requirements necessitates new materials and scalable processing methods based on self-assembly. This talk will describe a completely new approach of using ~0.7 to 5-nm-thick self-assembled molecular layers (SAMs) to inhibit interfacial diffusion and enhance interfacial adhesion. I will first describe the rationale for using SAMs as interface modifiers, and demonstrate that SAMs inhibit Cu diffusion and effect as much as ~5-fold increase in device lifetimes and decrease leakage currents by ~6 orders of magnitude. It will be shown that the SAM molecular length and terminal functional groups are key factors that determine their efficacy as diffusion barriers. Interfacial adhesion of Cu/SAM/silica and Cu/Silica structures measured by a 4-point-bending technique show that interfacial adhesion can be enhanced by more than a factor of 3 for SAMs with terminal groups that bond strongly with Cu (e.g., -SH group) on one end and the dielectric surface (e.g., via Si-O-Si bonds) on another. Electron spectroscopy measurements of fracture surfaces show that delamination occurs at the SAM/Silica interface, leaving the -SH or –COOH groups on the metal side of the interface. The interface-specific interactions immobilize Cu, and enable very promising barrier properties (factor of 3-4 higher lifetimes) even for sub-nanolayers. Based on the above, we will present a model to explain important factors that influence interfacial diffusion and adhesion in Cu/SAM/dielectric structures. If time permits, the strategy of forming self-organized interfacial nanolayers from supersaturated alloy films by solute segregation and phase formation will also be discussed.

3:10 PM Invited
Novel Conductive Oxide Coatings on Metallic Inteconnect for Intermediate-Temperature SOFCs: *Jiahong Zhu*[1]; [1]Tennessee Technological University, Dept. of Mech. Engrg., 115 W. 10th St., Box 5014, Cookeville, TN 38505 USA

This presentation gives a progress overview of the NSF Career Award "Novel Conductive Oxide Coatings on Metallic Inteconnect for Intermediate-Temperature SOFC Application". With the current trends in reducing the SOFC operation temperatures to the range of 500-800°C, ferritic steels are promoted as the candidate materials for the intermediate-temperature SOFC interconnect due to their low cost and ease of manufacture. However, under long-term stack operation, the increase of contact resistance due to the formation of surface oxide layer(s) and Cr migration to other cell components from the interconnects pose serious issues for these otherwise promising materials. Novel conductive spinel phases are investigated in this project as potential coatings to mitigate the current limitations of ferritic steels. Electrical conduction mechanism in substituted spinel phases are being assessed through systematic study of defect structure and electrical conductivity in these oxides. Sol-gel process is used to form the spinel coatings on commercial ferritic steels, while alloy design is explored to develop new-generation ferritic alloys capable of forming the desired spinel layer upon thermal exposure. The integrated educational activities, such as outreach to local high school teachers/students, enhancement of the undergraduate curriculum via familiarizing the students with both bulk material and coating processing, are also discussed.

3:40 PM Invited
CAREER: Fundamental Micromechanics and Materials Dynamics of Thermal Barrier Coating Systems Containing Multiple Layers: *Mark L. Weaver*[1]; [1]University of Alabama, Metallurgl. & Matls. Engrg., Box 870202, Tuscaloosa, AL 35487-0202 USA

This research focuses on understanding the fundamental concepts controlling the mechanical properties of thermal barrier coatings (TBC) as a function of thermal exposure, both time and temperature. This program goals are to: (1) Characterize the properties of the macro- and micro-constituents of the TBC system as a function of thermal exposure using model TBC systems; (2) Monitor and analyze the displacements of the TBC constituents during thermal cycling and isothermal oxidation coupled with mechanical strain; and (3) Develop models for TBC durability based on microstructural and mechanical evaluations of degradation mechanisms and processes. The goals of the educational plan are: (1) Enhance the undergraduate curriculum by providing students with "hands on" training in the study of commercially viable thick films and coating materials; (2) Encourage undergraduate students to pursue graduate studies in Metallurgical and Materials Engineering; and (3) Increase diversity by attracting underrepresented minority students to undergraduate and graduate study.

4:10 PM Break

4:25 PM Invited
Effects of Micro- and Nano-Scale Critical Dimensions on Mechanical Behavior of Model Metals and Alloys: *Richard P. Vinci*[1]; [1]Lehigh University, Dept. of Matls. Sci. & Engrg., 5 E. Packer Ave., Bethlehem, PA 18015 USA

It is now widely known that the mechanical behavior of metals and alloys can be strongly affected by the reduction of certain critical dimensions (e.g., grain size and film thickness) to the micro- or nano-scale. Furthermore, the techniques used to fabricate materials with these dimensions often introduce characteristics that are not typical of bulk materials. Understanding the ramifications of these phenomena is critical for proper material design and selection for future micro/nanoelectronic and micro/nanomechanical devices. In this presentation, several examples of these effects in model metals and their alloys will be explored and important open questions will be identified.

4:55 PM Panel Discussion with L. Christodoulou, N. Spaldin, M. Wuttig and Y. Chung

Multiphase Phenomena in Materials Processing: Session II

Sponsored by: Extraction & Processing Division, Light Metals Division, Materials Processing and Manufacturing Division, EPD-Process Fundamentals Committee, MPMD/EPD-Process Modeling Analysis & Control Committee, MPMD-Solidification Committee
Program Organizers: Ben Q. Li, Washington State University, School of Mechanical and Materials Engineering, Pullman, WA 99164-2920 USA; Stavros A. Argyropoulos, University of Toronto, Department of Materials Science and Engineering, Toronto, Ontario M5S 3E4 Canada; Christoph Beckermann, University of Iowa, Department of Mechanical Engineering, Iowa City, IA 52242 USA; Bob Dax, Concurrent Technologies Corporation, Pittsburgh, PA 15219 USA; Hani Henein, University of Alberta, Edmonton, AB T6G 2G6 Canada; Adrian S. Sabau, Oak Ridge National Laboratory, MS-602, Oak Ridge, TN 37831-6083 USA; Brian G. Thomas, University of Illinois, Department of Mechanical and Industrial Engineering, Urbana, IL 61801 USA; Srinath Viswanathan, Sandia National Laboratories, Albuquerque, NM 87185-1134 USA

Wednesday PM Room: 218B
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Christoph Beckermann, University of Iowa, Dept. of Mech. Engrg., Iowa City, IA 52242 USA; Bob Dax, Concurrent Technologies Corp, Pittsburgh, PA 15219 USA

2:00 PM Invited
The Multi-Fluid Model and its Application to Simulate Multiphase Flows in Materials Processing Systems: *Harald Laux*[1]; [1]SINTEF Materials Technology, Flow Tech., Alfred Getz vei 2, Trondheim 7465 Norway

Lecture and paper will focus on the derivation of the multi-fluid model, its features and the importance of multi-fluid modeling for multiphase flows in materials processing systems. Examples of CFD simulations from a variety of materials processing systems will be given including bubbly flows, liquid-liquid-gas flows, and granular flows. It will also be shown that multi-fluid models can be used to study solidification problems with complex microstructures. All examples are taken from the work at SINTEF Materials Technology, and are chosen such as to illustrate the salient features of multi-fluid modeling, its limitations, and its present and future usefulness in improving materials processing systems with multiphase flows.

2:25 PM Invited
Two-Phase Volume Averaging: Simulation Examples on Phase Separation During Phase Transition with Convection: *Andreas Ludwig*[1]; Menghuai Wu[1]; [1]University of Leoben, Dept. of Metall., Franz-Josef-Str. 18, Leoben 8700 Austria

Phase separation is frequently occurring during solidification accompanied by phenomena like melt convection, sedimentation or with two liquids Marangoni driven motion. In order to describe these phase separation phenomena a two-phase volume averaging model was designed specially for globular equiaxed solidification of binary alloys and decomposition and solidification of hyper-monotectic alloys. The model considers nucleation and growth of equiaxed grains or second phase droplets, motion and sedimentation of grains or droplet, feeding flow and solute transport by diffusion and convection. It allows the prediction of macrosegregations and the distributions of grain size or droplet size. Evaluations were made by comparing the predictions gained with simulation with experimental results. For example it is shown that the numerically predicted grain size distribution in a plate casting (Al-4wt%Cu) agrees reasonably well with the experimental analyses.

2:50 PM
Phase-Field Modeling of Solidification with Flow Due to Density Change: Ying Sun[1]; *Christoph Beckermann*[1]; [1]University of Iowa, Dept. Mech. & Industrial Engrg., 2412 SC, Iowa City, IA 52242 USA

A phase-field model is developed that accounts for the effect of density change during solidification as well as for flow. A diffuse interface description of the conservation equations is derived based upon the multiphase averaging method. The phase-field evolution equation is obtained by averaging a sharp-interface condition for the interface temperature that takes into account the effects of density difference between the solid and liquid, interface kinetics, curvature, pressure and stresses. Two different cases are examined: (1) the liquid and solid are both treated as viscous fluids with a single velocity inside the diffuse interface region, and (2) the liquid is a viscous fluid, but the solid is rigid, such that the phase velocities are different. The model is first tested for simple planar and spherical solidification fronts in the presence of a density difference. Then, results for 2-D dendritic growth are presented.

3:10 PM
Development of Microstructure During Sputtering of Polycrystalline Thin Films: *Max O. Bloomfield*[1]; Yeon Ho Im[1]; Timothy S. Cale[1]; [1]Interconnects for Gigascale Integration, Rensselaer, NY USA

Electrical and mechanical properties of thin films has been a primary interest of the microelectronics manufacturing industry for several decades. In recent years, as the thickness of these films has approached the length scale of the underlying grain structure, qualitative changes in properties of interest have shown up. This size dependence must be understood in the context of the manufacturing processes that produce the microstructure, and how the microstructure evolves during use. We show results of physically based simulations of grain structure development during physical vapor deposition (PVD) of a thin metallic film on a bare substrate. The simulations show how deposited material grows from an initial island stage, until coalescing into a blanket film. In our simulations, the mean free path of atoms in flight is taken to be long compared to the length scale of the grains, and thus the system is in a ballistic transport regime. Transport is solely along line-of-sight paths, and solid-gas collisions dominate over gas phase collisions. The flux of reactive species is calculated from a particle distribution function and a transmission probabilities from a Monte Carlo based transport code. Arriving metals atoms have relatively low energy, and sticking factors are close to unity, so deposition is non-conformal. Shadowing effects of larger, broad grains are accounted for, and the resulting simulation, which does not assume a solid on solid model, exhibits voiding. The grain boundaries that form as islands impinge upon each other are no longer available for deposition from the PVD source, but are allowed to evolve further under curvature driven motion, to minimize grain boundary energies. A finite element based level set code is used to track the resulting changes in geometry and topology, This method allows us to attach arbitrary properties, e.g., orientation and composition, to each grain. Another finite element code can be used to track transport and reaction in the fluid phase above the evolving surface for non-ballistic regime systems. These systems can include electrochemical and electroless plating.

3:30 PM Break

3:45 PM
Effects of Different Fraction Solids on the Fluidity of Mechanically Stirred A356 Al-Si Alloy: Sh. Nafisi[1]; O. Lashkari[1]; *M. R. Ghomashchi*[1]; A. Charette[1]; [1]University of Quebec, Ctr. for Univ. Rsch. on Aluminum, Dept. of Appl. Scis., 555 Univ. Blvd., Chicoutimi, Quebec G7H-2B1 Canada

One of the main parameters in casting and foundry technology is determination of the molten metal fluidity. This parameter becomes more important in the casting of thin wall parts and thus understanding and controlling fluidity plays an important role in soundness of cast parts. Semi-Solid Metal, SSM, slurry is a mixture of solid and liquid, mushy zone, with solid volume fractions of 0.1-0.5 and apparent viscosity close to oil viscosity at room temperature. It flows easily under pressure and capable of making complicated shapes with high degree of die filling and integrity. In this study, the ability of the semi-solid slurry to continue to flow is investigated through the effect of different fraction solids and holding temperatures. The results show that in mechanically stirred semi-solid A356 Al-Si alloy, increasing fraction of solid causes decreasing of fluidity. This critical parameter is also related to different process variables such as stirring speed and time and holding time after stirring. Furthermore, the interrelationship between microstructure evolution and fluidity is discussed.

4:05 PM
Multiphase Flow Modeling of Quenching Heat Treatment Process: *Mohammed Maniruzzaman*[1]; Richard Sisson[1]; [1]Worcester Polytechnic Institute, Ctr. for Heat Treating Excellence, Mech. Engrg. Dept., 100 Inst. Rd., Worcester, MA 01609 USA

The quenching of hot metal parts in a liquid quenchant involves several complex heat and fluid flow processes. Different heat transfer mechanisms occur at different stages of cooling, namely film and nucleate boiling and convection. The heat extraction rate varies by several orders of magnitude over these stages and is a function of quenchant type and its physical properties as well as fluid flow parameters (such as Reynolds number, Grashoff's number etc.). The majority of the heat extraction occurs during the boiling stage. Formation of a bubble and its growth and detachment from the hot surface play a major role in the heat-extraction. In this paper, a Computational Fluid

Dynamics (CFD) methodology is developed to simulate numerically the boiling heat transfer during quenching heat treatment process. Predicted heat extraction rate is compared with the experimental result.

4:25 PM
Natural Occurrence of Quasicrystal Shape Found at Nio, Yamaguchi, Japan: *Yasunori Miura*[1]; [1]Yamaguchi University, Chmst. & Earth Scis., Yoshida 1677-1, Yamaguchi 753-8512 Japan

Natural occurrence of quasicrystal type materials is shown from meteorite shower at Nio, Yamaguchi, Japan. The size of polyhedral shape is varied from 150-400micrometer (in major spherule) and 10-70micrometer (in micro-spherule). The texture is polyhedral shapes (from 10 to 70 micrometer). The composition is Fe-rich (90-96%Fe) mixture with minor Si, Al, Ca (sample No.N1P-3, homogeneous) and Si, Al, Mg, Na, Mn, Ca (No.sp-8, as tweedy texture) by shock wave mixing.

4:45 PM
Application of Multiphase Modelling to Hydromet Unit Operation Design: *Lanre Oshinowo*[1]; Lowy Gunnewiek[1]; Tom Plikas[1]; [1]Hatch Associates Ltd., 2800 Speakman Dr., Mississauga, Ontario L5K 2R7 Canada

The incorporation of advanced analysis tools, such as CFD, into the process plant design process has become possible through advances in commercially available computational fluid dynamics software and faster computers. Multiphase modelling is now typically employed in the design phase of process plant design at Hatch. The additional rigour allows process engineers to come closer to realising true virtual plant design. The objective of this paper is to outline the benefits and pitfalls in the application of multiphase modelling for rigorous design. Several application examples in the hydrometallurgical field will be presented.

Nanostructured Materials for Biomedical Applications: Session VI

Sponsored by: Electronic, Magnetic & Photonic Materials Division, EMPMD-Thin Films & Interfaces Committee
Program Organizers: Roger J. Narayan, Georgia Tech, School of Materials Science and Engineering, Atlanta, GA 30332-0245 USA; J. Michael Rigsbee, North Carolina State University, Department of Materials Science and Engineering, Raleigh, NC 27695-7907 USA; Xinghang Zhang, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Wednesday PM Room: 219A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Andrew Shreve, Los Alamos National Laboratory, Biosci. Div., Los Alamos, NM 87545 USA; Prashant Kumta, Carnegie Mellon University, Dept. of Matls. Sci. & Engrg., Pittsburgh, PA 15213 USA

2:00 PM Invited
Polymer/Inorganic Nanocomposites for Biomedical Applications: *Evangelos Manias*[1]; Ruijian Xu[1]; Alan J. Snyder[2]; James P. Runt[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 325-D Steidle Bldg., Univ. Park, PA 16802 USA; [2]Pennsylvania State University, Coll. of Medicine, The Milton S. Hershey Medical Ctr., Hershey, PA 17033 USA

This presentation provides an overview of our activities on polymer/inorganic nanocomposites for biomedical applications. The focus is on polymeric elastomers, in particular poly(urethane urea)s, and our strategies to reduce gas and water permeabilities upon formation of a nanocomposite with organically modified layered silicates (OLS). In contrast with conventional/macroscopic fillers the nanocomposites employing these high-aspect ratio 1nm thin particles can achieve high improvements in permeabilities (up to 400%), without sacrificing the elastomeric character, the strength, and the ductility of the polymer. A discussion tracing the materials properties to the molecular aspects of the nanocomposite structure will also be presented, so as to derive generally applicable design paradigms for polymer nanocomposites with desired properties.

2:35 PM Invited
Nanostructured Conducting Polymer Nanofibers: *Hsing-Lin Wang*[1]; Wenguang Li[1]; [1]Los Alamos National Laboratory, Chmst. Div., MSJ586, C-PCS, Los Alamos, NM 87545 USA

Polyaniline is one of the most promising conducting polymers for commercial applications because it is inexpensive, easy to make, and environmentally stable. In this work, we report the synthesis and characterization of polyaniline chiral nanofibers and their potential applications for chemical separations. Our results show that by varying the experimental parameters, nanofibers can have tunable chirality and can differ by five orders of magnitude. In addition, the thin film cast from the as-synthesized nanofiber solution exhibits micro- and mesoporous features. The as-synthesized chiral, polyaniline nanofibers have a very high surface area and chirality. These characteristics make them extremely attractive candidates for chiral stationary phase in column chromatography, where they separate drugs, amino acids, proteins, and molecules that have asymmetric carbons (chiral centers). This report will discuss the detailed characterization of these nanostructured polyaniline nanofibers by UV-Vis, CD, TEM, and X-ray spectroscopy.

3:10 PM Invited
Temperature-Responsive Polymers and Their Applications in Nanostructures: *Evangelos Manias*[1]; Mindaugas Rackaitis[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., 325-D Steidle Bldg., Univ. Park, PA 16802 USA

Stimuli responsive materials are central in biomedical applications involving chemical sensing and/or stimuli-driven actuation. A systematic series of temperature-responsive polymers were synthesized and studied, and the onset of their T-response was tailored by design of their monomer. Their T-response was studied both for their water solutions, and when they were end-tethered on a surface. Thermodynamic considerations for the monomer design, afford the possibility to fine-tune the lower critical solution temperature (LCST) point, at values ranging from 5 to 70°C, in water. Solubility studies and phase diagrams were done for the solutions, whereas water contact angle, ellipsometry, and atomic force microscopy were carried out for the end-grafted polymers, as a function of grafting density. Model microfluidic devices employing these polymers as T-responsive gates were also realized.

3:45 PM Invited
Peptide-Polymer Conjugates for Surface Modification and Nanoparticle Stabilization: *Phillip B. Messersmith*[1]; [1]Northwestern University, Biomed. Engrg., 2145 Sheridan Rd., TECH E311, Evanston, IL 60208 USA

Certain marine organisms secrete remarkable protein-based adhesive materials for adherence to the mineral, metal, and wood surfaces upon which they reside. For example, mussel adhesive proteins (MAPs) contain L-3,4-dihydroxyphenylalanine (DOPA), an amino acid that is believed to be responsible for the adhesive characteristics of MAPs. In this presentation we will describe our efforts to exploit the adhesive qualities of DOPA containing peptides to control cell behavior at surfaces. We have developed a simple strategy for solution modification of material surfaces utilizing conjugates of DOPA-containing peptides and the nonfouling polymer poly(ethylene glycol) (PEG). Exposure of a variety of material surfaces (e.g. gold, titanium, stainless steel, etc.) to a solution of DOPA-PEG polymer results in deposition of PEG onto the surface and significantly reduced protein and cell adsorption to the surface. Obvious applications of this strategy include protein and cell-resistant surfaces for medical applications, biofunctionalized surfaces, and stabilization of nanoparticles under physiological conditions.

4:20 PM
Nanostructured Magnetic Materials for Biomedical Applications: *Raju V. Ramanujan*[1]; [1]Nanyang Technological University, Sch. of Matls. Engrg., Blk. N4.1, Singapore 639798 Singapore

Biodegradable polymer coated magnetic systems, called magnetic carriers, have several important scientific and biomedical applications. The synthesis, by the ball milling process, of magnetic powders of iron oxide, cobalt and iron was carried out. The grain size of the powders following milling was in the nanometer range. The milling time required to form the minimum size of the powders was determined. The biodegradable polymer PLLA and theophyllin was subsequently coated around the powders by the solvent evaporation method. Various parameters such as milling time, agitation speed and polymer concentration were systematically varied to observe the change in magnetic carrier properties. The process parameters to produce magnetic carriers with a small average size, spherical morphology and narrow size distribution were determined. It was found that the agitation speed and polymer viscosity were important parameters to control the size and shape of the carriers. The surface morphology and magnetic properties of the carriers was also analyzed by SEM and

VSM, respectively. These results will be presented and their significance will be highlighted.

4:55 PM Invited
Nanoengineered Responsive Polymer Surfaces for Micro/Nanofluidic Bioanalytical Systems: *Qiao Lin*[1]; [1]Carnegie Mellon University, Mech. Engrg., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Recent advances in macromolecular engineering techniques allow for precisely controlled polymer chain length, uniformity, composition, topology and functionality. We exploit these advances to develop nanoengineered, intelligent polymer nanolayer coatings for surface modification of micro/nanofluidics-based bioanalytical systems. These polymer nanolayers, with a dry thickness ranging from 1 nm to 100 nm, are covalently grafted to the substrate surface with well-controlled chain densities and well-ordered chain orientation, and exhibit large changes in swelling and wettability properties in response to temperature changes. We discuss the synthesis and characterization of these polymer nanolayers, and their patterning using microfabrication techniques. The biomedical utility of this innovative surface modification technique is demonstrated by its application to microscale fluid handling. Specifically, we explore thermally responsive polymer nanolayers as a means to manipulate droplets and continuous flow in micro/nanofluidic systems, and to provide an intelligent solid phase for microchip chromatography.

Phase Transformations and Deformation in Magnesium Alloys: Deformation and Strengthening

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizer: Jian-Feng Nie, Monash University, School of Physics and Materials Engineering, Victoria 3800 Australia

Wednesday PM Room: 205
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Junichi Koike, Tohoku University, Dept. of Matls. Sci., Sendai 980-8579 Japan

2:00 PM Invited
Atomic Mechanisms of Grain Boundary Sliding and Migration in hcp Metals: *Robert C. Pond*[1]; David J. Bacon[1]; Anna Serra[2]; [1]University of Liverpool, Matls. Sci. & Engrg., Liverpool, Merseyside L69 3BX UK; [2]Universitat Politecnica de Catalunya, Matematica Aplicada III, Jordi Girona 1-3, Barcelona, Catalunya 08034 Spain

Atomic scale simulations of grain boundary structures in hcp metals have been investigated using lattice-statics and molecular dynamics. The interaction of crystal dislocations with interfaces, and the response of these structures to applied shear strains have been studied. The boundaries were initially planar, subject to periodic or fixed border conditions, and the number of atoms in each simulation was conserved. Despite these limitations, insights into the fundamental mechanisms of sliding and migration were obtained. These properties were dependent on boundary structure. In twin boundaries for example, impinging crystal dislocations usually decomposed into glissile twinning and sessile dislocations. In response to applied shear strains, twinning dislocations moved readily, while sessile defects acted as sources of twinning dislocations thereby causing local interface migration. On the other hand, the decomposition products of crystal dislocations reaching incommensurate interfaces became partially delocalised. Such interfaces offered very low resistance to sliding.

2:35 PM Break

3:10 PM Invited
Solid Solution Strengthening in Magnesium Alloys: *Pavel David Lukac*[1]; Zuzanka Trojanova[1]; [1]Charles University, Dept. of Metal Physics, Ke Karlovu 5, Praha 2 12116 Czech Republic

In this paper, the influence of solute atoms on the critical resolved shear stress (CRSS) of both basal and non-basal slip system is presented. The CRSS for the basal slip of Mg alloys increases as $c^{2/3}$ where c is the solute concentration in atomic fractions. The CRSS for glide in non-basal slip systems exhibits an anomalous temperature dependence and its concentration dependence is more complex. The paper presents an attempt to explain the effect of solute concentration on the deformation behaviour of Mg alloy polycrystals. The increased ductility observed in some Mg alloys may be attributed to the enhanced activity of pyramidal slip and the double cross slip with the solute concentration. The temperature and concentration dependence of ductility of a Mg alloy should be complex due to a non-monotonic dependence of the CRSS for non basal slip on the test temperature.

3:45 PM Invited
Modelling of the Precipitation Processes and Strengthening Mechanisms in Mg-Al-(Zn) Automotive Alloys: *Christopher R. Hutchinson*[1]; Jian-Feng Nie[1]; Stéphane Gorsse[2]; [1]Monash University, Sch. of Physics & Matls. Engrg., Clayton, Victoria 3800 Australia; [2]Université Bordeaux 1, Inst. de Chimie de la Matière Condensée de Bordeaux, Bordeaux 33608 France

Mg-Al based alloys are the best known Mg alloys and form the basis of the well known precipitation hardened AZ91 series. Precipitation from the supersaturated solid solution is thought to involve the direct formation of lath shaped equilibrium $Mg_{17}Al_{12}$ phase, predominatly on the basal planes of the matrix. The resulting hardening has been measured by several investigators and the microstructural evolution characterised. In any multi-phase metallic alloy, the strengthening arises from the competition between different hardening modes (peierls, grainsize, solid solution, orowan, composite etc.) In this presentation we report our efforts at modelling the precipitation (mean size and number density) and the hardening resulting from isothermal heat treatments of AZ91. The calculations are compared with experimental data and efforts are made to explain the interesting shape of the Hardness vs. Isothermal ageing time curves for AZ91.

Roytburd Symposium on Polydomain Structures: Domains in Ferroelectrics and Magnetics

Sponsored by: TMS, MPMD-Phase Transformations Committee-(Jt. ASM-MSCTS)
Program Organizers: Julia Slutsker, National Institute of Standards and Technology, CTCMS, Gaithersburg, MD 20899 USA; Greg B. Olson, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208 USA

Wednesday PM Room: 216A
March 17, 2004 Location: Charlotte Convention Center

Session Chair: Alexander L. Roytburd, University of Maryland, Matls. Sci. & Engrg., College Park, MD 20742 USA

2:00 PM Invited
Domain Formation at Phase Transitions in Ferroelectric Films with Semiconducting and Other Electrodes: Alexandre M. Bratkovsky[2]; *Arkadi P. Levanyuk*[1]; [1]Universidad Autonoma de Madirid, Depto. de Fisica de la Materia Condensada, C-III, Cantoblanco, Madrid 28049 Spain; [2]Hewlett-Packard Laboratories, 1501 Page Mill Rd., 1L, Palo Alto, CA 94304 USA

A second order phase transition in ideally homogeneous infinite ferroelectric plate with electrodes made of an ideal metal would proceed into a single-domain state. We investigate a realistic situation of a phase transition in a ferroelectric film with real electrodes made of a semiconductor or a metal. The phase transitions in ferroelectrics with semiconductor electrodes have been discussed long ago by Batra et al.[1] and more recently by Watanabe.[2] However, previous authors have used an inappropriate thermodynamic potential, based their conclusions on numerical calculations, and neglected or inadequately considered domain formation at the phase transition. They have reached a conclusion that in a ferroelectric with semiconductor electrodes the second order phase transition becomes first order, when the transformation proceeds into a single-domain state. We give a consistent analytical treatment of the problem and conclude that the phase transition remains second order with formation of a domain structure in otherwise ideal samples with practically any realistic electrodes. [1]I.P. Batra, P. Wurfel, and B.D. Silverman, Phys. Rev. B8, 3257 (1973). [2]Y. Watanabe, J. Appl. Phys. 56, 527 (1990).

2:35 PM Invited
Energy Minimization and Domain Structure in the Intermediate State of a Type-I Superconductor: Rustum Choksi[2]; *Robert V. Kohn*[1]; Felix Otto[3]; [1]New York University, Courant Inst., 251 Mercer St., New York, NY 10012 USA; [2]Simon Fraser University, Dept. of Math., Burnaby, BC V5A 1S6 Canada; [3]Universitaet Bonn, Angewandte Mathematik, Bonn 53115 Germany

We often "explain" observed microstructures using arguments based on energy minimization. Usually such arguments minimize the energy within a suitable ansatz; only rarely do they also justify the ansatz.

The present work is of the latter type. Our focus is the intermediate state of a type-I superconductor, in the simplest possible case: a plate under a transverse applied field. This is a two-phase problem, involving normal and superconducting domains. The energy, first formulated by Landau, involves a (long-range) magnetic term as well as (local) surface and condensation terms. Landau used energy minimization to predict that flux domains should branch. He missed, however, the problem's remarkable richness when the applied field is near-zero or near-critical. In these regimes there are two small parameters — the normalized surface tension and the volume fraction of one phase — and scaling law of the minimum energy depends on the relation between them.

3:10 PM Break

3:25 PM Invited
Effect of External Mechanical Constraints on Phase Stability and Poly-Domain Structures in Thin Films: S. Choudhury[1]; Y. L. Li[1]; Z. K. Liu[1]; *Long-Qing Chen*[1]; [1]Pennsylvania State University, Matls. Sci. & Engrg., Univ. Park, PA 16802 USA

Many new applications of materials require the growth of thin films on a substrate. It is known that the type of substrate and its crystallographic orientation can have profound effects on the phase transformations and domain structures, and hence properties of thin films as it was shown by Roytburd many years ago. In particular, a substrate can impose a thermal stress due to thermal expansion coefficient difference between the film and substrate or a coherency stress due to the difference in the stress-free lattice parameters of the film and substrate. For phase transformations which produce polydomain structures, we recently developed a phase-field model which can be employed to predict their stability and evolution in thin films. As an example, the phase diagram of a PbZr1-xTixO3 (PZT) film constrained by a much thicker substrate was studied using both thermodynamic calculations and phase-field approach. PZT undergoes a cubic to tetragonal or cubic to rhombohedral transformation in the bulk depending on the composition x. It was found that the ferroelectric transition temperature increases under a substrate constraint regardless the nature of the constraint, i.e., tensile or compressive. It is shown that the orthorhombic phase that does not exist in the bulk becomes stable under a tensile constraint, and the rhombohedral phase in the bulk is distorted in the constrained film. The various ferroelectric polydomain structures produced under different substrate constraints will be discussed.

4:00 PM Invited
Polydomain Formation in Ferroelectric Films and Thermodynamic Modeling of Polarization-Graded Ferroelectrics: *S. Pamir Alpay*[1]; Z.-G. Ban[1]; Joseph V. Mantese[2]; [1]University of Connecticut, Dept. of Metall. & Matls. Engrg., Inst. of Matls. Sci., Storrs, CT 06269 USA; [2]Delphi Research Laboratories, Shelby Township, MI 48315 USA

The thermodynamic theory of polydomain structures as applied to an epitaxial film-substrate couple where the film undergoes a cubic-tetragonal phase transformation is discussed. The parameters of polydomain structures and their dependence on the characteristics of the phase transformation, lattice misfits, the film thickness, and external fields are determined. An effective quantitative theoretical model is provided to explain observed experimental results and to predict and to control the microstructure in epitaxial ferroelectric films. The analysis can be elegantly summarized on a "domain stability map," a phase diagram in the plane of the misfit strain due to the mismatch between the film and the substrate and the tetragonality of the film. The second part of the presentation is concentrated on polarization-graded ferroelectrics, which exhibit behavior and properties that are not routinely observed from homogenous ferroelectrics. It is shown that the thermodynamic approach is quite general and readily expandable to graded ferromagnets, ferroelastics, and other ferroic systems with proper modifications.

4:35 PM Discussion: Problems and Perspectives of Design and Control of Domain Structures Mediators: G. B. Olson, A. L. Roytburd and J. Slutsker

Solid and Aqueous Wastes from Non-Ferrous Metal Industry: Session I

Sponsored by: Extraction & Processing Division, EPD-Waste Treatment & Minimization Committee
Program Organizers: Junji Shibata, Kansai University, Department of Chemical Engineering, Osaka 564-8680 Japan; Edgar E. Vidal, Colorado School of Mines, Golden, CO 80401-1887 USA

Wednesday PM Room: 214
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Mikiya Tanaka, National Institute of Advanced Industrial Science and Technology, Tukuba Japan; Ji-Whan Ahn, Korea Institute Geoscience and Mineral Resources, Teajon Korea

2:00 PM Invited
Production of the High Value Material, Alpha-$CaSO_4$ Hemihydrate Out of Spent $CaCl_2$ Solutions by Reaction with $H2SO_4$: *Seref Girgin*[1]; George P. Demopoulos[1]; [1]McGill University, Mining, Metals & Matls. Engrg., 3610 Univ. St., Wong Bldg., Montreal, Quebec H3A 2B2 Canada

$CaCl_2$ can either be found in spent leaching solutions or in waste brines from geothermal fields. One possible treatment option for these solutions is the production of saleable quality gypsum materials by reaction with H_2SO_4-an increasingly difficult to sell by-product of the non-ferrous metallurgical industry. One such high-value material is alpha-$CaSO_4$ hemihydrate (alpha-HH) finding uses in the building industry. Regeneration/production of HCl out of the spent $CaCl_2$ solutions is another benefit of such treatment process. It is indeed the scope of this work to develop an atmospheric process for the conversion of spent $CaCl_2$ solutions to high-value alpha-HH and HCl. In this paper, the crystallization of alpha-$CaSO_4$ hemihydrate out of such chloride solutions is described as a function of various parameters such as solution composition ($CaCl_2$ and HCl concentration, $H_2SO_4/CaCl_2$ ratio, etc.), method of H_2SO_4 addition, temperature and seeding.

2:30 PM Invited
Removal Treatment of Harmful Materials in Waste Water Using Temperature-Sensitive Polymer Gel: *Hideki Yamamoto*[1]; Akihiro Kushida[1]; Noriyuki Heamot[1]; Yuko Takami[1]; Norihiro Murayama[1]; Junji Shibata[1]; [1]Kansai University, Dept. of Chem. Engrg., Japan

The adsorption removal of harmful organic materials in wastewater has been carried out using the adsorption and desorption characteristics of a temperature -sensitive polymer gel which is synthesized from polyvinylalcohol (PVA). A new adsorption removal process using an air lifting type vessel has been designed and examined for practical use. At higher temperatures, the temperature-sensitive polymer gel shrinks because of discharging water, whereas, in contrast, at lower temperatures, the gel swells as a result of absorbing water. The reversibility of the volume change of the synthesized polymer gel is confirmed by changing temperature. The adsorption behavior of organic materials onto PVA polymer gels in water was investigated at various temperatures. The amount of adsorption of organic materials increases remarkably at temperatures higher than about 305K-320K. The saturated amounts of adsorption are about 0.26mmol/g-gel. The organic material in wastewater could be adsorbed and desorbed reversibly onto PVA polymer gel by the temperature swing. The mechanism of adsorption and desorption of organic materials onto the gel can be explained by the hydration and dehydration of the polymer gel. The driving force of the adsorption is considered to be the hydrophobic interaction between PVA polymer gel and organic compounds.

3:00 PM Invited
Treatment of Transition Metal Oxide Wastes by Plasma Driven Electrolysis: *Patrick R. Taylor*[1]; Wenming Wang[1]; Edgar E. Vidal[1]; [1]Colorado School of Mines, Plasma Procg. Lab., Dept. of Metall. & Matl. Engrg., 1500 Illinois St., Golden, CO 80401-1887 USA

An innovative process to treat transition metal oxide sludge from diverse sources is presented. This technology involves using reverse-polarity DC plasma driven electrolysis in molten oxides. The process results in the recovery of the transition metal and other heavy metals from waste. By-products generated throughout the processing will be converted in an environmentally acceptable slag. The significance of this technology is that the treatment of the waste is accomplished in one step which in turn lowers the cost of remediation. Moreover, this process uses high temperature properties of thermal plasmas, which makes it possible to use molten oxides instead of halides as electrolyte.

Chromium was successfully reduced from chromite through the plasma driven electro-reduction process. It was shown that the selection of the appropriate ionic conducting oxides was critical for the formation of a slag that would enhance the reduction of chromium oxide.

3:30 PM Break

3:45 PM
Production of Functional Inorganic Materials, $AlPO_4$-5, from Wastes in Aluminum Regeneration Industry: *Junji Shibata*[1]; Norihiro Murayama[1]; Hideki Yamamoto[1]; [1]Kansai University, Dept. of Chem. Engrg., Osaka 564-8680 Japan

The hydrothermal synthesis of functional inorganic material, $AlPO_4$-5, was carried out using aluminum dross as a starting material. The crystalline $AlPO_4$-5 was obtained by the hydrothermal reaction conducted in an autoclave. Ay the same time, $AlPO_4$-34 and $AlPO_4$ are formed as a by-product. The product, $AlPO_4$-5, has a porous structure comprising of aluminum phosphates, and its pore size is 7 Å. The crystal of $AlPO_4$-5 has hexagonal structure. The reaction mechanism of crystalline $AlPO_4$-5 was investigated from the surface property of precursor and the property of tri-ethyl amine as a reaction promoter.

4:05 PM
Fluid Bed Destruction of Aqueous and Other Wastes: A Study in Design: *Larry M. Southwick*[1]; [1]L.M. Southwick & Associates, 992 Marion Ave., Ste. 306, Cincinnati, OH 45229 USA

Much of industry uses fluid beds units to destroy or process waste streams. The beds may be composed of inert solids (such as sand) or of decomposition products (such as iron oxide from the pickling of steel). Feed materials are often aqueous slurries and may contain combustible materials. Such units frequently develop operating problems relating to the smoothness of fluidization, violent pressure disturbances or coarsening of bed material, often to the point of complete loss of operability. Data from one such unit (processing a dissolved air flotation slurry) will be presented and an analysis made which indicates a common source of such problems. The solution was applied and solved the operating problems on the unit. This analysis can also be applied to other types of fluid bed processing, such as roasting of sulfide-containing feed materials.

4:25 PM
Synthesis of Hydrotalcite from the Wastes Discharged in Aluminum Regeneration Process: *Norihiro Murayama*[1]; Mitsuaki Tanabe[1]; Hideki Yamamoto[1]; Junji Shibata[1]; [1]Kansai University, Dept. of Chem. Engrg., Osaka 564-8680 Japan

The synthesis of hydrotalcite, which is an inorganic anion exchanger and has layer structure of complex hydroxide, is curried out by using aluminum dross and $MgCl_2$ waste solution discharged from aluminum regeneration process. Various properties such as crystal structure, surface structure, specific surface area, thermal gravity and so on are investigated for the obtained reaction product. Hydrotalcite can be synthesized from the aluminum dross and the waste solution containing $MgCl_2$ by precipitation reaction. Interlayer distance of the obtained product is about 0.3nm. The obtained hydrotalcite changes to Mg-Al oxide by the calcination at 723K, and then the hydrotalcite is formed again by rehydration operation after the calcination. Physical properties of the hydrotalcate from the above wastes are almost similar to those from reagent.

4:45 PM
Hydrometallurgical Methods for Treating Copper Electrolytic Sludge: *N. I. Antipov*[1]; A. V. Tarasov[1]; V. M. Paretsky[1]; [1]State Research Center of Russian Federation, State Rsch. Inst. of Nonferrous Metals, 13, Acad. Korolyov St., 129515 Moscow Russia

A hydrometallurgical method developed in the Gintsvetmet Institute for treating anode sludge and based on sulfite leaching (pH 9.2 to 9.6) of gold or hydrochloric acid leaching in the presence of an oxidizing agent permits its efficient quantitative separation from silver at the head of the process, eliminating thereby the need for production of Dore metal and its further electrolytic refining. In the process of sludge treatment, gold and silver are separated into individual high-grade products, i.e., metal powders, and their refining is, therefore, significantly simplified and can be accomplished by the use of sodium sulfite as the main reagent playing the role of a complexing agent and reductant of gold, silver and selenium. Tellurium, lead and antimony are removed from the process circuit in the form of reach intermediate products. Elimination of roasting, sintering and smelting processes improves the environmental performance of the operations and reduces the operating cost for dust recovery and offgas treatment. Investigations conducted have demonstrated that sludge from the Balkhash smelter (Kazakhstan) and Kovoguty Krompahy smelter (Slovakia) can be successfully treated using the proposed technology with recoveries into corresponding metal powders of 99.57% and 95.45% of gold and 99.5% and 98.3% of silver, respectively.

Solidification of Aluminum Alloys: Gas Porosity/ Micro-Macro Segregation

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Solidification Committee
Program Organizers: Men Glenn Chu, Alcoa Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA; Douglas A. Granger, GRAS, Inc., Murrysville, PA 15668-1332 USA; Qingyou Han, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6083 USA

Wednesday PM Room: 207B/C
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Douglas A. Granger, GRAS Inc., Murrysville, PA 15668-1332 USA; Ralph Napolitano, Iowa State University, MSE, Ames, IA 50011 USA

2:00 PM Keynote
Hydrogen in Aluminum and its Alloys: *D. E.J. Talbot*[1]; [1]Consultant, 96 Bury St., Ruislip HA 47TG, Middlesex UK

The effects of hydrogen in aluminum and its alloys are discussed in terms of the occluded forms of hydrogen, kinetics of absorption reactions and control of hydrogen content. Theories of hydrogen solutions and implications for the stability of real solutions are explained. The origins of interdendritic porosity nucleated in the liquid fraction during solidification and secondary porosity nucleated from the solid as microscopic spherical voids are considered in relation to metal composition, hydrogen content, and casting parameters. Residues of ingot porosity survive fabrication as precursors of macroscopic defects whose development depends inter alia on hydrogen absorbed during heat treatments applied in processing. Two potent sources are humid air and surface contamination. Humid air supplies hydrogen by diffusion of OH- ions through the oxidation product to the oxide/metal interface where they are reduced to hydrogen. Oxidation products offering significant diffusion paths include h-alumina on pure aluminum. MgO with extrinsic OH- cations on aluminum-magnesium alloys and LiOH on aluminum-lithium alloys. Catalysis by sulphur contamination and passivation with fluoride vapours are discussed. A quantitative assessment of pore nuclei as sinks for absorbed hydrogen is given, and precipitation of LiH as an alternative sink in aluminum-lithium alloys is explained. Surface contamination implanted by rolling provides a finite source of hydrogen that can diffuse into the metal. Theory and practice of gas purging to reduce hydrogen contents of liquid metal are described with particular reference to in-line treatment with insoluble active gas mixtures. Interdendritic porosity can be avoided by such treatments, but some secondary porosity is inevitable; and its effects are minimized by appropriate onward processing. Facilities to determine hydrogen contents in the liquid by Telegas type instruments and in the solid by vacuum extraction are recommended.

2:30 PM
Effect of Hydrogen Porosity and As-Cast Grain Structure on the Mechanical Properties of Cast and Forged Al-1.0%Mg-0.6%Si Alloy: *Makoto Morishita*[1]; Kiminori Nakayama[2]; Men Glenn Chu[3]; [1]Kobe Steel, Ltd., Matls. Rsch. Lab., 15, Kinugaoka, Moka, Tochigi 321-4367 Japan; [2]Kobelco Research Institute, Inc., Engrg. Mech. Div., 1-5-5, Nishi-ku Takatsukadai, Kobe, Hyogo 651-2271 Japan; [3]Alcoa Technical Center, Solidification & Molten Metal Proc. Fundamentals Div., 100 Tech. Dr., Alcoa Ctr., PA 15069 USA

Hydrogen porosity is considered an undesirable feature in aluminum wrought alloy ingots. However, its impact on the mechanical properties of fabricated products is rarely found in the literature. The purpose of this paper is to report the influence of hydrogen porosity and as-cast grain structure on the mechanical properties of an experimental Al-1.0%Mg-0.6%Si alloy. The samples were cast using a bench scale apparatus at the Alcoa Technical Center and subsequently preheated and forged at Kobe Materials Research Laboratory. In this investigation, cast samples were prepared with three different levels of hydrogen and two levels of grain refinement. They were then preheated and forged with three different amounts of reduction. The mechanical properties of both as-cast and forged samples were measured using tensile and toughness tests. The results from these tests will be presented and discussed.

2:50 PM
Identifying Sources of Gas Causing Porosity Defect in the Lost Foam Aluminum Castings: *Wanliang Sun*[1]; Harry E. Littleton[1]; Charles E. Bates[1]; [1]University of Alabama, Matls. Sci. & Engrg., 917 Bldg., 1530 3rd Ave. S., Birmingham, AL 35294-4480 USA

During the metal filling of the Lost Foam Casting (LFC), existence of the gaseous pyrolysis products around the molten aluminum makes the understanding of the gas porosity defect formation in the lost foam aluminum castings more complicated. In this study, the sources of the gas causing the porosity defects were identified through series of experiments. First, the surfaces of the gas pores in the lost foam aluminum casting, RPT (Reduced Pressure Test) sample and aluminum sand casting were studied visually. Then, the surface of the gas pores in the LFC was studied with SEM (Scanning Electric Microscope) and EDX (Energy Disperse X-ray). Carbon, oxygen and aluminum were detected on the surface of some of the gas pores. Thirdly, the formation of the gas pores in the lost foam casting was visualized using the state-of-art real time X-Ray technology. This experiment provides further evidence that the gas pores in the LFC is predominantly pyrolysis products related.

3:10 PM
Association of Hydrogen with Lithium in Aluminum Alloys: *Dilys M. Henry*[1]; David E.J. Talbot[2]; [1]DuPont Corporate Research & Development, Corp. Failure Analysis, Experimental Sta., E304/C331, Wilmington, DE 19805 USA; [2]Consultant, 96 Bury St., Ruislip HA 47TG, Middlesex UK

The solubilities of hydrogen in the alpha-phase of aluminum alloys containing lithium are an order of magnitude greater than in other alloys. Anomalous values for the solution enthalpy and entropy suggest that the additional solubility is due to special sites at which hydrogen atoms are strongly bound. Support for this view is a marked reduction in the mobility of hydrogen the special sites are intuitively identified as the clusters that are the precursors of lithium-rich precipitates responsible for age-hardening. The present work produced further support for an association of hydrogen with the clusters. Small angle neutron diffraction of AA8090, with and without occluded hydrogen introduced during solutionizing at 450°C and at 500°C, identified a reciprocal constraint on the mobility of lithium. Vant' Hoff Isobars for hydrogen solution in aluminum-lithium alloys exhibit discontinuities at critical temperatures that suggest a change in the association of hydrogen with lithium, possibly an order/disorder transformation.

3:30 PM Break

3:50 PM Keynote
Modeling Segregation and Porosity in an Aluminum Casting Alloy: *David R. Poirier*[1]; Pil K. Sung[1]; [1]University of Arizona, Matls. Sci. & Engrg., Tucson, AZ 85721 USA

The dendritic solidification of an aluminum casting alloy is simulated as a means to study the transport phenomena of alloy elements and hydrogen. The transport is modeled using porous media theory, in which mass, solutal species, energy, and momentum conservations form the underlying set of equations. The set solution is effected by a finite element code. Examples that reveal the formation of macrosegregation and the formation of porosity in castings are presented. These will include the effect of high over-pressures during solidification to suppress porosity.

4:20 PM
Modeling the Effects of Mold Topography on Aluminum Cast Surfaces: *Lijian Tan*[1]; Nicholas Zabaras[1]; [1]Cornell University, Matls. Proc. Design & Control Lab., Sibley Sch. of Mech. & Aeros. Engrg., 188 Frank H. T. Rhodes Hall, Ithaca, NY 14853-3801 USA

A comprehensive review will be provided of recent computational studies for the development of a finite element analysis based design methodology with which casting mold surface topographies can be tuned to produce required surface features and microstructural properties of aluminum ingots. A coupled thermomechanical and melt flow analysis has been developed that together with air-gap nucleation and contact modeling algorithms allows us to study the effects of mold topography on cast surfaces in the early stages of aluminum solidification. A sensitivity analysis to quantify the importance of mold topography on the shell surface morphologies will also be discussed. Mold surface topographies, which consist of unidirectional and bidirectional groove textures, are generated to elicit a radiator-like effect at the mold-casting interface. The rate of heat extraction, the evolution of near-surface cast microstructure, and shell macro morphology can be controlled, once the proper balance between mold surface area extension and the degree of imperfect wetting at the instant solidification starts is determined. A control of the surface features in aluminum casting is important in minimizing costly post-casting surface milling or scalping.

4:40 PM
Solidification Morphology and Structure of Cast Al-Li 2090 Alloy at Low Superheat: Chandan Mahato[1]; Mohamed Shamsuzzoha[1]; *Nagy El-Kaddah*[1]; [1]University of Alabama, Dept. of Metallurgl. Engrg., Box 870202, Tuscaloosa, AL 35487 USA

The effect of low superheat on solidification morphology and microstructure of Al-Li 2090 alloy has been investigated using the Magnetic Suspension Melting (MSM) process, which is capable of remelting and casting aluminum at superheats as low as 1°C. In this study, the metal was solidified unidirectionally in a cylindrical ceramic mold with a chill stainless steel block at the bottom. The castings were analyzed by microscopic and image analysis techniques. Metallographic examination showed that the cast metal exhibited fine-grained equiaxed structure throughout the ingot, with an average grain size of about 80μm. The average grain size was found to be independent of both the cooling and solidification rates in the mold. TEM examination showed that the matrix exhibits a super lattice type structure of coherent δ' (Al_3Li) phase with minor amount of T1 (Al_2CuLi) needles in the matrix. Chemical analysis of the ingots showed typical inverse segregation of Cu in aluminum alloys in the direction of solidification, and very little segregation of Zr.

Surfaces and Interfaces in Nanostructured Materials: Coatings and Surface Modification

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Surface Engineering Committee
Program Organizers: Sharmila M. Mukhopadhyay, Wright State University, Department of Mechanical and Materials Engineering, Dayton, OH 45435 USA; Arvind Agarwal, Florida International University, Department of Mechanical and Materials Engineering, Miami, FL 33174 USA; Narendra B. Dahotre, University of Tennessee, Department of Materials Science & Engineering, Knoxville, TN 37932 USA; Sudipta Seal, University of Central Florida, Advanced Materials Processing and Analysis Center and Mechanical, Materials and Aerospace Engineering, Oviedo, FL 32765-7962 USA

Wednesday PM — Room: 217A
March 17, 2004 — Location: Charlotte Convention Center

Session Chair: Narendra B. Dahotre, University of Tenessee, Dept. of Matls. Sci. & Engrg., Knoxville, TN 37932 USA

2:00 PM Invited
Modification of Nanotube-Based Materials by Ion Beam Deposition: Computational Studies: *Susan B. Sinnott*[1]; [1]University of Florida, Matls. Sci. & Engrg., 154 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA

Classical molecular dynamics simulations with many-body empirical potentials are used to study the chemical modification of carbon nanotube based materials. Chemical modification of nanotubes is important for controlling their adhesion to polymers in composites. The simulations focus on the use of ion beam deposition to modify the structure of empty nanotubes and of nanotubes filled with buckyballs. The results show that the tube walls can be chemically functionalized and that cross-links can be produced between adjacent nanotubes in a bundle, between nanotube walls within a multi-walled tube structure, and between the nanotubes and the buckyballs. Additionally, the direct modification of nanotube-polymer composites is considered. The simulations show that this approach can produce cross-links between the tubes and polymer chains in situ, but that there are important structural dependencies in the results. The support of the National Science Foundation through grant CHE-0200838 is gratefully acknowledged.

2:25 PM
Nanoscale Silver Particle Films for High-Temperature Packaging of Semiconductor Devices: *Zhiye Zhang*[1]; Jesus Noel Calata[2]; John G. Bai[2]; Guo-Quan Lu[3]; Yanjing Liu[4]; [1]Virginia Tech, The Bradley Dept. of Elect. & Computer Engrg., Whittemore Hall 618, Blacksburg, VA 24061 USA; [2]Virginia Tech, Dept. of Matls. Sci. & Engrg., 213 Holden Hall, Blacksburg, VA 24061 USA; [3]Virginia Tech, The Bradley Dept. of Elect. & Computer Engrg. & Matls. Sci. & Engrg., Blacksburg, VA 24061 USA; [4]Luna Innovations, Blacksburg, VA 24060 USA

Nanometer-thick silver films were deposited on various substrates (silicon, glass, ceramic, and flexible Kapton© films) by dipping in

stable aqueous solutions containing silver nanoparticles using an electrostatic self-assembly technique at room temperature. The silver particles were synthesized by reduction of silver nitrate with sodium borohydride in a solution containing PAA. Characterization of the films using SEM, AFM and XPS shows that the nano-sized silver particles ranging from 30 to 50 nm in diameter are packed closely and homogeneously on the substrates. Silver nanoparticles could lead to significantly lower sintering temperatures with a potential application in high-temperature interconnections of future electronic devices such as SiC. Preliminary sintering tests on the films show widespread neck formation between particles at temperatures as low as 250°C. Further research and investigation of potential applications are ongoing.

2:45 PM

Laser Induced In Situ Synthesis of Nanocomposite Carbide Coating on Steel: Anshul K. Singh[1]; *Narendra B. Dahotre*[1]; [1]University of Tennessee/UTSI, Dept. of Matls. Sci. & Engrg., 10521 Rsch. Dr., Ste. 400, Knoxville, TN 37932 USA

The stress on reducing the size of most contemporary equipments has amplified the importance of nanoscale technology significantly which thus augments the importance of surface properties of materials. With the aim of synthesizing a nano composite carbide coating, a 2.5 KW Nd: YAG laser was employed on the surface of AISI 1010 steel deposited with a precursor powder mixture of Fe, Ti, Cr and C. In-situ formation of ultra hard nanoscale carbide particles (TiC and chromium carbides) dispersed in a Fe-based matrix was observed. The nano composite coating thus achieved has far superior surface properties viz. hardness and wear resistance.

3:05 PM

Chemical Bonding and Morphology of the Thin Carbon Films Created on SiC Surface by the Carbide-Derived Carbon (CDC) Process: *Alexander V. Zinovev*[1]; Jerry F. Moore[1]; Michael J. Pellin[1]; John A. Carlisle[1]; Orlando H. Auciello[1]; John N. Hryn[1]; [1]Argonne National Laboratory, Matl. Sci. Div., 9700 S. Cass Ave., Argonne, IL 60439 USA

The treatment of silicon carbide ceramic in chlorine contained gas mixture was carried on. Due to the energetically favored reaction of Cl_2 with Si rather than C the selective etching of the SiC can take place, leading to the formation of a carbide-derived carbon (CDC) film. The result of XPS and also Raman spectroscopy, AES and SEM studies of chemical conversion of single crystal SiC and industrial polycrystalline silicon carbide surface treated at different gas concentrations and different temperatures will be presented. XPS analysis of the carbon C1s fundamental peak and the valence band shows that created CDC films are not phase pure but consist of a mixture of sp2 and sp3 bonded carbon. Differences observed in the conversion rate and phases formed on different types of SiC substrates (single crystal or industrial ceramics) will be discussed and are likely due to grain boundary and surface morphology effects.

3:25 PM

Nanometer-Scale Coatings for Nano-Structured Solids: *Sharmila M. Mukhopadhyay*[1]; [1]Wright State University, Mech. & Matls. Engrg., 3640 Colonel Glenn Hwy., Dayton, OH 45435 USA

The concept of surface coatings for property modification is not new, but when the bulk structure to be coated is itself <100 nm in dimension, new challenges emerge. The coating has to be substantially thinner than bulk dimensions, yet be functional. In this presentation, it will be shown that some plasma assisted coatings having thickness in the 1-5 nm range can be very effective for modification of nanofibers, near net shape cellular foams, and other porous materials. Two types of coatings are focused upon initially: (i) those for enhancement surface reactivity and bonding, and (ii) those that make the surface inert. The former has been achieved by a nano-layer of SiO2-type compound, and the latter has been achieved by 1-2 nm layer of -CF2- chains. The initial stages of growth of these layers (from atomic clusters to complete mono-layers) have been studied. XPS and AFM results appear to correlate very well with physical properties such as wettability, bond formation and fluid infiltration. Additional aspects of these coatings specific to applications, such as permeability through porous solid, durability, and future possibility of creating multi-layer and multi-functional nano-coatings will be discussed.

3:45 PM

Auto-Organized Nanostructures in the Ti-Al-N Thin Film System: *Lars Hultman*[1]; [1]Linköping University, Thin Film Physics Div., IFM, Linkoping S-581 83 Sweden

The process of age hardening as a means for advanced surface engineering of nanostructured materials has been evidenced in thin film applications. A model system, Ti1-xAlxN, was chosen as such coatings are known for their excellent wear resistance enabling improved machining processes like high-speed and dry cutting. Physical vapour deposition methods with relatively low substrate temperatures are employed to produce supersaturated solid solutions of the material by virtue of the kinetic limitations during synthesis. It is shown using TEM, HREM, XRD, differential scanning calorimetry, and nanoindentation that Ti1-xAlxN coatings with compositions in the miscibility gap initially undergo spinodal decomposition into coherent cubic-phase nanometer-size domains, causing an increase in hardness at elevated temperatures. These intermediate metastable domains transform into their stable phases TiN and AlN during further thermal treatment. Activation energies for the processes indicate defect-assisted segregation of Ti and Al. The findings are corroborated by ab initio calculations of phase stability and molar volume for competing phases. It is inferred that the success of Ti1-xAlxN coatings is not only based on its superior oxidation resistance, but also on its ability for self-adaptation to the thermal load applied during cutting by age hardening. The findings and experimental approach have implications also for other ternary and multinary ceramic systems including the group-III nitride alloys.

4:05 PM

Effect of Processing Parameters on Properties of Nanocrystalline FeCrP Electrodeposits: *C. T. Kunioshi*[1]; N. B. de Lima[1]; O. V. Correa[1]; L. V. Ramanathan[1]; [1]Cidade Universitaria, Matls. Sci. & Tech. Ctr., Inst. de Pesquisas Energeticas e Nucleares, Av. Prof. Lineu Prestes 2242, São Paulo 05508-000 Brazil

This paper presents the effect of processing parameters such as bath composition, additives to the bath, temperature and current density on the composition, morphology and corrosion resistance of nanocrystalline FeCrP electrodeposits. Deposits with average size of 1.5nm were obtained at current densities up to 150 mA.cm-2 and with bath temperatures up to 50ºC. In the presence of formic acid as a complexing agent and aging of the bath, uniform, adherent and nanocrystallline deposits with 9-17%Cr were obtained. The crystallite size decreased with increase in phosphorous content, and this in turn was influenced by the sodium hypophosphite content of the electrolytic bath. Electrochemical measurements revealed overall increase in corrosion resistance with increase in P content.

4:25 PM

Influence of Surface Deposited Nanosized Rare Earth Oxide Gel Morphology on High Temperature Cyclic Oxidation Behavior of Fe20Cr Alloy: *S. M.C. Fernandes*[1]; L. V. Ramanathan[1]; [1]Cidade Universitária, Matls. Sci. & Tech. Ctr., Inst. de Pesquisas Energéticas e Nucleares (IPEN), Av. Prof. Lineu Prestes 2242, São Paulo 05508-000 Brazil

The use of rare earths to increase the high temperature oxidation resistance of chromia and alumina forming alloys is well known. The rare earths can be added as elements to the alloys or applied on the metal surface as oxides. Several methods have been used to apply rare earth oxide coatings on metal surfaces and the sol-gel technique has been shown to be very efficient. This technique generates nano-sized oxide particles. The influence of sol-gel processing parameters on rare earth (RE) oxide (Y2O3, La2O3, CeO2, Nd2O3, Pr2O3, Dy2O3) morphology has been studied. The influence of RE oxide gel morphology and nature on cyclic oxidation behavior (RT-900ºC) of Fe20Cr alloy has also been studied. This paper presents the results of these studies are discusses the role of RE oxide gel morphology (size, shape, distribution) and RE ion radius on cyclic oxidation resistance of the chromia forming alloy.

4:45 PM

A FIB-SIMS Study of Interfaces in TiN/Cu Multilayered Thin Films: *Alessio Lamperti*[1]; Gregory Abadias[2]; Paolo Maria Ossi[1]; Carlo Enrico Bottani[1]; Riccardo Levi-Setti[3]; [1]Politecnico di Milano, Dipartimento di Ingegneria Nucleare, Via Ponzio, 34-3, Milano I-20133 Italy; [2]Université de Poitiers, Lab. de Métallurgie Physique - UMR CNRS 6630, SP2MI Téléport 2, BP 30179, Futuroscope-Chasseneuil Cedex F-86962 France; [3]University of Chicago, Enrico Fermi Institute, 5514, S. Ellis Ave., Chicago, IL 60637 USA

Among the methods to prepare nanoscale superhard composite coatings the combination of a hard nitride phase (TiN) with a soft metallic phase (Cu) improves coating toughness, while retaining its high hardness. A critical role to obtain a multilayer film with excellent properties is played by the sharpness of its interfaces. Multilayered TiN(1.5-9.1 nm)/Cu(3.4-11.9 nm) superlattices were deposited by dual ion beam sputtering. Previous low-angle XRD and High Resolution TEM showed the presence of several interface defects that could destroy superlattice periodicity. Here Focused Ion Beam - Secondary Ions Mass Spectrometry measurements were performed to study the sharpness and the degree of element intermixing at interfaces as well

as the interface symmetry/asymmetry, with a vertical resolution of few nanometers and 50 nm lateral resolution. The obtained results are critically discussed.

The Didier de Fontaine Symposium on the Thermodynamics of Alloys: Joint Session with Computational Thermodynamics and Phase Transformations II

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)

Program Organizers: Diana Farkas, Virginia Polytechnic Institute and State University, Department of Materials Science and Engineering, Blacksburg, VA 24061 USA; Mark D. Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Gerbrand Ceder, Massachusetts Institute of Technology, Department of Materials Science and Engineering, Cambridge, MA 02139 USA; Christopher Mark Wolverton, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48121-2053 USA

Wednesday PM Room: 216B
March 17, 2004 Location: Charlotte Convention Center

Session Chair: TBA

2:00 PM Invited
Modern Computational Materials Science: The Problem of Alloy Phase Stability in Complex Materials: *A. J. Freeman*[1]; [1]Northwestern University, Dept. of Physics & Astron., 2145 Sheridan Rd., Tech F-269, Evanson, IL 60208 USA

This talk, dedicated to Didier de Fontaine, reviews developments in computational electronic structure theory which have led to the present state of the art determination of alloy phase stability in complex systems, including multi-component aluminide intermetallics made up of transition and rare-earth metals, the prediction of new phases with ternary additions in bulk and at surfaces and interfaces, the dependence of structural phase stability on magnetism, etc. The special problems of treating rare-earth 4f electrons in multi-component intermetallics will be discussed. Illustrative examples will be given on our studies of aerospace materials obtained with our highly precise full-potential first-priniciples methods, which are now capable of treating complex structures with a large number of atoms/cell and to calculate their structural, electronic, magnetic, optical and, more recently, mechanical properties. Currently, the objective of our research is to determine the microscopic mechanisms governing the deformation and fracture behavior of ordered intermetallic aerospace alloys in order to contribute to the development of a fundamental basis for computer-aided alloys design. The most important and challenging goal of our research is to bridge the gap between a microscopic quantum-mechanical description of the chemical bonding and the mesoscopic phenomena which govern the mechanical response of intermetallics. A key feature of the work is our use of a combined approach, which includes first-principles calculations of cleavage/shear energetics and analysis of the dislocation structure within the generalized 2D Peierls-Nabarro model with ab-initio parameterization of the restoring forces. This approach was applied to studies of (1) homogeneous intermetallic alloys with emphasis on the role of structural stability on the mechanical properties and the yield stress temperature anomaly, and (2) heterogeneous intermetallic systems, including investigations of eutectic composites and the temperature dependence of the lattice misfit in gamma/gamma superalloys. Work supported by the AFOSR, performed in collaboration with Yu. N. Gornostyrev, O. Yu. Kontsevoi, and N.I. Medvedeva.

2:30 PM Invited
Ab Initio Alloy Thermodynamics: Recent Progress and Future Directions: *Axel van de Walle*[1]; Mark D. Asta[1]; Gerd Ceder[2]; Christopher Woodward[3]; [1]Northwestern University, Matls. Sci. & Engrg., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [2]Massachusetts Institute of Technology, Matls. Sci. & Engrg., Rm. 13-5056, 77 Mass. Ave., Cambridge, MA 02139 USA; [3]Air Force Research Laboratory, Wright-Patterson AFB, OH 45433 USA

Ab initio calculations of the thermodynamic properties of alloys have traditionally suffered from two main shortcomings. First, properly accounting for nonconfigurational sources of entropy is a challenging task. Second, commercial alloys typically consist of numerous components, while first-principles calculations of the properties of multicomponent alloys have rarely been undertaken. Although the paucity of first-principles phase diagrams calculations of multicomponent systems including nonconfigurational entropy contributions has often been attributed to their heavy computational requirements, we show that the development of automated computational tools goes a long way towards making such task practically feasible. We describe recent progress in the development of a software package, the Alloy Theoretic Automated Toolkit (ATAT), which substantially simplifies the tasks necessary for the calculations of thermodynamic properties from first-principles. Examples of applications to the Ti-Al and Ni-Al binary systems as well as multicomponent systems are presented. The calculated properties go beyond the ones typically included in thermodynamic databases. Thanks to the atomistic nature of the calculations, the properties of coherent interfaces as well as short-range order parameters can also be obtained from the same underlying formalism.

3:00 PM
Experimental and Theoretical Evidence for Carbon-Vacancy Binding in Austenite: *Ronald Gibala*[1]; Christopher Wolverton[2]; [1]University of Michigan, Dept. of Matls. Sci. & Engrg., 2300 Hayward, 2026 H.H. Dow Bldg., Ann Arbor, MI 48109-2136 USA; [2]Ford Motor Company, Scientific Rsch. Lab., Dearborn, MI 48121-2053 USA

We have examined several sources of experimental data on face-centered cubic iron-carbon alloys and carbon-containing austenitic alloys which imply that the binding energy between interstitial carbon solutes and vacancies is large, of the order 40-60 kJ/mol, and that vacancies are the more mobile of the point defects. The experimental data include point-defect anelasticity, self diffusion, high-temperature steady-state creep deformation, strain aging and strain-age hardening, and radiation damage. As a complement to these data, we have performed first-principles gradient-corrected density functional calculations to determine directly the binding energy of nearest-neighbor carbon-vacancy pairs. A value of 36 kJ/mol is obtained, and the calculations suggest additional but more modest binding of second nearest-neighbor pairs. Other stability calculations, including ones for metastable carbides in the iron-carbon system, have been made and will be reported.

3:20 PM
Driving Force for Nanometer-Scale Composition Modulation in Fe-Ag/Mo(110) Alloy Films: Bo Yang[1]; *Mark D. Asta*[1]; Vidvuds Ozolins[2]; [1]Northwestern University, Dept. of Matls. Sci. & Engrg., 2225 N. Campus Dr., Evanston, IL 60208 USA; [2]University of California, Dept. of Matls. Sci. & Engrg., Los Angeles, CA USA

Compositionally-modulated structures in two-dimensional Fe-Ag/Mo(110) alloys are studied by first-principles within a recently developed hybrid continuum/atomistic model for the elastic energy (V. Ozolins, M. Asta and J. J. Hoyt, Phys. Rev. Lett. 88, 096101 (2002)). The experimentally observed composition-modulation peridocities of ~2 nm can be understood as arising from a competition between "chemical" contributions to the alloy mixing energy, favoring phase separation, and elastic energy which favors ordering. Monte-Carlo simulations predict that Fe-Ag/Mo(110) stripe structures are thermally stable to disordering up to relatively high temperatures of ~900 K. Simulations are employed to examine the regions of stability of higher-order structures found in a zero-temperature phase diagram featuring a "devil's staircase" of striped phases. We highlight the numerous ways in which our work has been influenced by Prof. de Fontaine's seminal contributions to the theory of size-mismatched alloys and long-period superstructures.

3:40 PM Break

3:50 PM Invited
Structure and Dynamics of Light Metal Hydrides: *Vidvuds Ozolins*[1]; Eric H. Majzoub[2]; David R. Tallant[3]; [1]University of California, Dept. of Matls. Sci. & Engrg., PO Box 951595, BH 6532F, Los Angeles, CA 90095-1595 USA; [2]Sandia National Laboratories, Matls. Chmst., PO Box 969, Livermore, CA 94551-0969 USA; [3]Sandia National Laboratories, PO Box 5800, MS 1411, Albuquerque, NM 87185 USA

Light metal hydrogen compounds represent an interesting new paradigm for reversible hydrogen storage. The recent surge in research has been motivated by the discovery that doping with small amounts of Ti makes enhances the kinetics of hydrogen adsorption and desorption reactions by many orders of magnitude. However, the role that Ti plays in accelerating these processes is poorly understood. We will report on combined first-principles and experimental studies of the structural, dynamical and thermodynamical properties of sodium alanates. Results on the physical properties of stable ordered com-

pounds, lattice defects, hydrogen dynamics, and energetics of Ti substitution sites in bulk alanates will be presented.

4:20 PM Invited
Phase Stability and Instability in Bulk Metallic Glass Forming Systems: *Don Nicholson*[1]; Miguel Fuentes-Cabrera[1]; Mike Widom[2]; Yang Wang[2]; Marek Mihalkovic[3]; [1]Oak Ridge National Laboratory, One Bethel Valley Rd., Oak Ridge, TN 37831 USA; [2]Carnegie Mellon University, PA USA; [3]Slovakia Academy of Sciences, Inst. of Physics

Results of first principles calculations of the energy of compounds, alloys, liquids, and amorphous alloys in glass forming composition ranges are presented. Density functional calculations of the diffusion in the liquid state are compared to embedded atom calculations (EAM). The EAM, which was fit exclusively to first principles results, reproduces well the liquid diffusion and was used to calculate diffusion during quenching of liquids. Chemical, magnetic, and topological contributions to glass formability are discussed. Work supported by DARPA/ ONR Grant N00014-01-1-0961 and DOE Office of Basic Energy Science under subcontract DEAC05-00OR22R725464 with UT-Battelle, LLC.

4:50 PM
Impurities Block the Alpha to Omega Martensitic Transformation in Titanium: *Dallas R. Trinkle*[1]; Richard G. Hennig[1]; Johann Bouchet[2]; Srivilliputhur G. Srinivasan[2]; Robert C. Albers[2]; John W. Wilkins[1]; [1]Ohio State University, Physics, 174 W. 18th Ave., Columbus, OH 43210 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87544 USA

The pressure driven martensitic phase transition from alpha to omega Ti has not been found in commercial A-70 Ti and Ti-6Al-4V alloys at pressures up to 35 GPa. It is believed to be retarded by O impurities in A-70 Ti and by Al in Ti-6Al-4V alloys. Ab initio calculations determine the energies and sites of interstitial (O, N, C), substitutional impurities (Al, V), and self-defects. Interstitial impurities are preferably located in the octahedral sites of both phases over the hexahedral sites. Nudged elastic band calculations reveal the influence of the impurities on the transition barrier. The octahedral site in alpha transforms into the octahedral or hexahedral sites in omega on a two to one basis. We predict that impurities in A-70 Ti and Ti-6Al-4V shift the omega phase energy relative to alpha, and increase the energy barriers, thus retarding the alpha to omega transition.

5:10 PM
Atomistic and Microstructural Ordering Processes Studied by Hybridized Calculation of Phase Field and Cluster Variation Methods: *Munekazu Ohno*[1]; Tetsuo Mohri[1]; [1]Hokkaido University, Grad. Sch. of Engrg., Div. of Matls. Sci. & Engrg., Kita-13 Nishi-8, Kita-ku, Sapporo 060-8628 Japan

In order to describe the atomistic and the microstructural ordering processes simultaneously, we attempt hybridized calculations of the Phase Field Method (PFM) and the Cluster Variation Method (CVM). The non-uniform chemical free energy in the PFM is obtained based on the atomistic free energy formulated by the CVM. The gradient energy coefficient which determines the spatial scale and controls the anisotropic effects of the interfacial energy is determined within the CVM. Our particular focus is placed on the L10 ordered system. The one-dimensional calculation shows that the anti-phase boundary is necessarily anisotropic in the L10 system when only the neareset neighbor pair interaction is considered. Two-dimensional calculation reveals microstructural feature during the disorder-L10 transition.

5:30 PM
Shock-Induced Melting and Resolidification of Cu and Si: A Molecular-Dynamics Study: *Babak Sadigh*[1]; George Gilmer[1]; Jeffrey Colvin[1]; Bryan Reed[1]; Mukul Kumar[1]; [1]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci., 7000 E. Ave., L-353, Livermore, CA 94550 USA

We present a comprehensive study of shock-induced melting and resolidification of Cu and Si, using large-scale molecular-dynamics simulations with supercells containing up to a million atoms. We calculate the nucleation rate of voids upon release of the shock wave and evolution of rarefaction waves. We further study the kinetics of melting/ resolidification by analyzing in detail the motion of solid/liquid interfaces in atomistic systems when the two phases coexist. We study the role of the pressure waves in driving the phase transformation by analyzing the evolution of local stress and temperature in the interfacial regions. Using information from solid-liquid interfacial free energy calculations, we attempt to parametrize a macroscopic phase-field model that can predict the microstructural evolution of shock-melted and resolidified Cu and Si. This work was performed under the auspices of the US Dept. of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

The Role of Grain Boundaries in Material Design: Grain Boundary Segregation, Diffusion, Damage

Sponsored by: Materials Processing and Manufacturing Division, ASM/MSCTS-Texture & Anisotropy Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS)
Program Organizers: Brent L. Adams, Brigham Young University, Department of Mechanical Engineering, Provo, UT 84602-0001 USA; Thomas R. Bieler, Michigan State University, Department of Chemical Engineering and Materials Science, East Lansing, MI 48824-1226 USA

Wednesday PM Room: 218A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Thomas R. Bieler, Michigan State University, Dept. of Chem. Engrg. Matls. Sci., E. Lansing, MI 48824-1226 USA; David Field, Washington State University, Mech. & Matls. Engrg., Pullman, WA 99164-2920 USA

2:00 PM Invited
Twin Boundary Formation in Thin Copper Films: *David P. Field*[1]; [1]Washington State University, Mech. & Matls. Engrg., Box 642920, Pullman, WA 99164-2920 USA

In many applications, grain boundary design is aimed at increasing the fraction of special boundaries to improve material properties. In FCC metals, this process typically involves processing techniques that maximize the fraction of twin boundaries in the material. As with bulk materials, thin film microstructures depend upon details of the film processing. For damascene processing of lines the bath chemistry, barrier layer materials and thicknesses, seed layer, line height to width ratio, and annealing temperature and time all play a role in the developing microstructure. This study shows the effects of various processes on grain size and twin boundary fraction. The fraction of twin boundaries is maximized for a particular bath chemistry and annealing temperature by imposing a given line height to width ratio which controls the stress state during annealing.

2:25 PM
Microstructural Engineering of Copper Shaped-Charge Liners: *Kerri J.M. Blobaum*[1]; James S. Stölken[2]; Mukul Kumar[3]; [1]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci., L-370, 7000 East Ave., Livermore, CA 94550-9234 USA; [2]Lawrence Livermore National Laboratory, Mech. Eng., L-129, 7000 East Ave., Livermore, CA 94550-9234 USA; [3]Lawrence Livermore National Laboratory, Chmst. & Matls. Sci., L-356, 7000 East Ave., Livermore, CA 94550-9234 USA

Using a combination of experiments and modeling, we are developing methods for engineering the microstructure of copper shaped-charge liner cones to improve their performance. It is known that factors such as grain size and morphology, texture, and grain boundary character influence the liner jet break-up time, but their coupled effects are not well understood, particularly at the strain rates experienced by the high explosive-driven liners. The deformation experienced during the expansion of the shaped-charge liner jet is akin to superplastic forming, where the strain rate sensitivity and strain hardening characteristics greatly influence the uniform stretching of the material by delaying the onset of plastic instability. Similarly, it is hoped that increasing the fraction of special boundaries decreases the total interfacial area for void nucleation in the microstructure. Here, we describe a systematic study of the effects of engineered microstructures on mechanical properties. Using finite element analysis, a multi-step back-extrusion/annealing process was developed for forming the liners. This process enabled grain boundary engineering of the final microstructure. Results from mechanical testing of these samples, as well as conventionally processed liners, at a range of strain rates and temperatures will be presented. This systematic investigation of the constitutive response of the grain boundary engineered microstructures will be discussed in the context of the performance of shaped-charge liners. This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

2:45 PM
The Effect of Grain Boundary Misorientation, Inclination, Crystal Orientation, and Stress State on Microcrack Initiation in Duplex TiAl Grain Boundaries: Alireza Fallahi[1]; Kris Boyapati[1]; *Thomas R. Bieler*[1]; Martin A. Crimp[1]; Amir Zamiri[2]; Farhang Pourboghrat[2]; Darren E. Mason[3]; [1]Michigan State University, Chem.

Engrg. Matl. Sci., 2527 Engrg. Bldg., E. Lansing, MI 48824 USA; [2]Michigan State University, Mech. Engrg., E. Lansing, MI 48824 USA; [3]Albion College, Dept. of Math. & Compu. Sci., Albion, MI USA

Prior investigations on microcrack initiation and slip transfer in duplex TiAl by Simkin et al. (Scripta Mater. vol. 49, 149-154, 2003) showed that microcracks nucleate primarily in g-g grain boundaries. Simkin developed a fracture initiation parameter based upon the external stress state, available slip and twinning systems, crystal orientations, and Schmid factors that was able to predict the propensity of a boundary to nucleate microcracks arising from twin interactions with a grain boundary. This parameter did not fully consider the effects of grain boundary inclination with respect to the stress state, nor the effects of local elastic anisotropy on the stress state in the boundary. Serial sectioning on Simkin's specimen has been done to determine grain boundary inclinations. FEM analysis of local microstructure regions that incorporate crystal orientations coupled with tensorial descriptions of elastic stiffness are used to estimate the local stress tensor in the boundary regions. The effect of this additional detailed information on the fracture initiation parameter values and their variability are analyzed and discussed, to determine if a physically based deterministic threshold for microcrack initiation can be identified. Supported by AFRL NO. F49620-01-1-0116 (Craig Hartley).

3:05 PM Break

3:25 PM Cancelled
Impact of Grain Boundary Character on Grain Boundary Dynamics

3:50 PM
Accurate Quantification of Local Boundary Excess by STEM X-Ray Mapping: *David B. Williams*[1]; Masashi Watanabe[1]; Chunfei Li[2]; [1]Lehigh University, Dept. of Matls. Sci. & Engrg., 5 E. Packer Ave., Bethlehem, PA 18015 USA; [2]Portland State University, Dept. of Physics, 1719 SW 10th Ave., Portland, OR 97201 USA

X-ray mapping in a scanning transmission electron microscope is one of the most useful approaches to characterize boundary segregation with high (nanometer-level) spatial resolution. However, the quantified segregant composition is not always reliable, because the measurement is influenced by the specimen thickness, boundary inclination and other experimental conditions. The boundary excess coverage, which is less sensitive to the specimen and experimental conditions, is a better measure of segregation, but requires knowledge of the specimen thickness. In this study, the local thickness was determined via the ?ê-factor method, which simultaneously provides specimen composition and thickness at the analysis point, using X-ray mapping, and hence the boundary excess is determined locally. In addition, by applying an orientation imaging technique in a transmission electron microscope, the measured values of the local boundary excess can be linked to the crystallographic misorientation across the boundary, thus permitting unique structure-chemistry-property correlations.

4:10 PM
Coalescence of Two Particles with Different Sizes by Surface Diffusion: *Joachim H. Schneibel*[1]; Pavlo Sachenko[1]; Wen Zhang[2]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA; [2]Oakland University, Dept. of Math. & Stats., Rochester, MI 48309 USA

Focused ion beam milling was used to drill through polycrystalline 10 mm thick copper foil to machine disc-shaped particles with different sizes that were bonded to each other by a short grain boundary segment, i.e., they formed a neck. During subsequent annealing, sintering of the two particles, i.e., neck growth, was observed. Modeling of the sintering assuming only surface diffusion was in good agreement with the shape evolution observed during annealing. However, once the discs attained a shape close to equilibrium, the numerical computation had difficulty in proceeding further. It will be shown that two particles with different sizes exhibiting only surface diffusion cannot reach equilibrium, and this is the reason why the computation could not proceed. This work was sponsored by the National Science Foundation under grant DMR-9996087, and by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under Contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:30 PM
Effects of Diffusion Induced Recrystallization on Cu-Ni Interdiffusion: Stephen M. Schwarz[2]; Brian W. Kempshall[2]; *Lucille A. Giannuzzi*[1]; [1]FEI Company, One Corp. Way, Peabody, MA 01960-7990 USA; [2]Nanospective Inc., 12565 Rsch. Pkwy., Ste. 300, Orlando, FL 32826 USA

The interdiffusion of epitaxially electrodeposited Ni on single crystal Cu has been studied. Diffusion induced recrystallization (DIR) was observed to occur at the original Cu-Ni interface after diffusion annealing at temperatures between 500 to 650°C for 120 to 200 hours. FIB/TEM techniques were used to compare interdiffusion regions that exhibited DIR versus regions that did exhibit DIR. The DIR regions showed an increase in diffusion lengths and diffusivity by orders of magnitude compared to the non-DIR regions. Diffusivity values obtained for the non-DIR regions from this study are the lowest values for the Cu(Ni) system compared to known literature values, implying that previous values may contain grain boundary contributions. This work was made possible through the support of NSF DMR Award #9703281.

4:50 PM
On the Role of Grain Boundaries in Determining the Rate Process of Deformation of Materials at High Temperatures: *D. H. Sastry*[1]; [1]Indian Institute of Science, Dept. of Metall., Bangalore 560012 India

At temperatures greater than 0.5 on the homologous temperature scale, the deformation of materials is believed to be controlled by a lattice duffusion mechanism. However, in polycrystals, grain boundaries also play an important role and processes such as grain boundary sliding become dominant. In some cases the rate of grain boundary sliding could be the strain rate (creep rate) controlling step. To understand the exact behavior of grain boundaries, studies on single crystal must be compared with those on polycrystal material. This approach is often laborious. Fortunately, by utilising the 'impression creep' technique, polycrystalline material can be used to understand the deformation behavior of the single crystal also i.e., investigation with as well as without grain boundary influence can be carried out on the same material. The present study describes the results of impression creep testing on some metals at high temperatures. Thermal activation parameters such as activation energy and activation area are evaluated when the interior of grain only is deforming and a comparison is made with those obtained when the grain boundaries are also involved in the creep deformation. The results indicate that, under the experimental conditions employed, grain boundary sliding is not the rate controlling step in high temperature creep of the selected polycrystalline materials. However, it is established that the impression creep technique, applied to polycrystal samples of small size, is capable of giving information on single crystal as well as polycrystal deformation behavior.

Third International Symposium on Ultrafine Grained Materials: Superplasticity, Creep & Thermal Stability

Sponsored by: Materials Processing & Manufacturing Division, MPMD-Shaping and Forming Committee
Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; Terence G. Langdon, University of Southern California, Departments of Aerospace & Mechanical Engineering and Materials Engineering, Los Angeles, CA 90089-1453 USA; Terry C. Lowe, Metallicum, Santa Fe, NM 87501 USA; S. Lee Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright Patterson AFB, OH 45433 USA; Dong H. Shin, Hanyang University, Department of Metallurgy and Material Science, Ansan, Kyunggi-Do 425-791 Korea; Ruslan Z. Valiev, Institute of Physics of Advanced Material, Ufa State Aviation Technology University, Ufa 450000 Russia

Wednesday PM Room: 207A
March 17, 2004 Location: Charlotte Convention Center

Session Chairs: Terence G. Langdon, University of Southern California, Aeros. & Mech. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA; Amiya K. Mukherjee, University of California, Chem. Engrg. & Matls. Sci., Davis, CA 95616 USA; Sergey V. Dobatkin, Baikov Institute of Metallurgy & Materials Science, Moscow 119991 Russia

2:00 PM Invited
Superplasticty in Nanocrystalline Materials: *Amiya K. Mukherjee*[1]; Nathan A. Mara[1]; Alla V. Sergueeva[1]; [1]University of California, Chem. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA

High strain rate and/or low temperature superplasticity was observed in 1420 aluminum alloy and in Ni_3Al. The enhanced plasticity demonstrated in these materials is attributed to a refinement in grain size to the nano or near-nano range through the use of High Pressure

Torsion (HPT). Grain sizes after HPT were approximately 100 nm for the 1420 aluminum alloy, and 50 nm for Ni_3Al. Tensile testing was carried out at strain rates up to 10^{-1} and temperatures up to 725°C. Tensile curves typically exhibit high flow stresses and a degree of strain hardening that cannot be adequately described by conventional recovery mechanisms such as grain growth. Room-temperature TEM in-situ straining experiments reveal little dislocation activity and no evidence of dislocation pile-ups. It is suggested that the dominant deformation mechanism for elevated temperature plasticity in this study is grain boundary sliding and rotation. (NSF-DMR-0240144).

2:20 PM
High Strain Rate Superplasticity in a Spray-Cast Aluminum Alloy Processed by Equal-Channel Angular Pressing: *Cheng Xu*[1]; Terence G. Langdon[1]; [1]University of Southern California, Depts. of Aeros. & Mech. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

A spray-cast aluminum 7034 alloy, containing 11.5% Zn, 2.5% Mg, 0.9% Cu and 0.20% Zr, was processed by Equal-Channel Angular Pressing (ECAP) at 473 K to produce an ultrafine grain size of ~0.3 μm. The mechanical properties of the alloy were investigated using tensile testing at high temperatures over a range of strain rates. The results show that the as-pressed alloy yielded superplastic elongations of >1000% when testing at a temperature of 673 K with strain rates at and above $\sim 10^{-2}$ s^{-1}. The strain rate sensitivity was measured as ~0.33, thereby suggesting that dislocation glide may be an important deformation mechanism.

2:35 PM
Structure Formation During High Pressure Torsion and Heating of Low-Carbon Steels With Different Initial States: *Sergey Vladimir Dobatkin*[1]; Nikolay Alexander Krasilnikov[2]; Kazimir Iosiph Yanushkevich[3]; [1]Russian Academy of Science, Baikov Inst. of Metall. & Matl. Sci., Dept. of Phys.-Mech. Problems of Bulk Nanomatls., Leninky prospekt 49, Moscow 119991 Russia; [2]Ufa State Aviation Technical University, ul. K. Marksa, 12, Ufa 450000 Russia; [3]Institute of Solid State and Semiconductor Physics of the National Academy of Sciences of Belarus, Lab. of Physics of Magnetic Matls., P. Brovki str, Minsk 220072 Belarus

The present work was aimed to study the formation of UFG- (d < 100 nm) and submicrocrystalline (SMC) structure (100 nm < d < 1000 nm) in 0.12%C-0.85%Mn-0.65%Si and 0,1%C-1.12%Mn-0.08%V-0.07%Ti low carbon steels during severe cold and warm deformation in torsion under high pressure (HPT) and subsequent heating. Steels were either hot rolled or quenched prior to torsion. SPD of these steels at room temperature results in the emerging of nanoscale structure comprised of oriented cell-like fragments and fine grains. SPD of the initially quenched steels leads to a more dispersed nanoscale structure than that of initially hot rolled steels. Changing of microhardness, electrical resistance and structure during heating of SPD steels was studied. Heating on 500°C results in SMC structure with the grain size 300-500 nm.

2:50 PM
High Temperature Deformation Behavior of Ultra-Fine Grained Ti-6Al-4V Alloy: J. H. Kim[1]; Y. G. Ko[1]; S. Y. Han[2]; *C. S. Lee*[1]; D. H. Shin[2]; S. L. Semiatin[3]; [1]Pohang University Science and Technology, Dept. of Matls. Sci. & Engrg., San 31, Hyoja-dong, Namgu, Pohang, Gyeong-buk 790-784 S. Korea; [2]Hanyang University, Dept. of Metall. & Matls. Sci., Ansan, Gyeong-gi 425-791 S. Korea; [3]Wright-Patterson Air Force Base, AFRL, Wright-Patterson AFB, OH 45433-7817 USA

In this study, high temperature deformation behavior of ultra-fine grained Ti-6Al-4V alloy has been investigated on the basis of the inelastic deformation theory which consists of grain matrix deformation and grain boundary sliding. Microstructure of coarse equiaxed grains (~11 micrometer in diameter) has been significantly refined to 0.3 micrometer with high angle boundaries after 4 times of isothermal equal channel angular (ECA) pressing at 873K. Load relaxation test has been performed at the temperature range of 773K~973K to enlighten the deformation mechanisms operating at specific temperatures and to find optimum superplastic deformation conditions for ultra-fine grained microstructures. Also, main efforts have been devoted to analyze quantitatively the relative amount of each deformation mode, i.e., dislocation glide, dislocation climb and grain boundary sliding operating at specific temperatures. Tensile test of ultra-fine grained specimens has also been carried out to verify the optimum superplastic conditions.

3:05 PM
The Mechanical Properties and Microstructure of Equal Channel Angular Pressed Al-Cu-Mg Alloy Fabricated by Spray Deposition: *Kyung H. Chung*[1]; Dong H. Shin[2]; Enrique J. Lavernia[1]; [1]University of California, Chem. Engrg. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Hanyang University, Metall. & Matls. Engrg., Ansan, Kyunggi-do 425-791 Korea

The porous spray deposited Al-4.4Cu-0.8Mg alloy was equal channel angular pressed (ECAPed) to produce fine-grained bulk material without the limitation over cross-sectional dimensions. The ECAP of the spray deposited Al-Cu-Mg alloy is successfully performed at 200°C. After a single pass, the density becomes full and the remaining pore size is reduced to less than 3μm from about 20μm. With repeated ECAP passes, the microstructure becomes more equiaxial and homogeneous. The grain size reduces to the 100 ~ 250 nm range after four passes. After ECAP, the room temperature strength is increased up to 110%, and that is attributed to the reduced amount of pore, the increased dislocation density and the reduced grain size. Additionally, the static heat treatments were applied at the spray deposited and ECAPed Al-Cu-Mg alloy. The gging condition will be optimized and the microstructural characteristics will be investigated with focusing on the precipitation behavior. The grain stability also will be examined during the aging process and the relationship between the microstructural features and mechanical properties will be extensively investigated.

3:20 PM
Grain Refinement Mechanisms with Large-Strain Deformation of Zirconium: *M. E. Kassner*[1]; S. A. Barrabes[1]; M.-T. Perez-Prado[2]; [1]University of California, Mech. & Aeros. Engrg., 9500 Gilman Dr., EBU II, La Jolla, CA 92093-0411 USA; [2]CENIM-CSIC, Madrid Spain

Large strain deformation of pure zirconium at elevated temperature in both tension and torsion was studied. POM, TEM and EBSP reveal substantial grain refinement with large strain deformation. The purpose of this work was to address the dynamic restoration and grain-refinement mechanisms in pure _lpha-zirconium deformed to large strains at 600-800°C. The restoration mechanisms examined included dynamic recovery, grain growth, and various dynamic recrystallization mechanisms. Specifically, the presence of dynamic recrystallization during high temperature mechanical testing (400°C to 750°C) was assessed. This analysis also assisted with the interpretation of the activation energies for creep-plasticity.

3:35 PM
Grain Growth Inhibition in Nanocrystalline Al-Sc Alloys: N. Burhan[1]; *M. Ferry*[1]; [1]University of New South Wales, Sch. of Matls. Sci. & Engrg., Sydney, NSW 2052 Australia

An ultrafine-grained microstructure in a range of Al-Sc alloys was produced by Equal Channel Angular Extrusion (ECAE). The alloys were solution treated prior to deformation, deformed by ECAE then aged at low temperature to produce a sub-micron grained microstructure with a high fraction of high angle grain boundaries (HAGB) decorated with nanosized Al_3Sc particles. General grain stability and particle/grain boundary interactions were studied using electron backscatter diffraction (EBSD) in the scanning electron microscope and transmission electron microscopy. The fine-grained microstructures were found to be highly stable during annealing at elevated temperatures due to Zener pinning from the fine Al_3Sc particles. The volume fraction and size of fine particles and their rate of coarsening were found to have a strong influence on grain growth. The grain stability in this alloy system was compared with recent models of grain coarsening in particle-containing materials.

3:50 PM Break

4:00 PM
Microstructural Evolution During Superplastic Deformation in ECA Pressed Al-Mg-Sc Alloys: *Minoru Furukawa*[1]; Kazuko Furuno[2]; Keiichiro Oh-ishi[2]; Zenji Horita[2]; Terence G. Langdon[3]; [1]Fukuoka University of Education, Dept. of Tech., 1-1, Akama-Bunkyomachi, Munakata, Fukuoka 811-4192 Japan; [2]Kyushu University, Dept. of Matls. Sci. & Engrg., Fukuoka 812-8581 Japan; [3]University of Southern California, Depts. of Aeros. & Mech. Engrg. & Matls. Sci., Los Angeles, CA 90089-1453 USA

ECAP was conducted to achieve grain refinement in Al-0.5%Mg-0.2%Sc and Al-1%Mg-0.2%Sc alloys. The ECAP was performed using dies having internal channel angles of either 60 or 90° where these angles produce equivalent strains of 1.6 and 1.1 for each pass, respectively. Tensile testing at 673K of both alloys showed the elongations to failure increase with increasing equivalent strain. When pressing the Al-0.5%Mg-0.2%Sc and Al-1%Mg-0.2%Sc alloys for 6 passes using a 60° die, the elongations to failure were 340% and 800% at 673K, respectively. Observations using EBSP revealed that, in the as-pressed condition, both alloys had textures and a high fraction of low-angle boundaries. Whereas the texture and a high fraction of low-angle boundaries remained after failure at 340% in the Al-0.5%Mg-0.2%Sc alloy,

the texture was removed and there was a large fraction of high-angle boundaries after failure at 800% in the Al-1%Mg-0.2%Sc alloy.

4:15 PM
Neutron Diffraction Study of Super-Plastic Al-Alloy Processed by ECAP: *Sven C. Vogel*[1]; David J. Alexander[2]; Irene J. Beyerlein[3]; Mark A.M. Bourke[1]; Donald W. Brown[1]; Bjorn Clausen[1]; Saiyi Li[1]; [1]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-6, MS G770, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, T-3, MS B216, Los Alamos, NM 87545 USA

The use of severe plastic deformation techniques such as equal channel angular pressing, (ECAP) has been shown to refine metal microstructures giving advantageous mechanical properties. In this presentation, we will report results of in-situ neutron diffraction loading measurements on the neutron spectrometer SMARTS at LANSCE performed on an Al-Mg-Sc alloy processed by ECAP. This material was reported to exhibit super-plasticity when deformed in tension at elevated temperatures. In-situ loading experiments provide, among other information, crystal lattice-plane dependent stress-strain curves allowing to understand and predict the macroscopic material performance. Using SMARTS, tensile testing at elevated temperatures is possible simultaneously with the neutron diffraction, providing unique insights into the deformation behavior of the probed material. This experiment is the first application of this technique to a material exhibiting super-plasticity. We will report results from tensile tests of pure Al as a reference material and the Al-Mg-Sc alloy both at room-temperature and at temperatures for which the alloy was reported to exhibit super-plasticity.

4:30 PM
Influence of Processing Route on Creep of Ultrafine Grained Aluminium Prepared by ECAP: *Vaclav Sklenicka*[1]; [1]Academy of Sciences of the Czech Republic, Inst. of Physics of Matls., Zizkova 22, Brno CZ-616 62 Czech Republic

Extremely coarse-grained high-purity aluminum was subjected to equal-channel angular pressing (ECAP) and one or repetitive pressing followed either route A,B or C. Creep tests were conducted at 473 K on billets after one pass and after repetitive pressing (up to 12 ECAP passes). In essence, the creep resistance of an ultrafine grained aluminum after ECAP pressing is shown to be considerably improved to an unpressed material. The minimum creep rate for the ECAP material is about one to two orders of magnitude less than that of coarse-grained material. Further, the ECAP material exhibits markedly longer creep life than unpressed material but the creep life of ECAP material decreases with increasing number of ECAP passes. It is suggested that the effect of processing route on creep behaviour may be explained by homogenization of the microstructure and microtexture of the ECAP material.

4:45 PM
Indentation Behavior of Aluminum Prepared by Equal Channel Angular Extrusion: *Fuqian Yang*[1]; Lingling Peng[1]; Kenji Okazaki[1]; [1]University of Kentucky, Chem. & Matls. Engrg., 161 Anderson Hall, Lexington, KY 40506 USA

Equal channel angular extrusion (ECAE) as one of severe plastic deformation methods to produce ultra-fine structure in bulk samples and billets has become a relatively active area. The as-deformed material by the ECAE process is in a non-equilibrium state and displays unique properties, such as high hardening and superplastic behavior. The present work examines the plastic deformation of as-ECAE Al by using indentation technique. The indentation load was found to be proportional to the 3/2 power of the maximum indentation depth. The as-ECAE Al displayed different unloading behavior from the annealed Al due to the large plastic deformation introduced in the ECAE process, which suggests the change of microstructure. A relation between the plastic energy dissipated in the indentation and the indentation load was derived by using dislocation theory - the plastic energy is proportional to the 3/2 power of the indentation load, which was supported by the experimental results.

5:00 PM Award Ceremony

5:15 PM Panel Discussions

Notes

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