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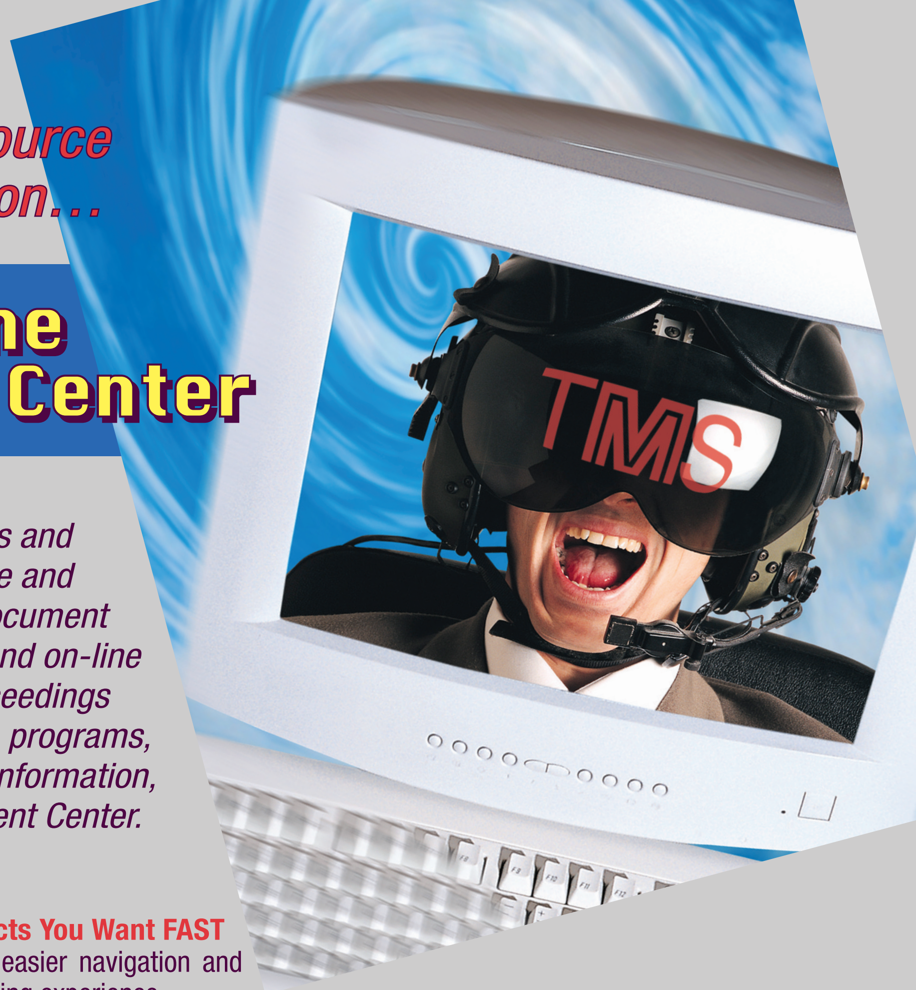

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  Gather individual papers and articles from TMS proceedings volumes, *JOM*, *Journal of Electronic Materials*, and *Metallurgical and Materials Transactions A* and *B* to create a one-of-a-kind publication that meets your needs. TMS will compile them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

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http://doc.tms.org

TMS2003

132nd Annual International Meeting & Exhibition

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

This document comprises

THURSDAY'S TECHNICAL PROGRAM

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

For your convenience, we have also included details on

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

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

See you in San Diego!

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

AN INTERNATIONAL EVENT IN SCIENCE AND ENGINEERING

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

This year's meeting will feature programming by

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

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

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

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

WANT TO BE PART OF THE ACTION?

Registration is easy.

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

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

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

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

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

INTERESTED IN BUSINESS OPPORTUNITIES?

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

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

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

CONFERENCE PROCEEDINGS: THE RECORDS OF EVENTS

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

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

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

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

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

The following symposia will be documented in upcoming TMS periodicals.

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

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

ADDITIONAL RESOURCES

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

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

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

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

TMS2003

ADVANCE REGISTRATION FORM

Advance Registration Deadline: February 3, 2003

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

AM03-PDF

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

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

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

1. ☐ Dr. ☐ Prof. ☐ Mr. ☐ Mrs. ☐ Ms.

Member of: ☐ TMS ☐ ISS ☐ SME ☐ SPE   Member Number: ____________

Last Name ____________ First Name ____________ Middle Initial ____

Employer/Affiliation: ____________ Title: ____________

Address: ☐ Business ☐ Home ____________

City: ____________ State/Province: ____________ Zip/Postal Code: ____________ Country: ____________

Telephone: ____________ Fax: ____________ E-Mail: ____________

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

2. Registration Fees:

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

Registration TOTAL $ ________

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

3. Social Function Tickets:

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

Social Function TOTAL $ ________

4. Tutorial Luncheon Tickets:

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

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

Order Number	Title	Shipping Weight	Quantity	SubTotal Weight	Member Price	Non-member Price	Sub-Total Price
03-5352	Electron Microscopy (Meshii)	3	______	______	$96	$137	$ ______
03-5344	Metallurgical & Materials Processing (Yazawa)—Three-volume Set	11	______	______	$337	$538	$ ______
03-5468	Vol. 1: Fundamentals & New Technologies *(only)*	4	______	______	$154	$220	$ ______
03-5476	Vol. 2: High-Temp. Metal Production *(only)*	4	______	______	$154	$220	$ ______
03-5484	Vol. 3: Aqueous & Electrochemical Process *(only)*	3	______	______	$112	$159	$ ______
03-5328	EPD Congress 2003	3	______	______	$87	$123	$ ______
03-531X-G	Light Metals 2003 *(book and CD set)*	7	______	______	$158	$226	$ ______
03-5360	Friction Stir Welding and Processing II	3	______	______	$110	$157	$ ______
03-5336-G	Magnesium Technology 2003 *(book and CD set)*	3	______	______	$113	$161	$ ______
03-5395	High Temperature Alloys	2	______	______	$75	$107	$ ______
03-5387	Hot Deformation of Aluminum Alloys 2003	4	______	______	$123	$175	$ ______
03-5441	Materials Lifetime Science & Engineering	2	______	______	$91	$130	$ ______
03-5379	Surface Engineering in Materials Science II	2	______	______	$124	$177	$ ______
03-5522	Energy Efficient Manufacturing Processes (4th MPMD Global Symp.)	2	______	______	$65	$92	$ ______

SubTotal $ ________

WEIGHT AND ZONE CHART

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

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

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

$5 handling fee per order shipped. $ ________

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

Publications TOTAL $ ________

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

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

Short Course TOTAL $ ______ $ ______

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

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

8. Payment enclosed:

☐ Check, Bank Draft, Money Order

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

☐ Credit Card Expiration Date: ____________

Card No.: ____________

☐ Visa ☐ MasterCard ☐ Diners Club ☐ American Express

Cardholder Name: ____________

Signature: ____________

9. TOTAL FEES PAID $__________

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

TMS2003

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

HOUSING RESERVATION FORM

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

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

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

Arrival Date ________________ Departure Date ________________

Last Name ________________ First Name ________________ MI ________

Company ________________

Street ________________ Address ________________

City ________________ State/County ________ Zip/Postal Code ________ Country ________

Daytime Phone ________________ Fax ________________

Additional Room Occupants ________________

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

Non-Smoking Room Requested ________________ Special Needs ________________

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

1. ________________
2. ________________
3. ________________

Type of Accomodations: (check one)

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

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

HEADQUARTERS

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

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

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

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

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

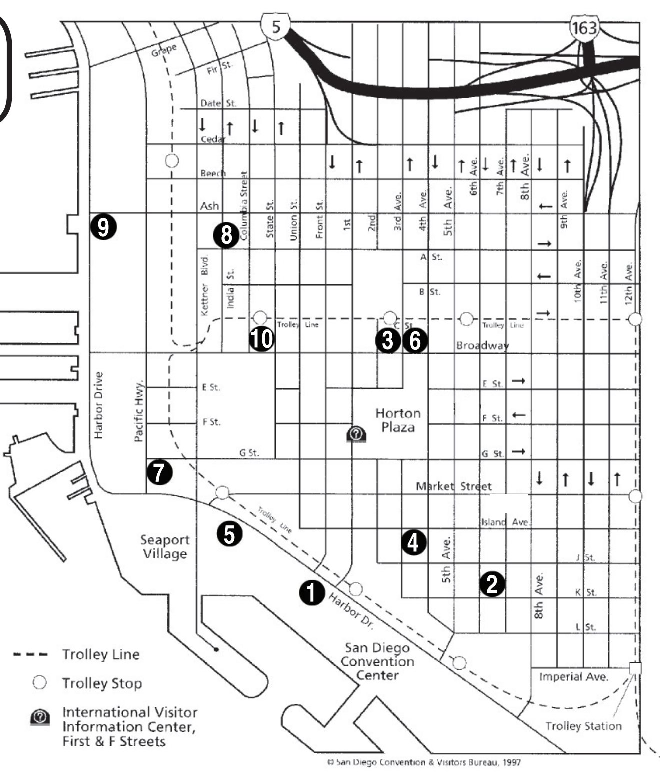
6. **U.S. Grant Hotel**
$179/single • $179/double

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

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

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

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



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

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

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

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

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

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

Account Number ________________ Expiration Date ________________

Card Holder Name ________________ Authorized Signature ________________

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

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

Thursday AM Room: 10
March 6, 2003 Location: San Diego Convention Center

Session Chair: Reza Abbaschian, University of Florida, Matls. Sci. & Eng., Gainesville, FL 32611-6400 USA

8:30 AM
Ampoule Design Experimentation for Microgravity Processing of Metal Alloys: *D. A. Downs*[1]; J. B. Andrews[1]; [1]University of Alabama-Birmingham, Microgravity Solidification Lab., Dept. of Matls. Sci. & Eng., Rm. 254 BEC, 1150 10th Ave. S., Birmingham, AL 35294 USA

The Coupled Growth in Hypermonotectics (CGH) experiment involves the directional solidification of aluminum-indium alloys and is designed to investigate the fundamental mechanisms of monotectic solidification. Initial experiments carried out during the STS-78 Life and Microgravity Spacelab (LMS) Mission used specially designed aluminum nitride ampoule assemblies. The specialized design of these ampoules is critical to obtaining correct, consistent, processing conditions. Past and current ampoule development efforts have highlighted ampoule design features that promote desirable performance as well as features that proved troublesome. The results of continued furnace testing of different ampoule configurations will be presented.

8:50 AM
New Optical Method for Measuring Densities of Containerless Processed Liquid Metals: *J¸rgen Brillo*[1]; Ivan Egry[1]; [1]Deutsches Zentrum f¸r Luft-und Raumfahrt e.V. (DLR), Inst. f¸r Raumsimulation, Linder Hˆhe, Kˆln-Porz D-51174 Germany

We have developed a new experimental technique for density determination of electromagnetically levitated metallic liquids. This method employs an enlarged beam of parallel laser light to produce a shadow image of the sample. The shadow is recorded by a digital CCD-camera and the images are analysed using an edge detection algorithm. The circumference is fitted by Legendre polynomials which allows to calculate the volume. The advantage of this procedure compared to those techniques recording the sample image directly is that interferences caused by the samples movement are being avoided. Hence there is no change of the apparent size of the sample. In addition, effects that might occur from the sampleís radiation are suppressed as there is a constant contrast between the shadow and the background. A detailed description will be given and first results on measurements of the Ni-Cu- Fe- system will be presented.

9:10 AM
Thermophysical Property Measurements of Molten Metals and Ceramics by Electrostatic Levitation: *Takehiko Ishikawa*[1]; Paul-Francois Paradis[1]; Jianding Yu[1]; Tomotsugu Aoyama[1]; Shinichi Yoda[1]; [1]National Space Development Agency of Japan, 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan

Thermophysical property measurements, in particular density, vapor pressure, surface tension, and viscosity, proved to be very challenging by means of electrostatic levitation when dealing with refractory metals (e.g. Mo, Ta), high vapor pressure metals (e.g. Ti, Mo), and ceramics (e.g. oxides). The main issues to be addressed are the levitation initiation, the time required to achieve a molten state of a sample, the high luminosity of the droplet, and, sometimes, severe evaporation. This paper briefly summarizes the procedures and methods adopted by the National Space Development Agency of Japan with an electrostatic levitator furnace to enable thermophysical property measurements of the aforementioned materials. Typical data that would have been impossible to obtain without the use of these techniques and procedures, namely the density of oxides (e.g. $BiFeO_3$, YAG), the vapor pressure of Ti, and the surface tension and viscosity of Mo are reported for superheated and undercooled phases.

9:30 AM
Accurate Thermophysical Property Measurements of Liquid Si by Electrostatic Levitation: *Zhenhua Zhou*[1]; Won-Kyu Rhim[1]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., Pasadena, CA 91125 USA

Several improvements have been done with the high vacuum electrostatic levitator in Caltech. Four laser beams with the tetrahedral arrangement heating technology was developed to greatly reduce the temperature gradient inside the levitated sample. A new image processing technology was adopted to get precise sample volumes. The DOAP (Division-of-amplitude photopolarimeter) was installed and used to measure the spectral emissivity of the liquid Si down to the highly undercooled region. The spectral emissivity data together with the single color pyrometer can give the accurate temperature of the levitated Si sample. Based on these new improvements, accurate temperature dependent thermophysical properties such as density, thermal expansion coefficient, spectral emissivity, specific heat over total hemispherical emissivity, viscosity and surface tension of liquid Si can be obtained. The contactless measurements and the uniform temperature distribution inside the levitated sample together with the accurate temperature measurement will result in more reliable data than before.

9:50 AM
A Passive Thermal Carrier (Lotec) for Temperature Sensitive Materials Being Moved to and from the International Space Station and for Shuttle (Sts) Flights: *F. C. Wessling*[1]; J. M. Blackwood[1]; H. R. Holt[1]; [1]The University of Alabama-Huntsville, Dept. of Mechl. & Aeros. Eng., Huntsville, AL 35899 USA

A Low Temperature, low Energy Carrier (LoTEC) maintains steady state temperatures without the use of power for transporting temperature sensitive materials to and from the International Space Station. LoTEC uses a combination of high thermal resistance insulation, careful thermal design and phase change materials to maintain temperature. It has been tested with phase change materials for three different interior temperatures (-16C, 0C, +4C). These temperatures are usable for preservation of many biological materials during transport. The exterior dimensions of LoTEC are approximately 250.7 x 436.9 x 514.1 mm. Thus, LoTEC fits into a standard mid-deck locker, an Express Rack, a SpaceHab rack or a rack in the Multi-Purpose Logistics Module (MPLM). LoTEC has an empty mass of approximately 9.6 kg and an internal volume of 22 Liters. This volume is reduced by the amount and type of phase change material (PCM) used. For example, eight kg of water ice and its containers would take up approximately half of the internal volume, leaving 11 Liters of space for the payload. Several different configurations have been considered to accommodate different payload dimensions. The interior dimensions of LoTEC are approximately 406 x 343 x 158 mm. Measured energy gain by LoTEC is approximately 0.14 watts per degrees Celsius. Performance data in the form of temperature versus time curves are included. Eight kg of PCM gives approximately eight to ten days of constant temperature at typical STS interior temperatures. The temperature distribution in LoTEC is dependent on the PCM containers, the PCM used, and the location of the containers in LoTEC. Small, stand-alone data loggers record the temperatures.

10:10 AM Break

10:30 AM
Development Status of Electrostatic Levitation Furnace(ELF) for Kibo: Keiji Murakami[2]; Takehiko Ishikawa[2]; Kazunori Kawasaki[1]; Shoji Muramatsu[1]; Hiroaki Asahi[1]; *Takahiro Nishimura*[1]; [1]IHI Aerospace Company, Ltd., Space Sys. Dept., 900 Fujiki, Tomioka, Gunma 370-2307 Japan; [2]National Space Development Agency of Japan, Space Utilization Rsrch. Ctr., 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505 Japan

The development status of Electrostatic Levitation Furnace (ELF) system for KIBO is introduced. Several new functions and properties are studied. (1) Small and Vacuum Chamber: It succeed in the miniaturization of chamber by adoption of small components and the device of a layout. The chamber will provide the pressurized condition as well as the very high level vacuum condition. (2) Tetrahedral Laser Heating: Four lasers are placed as tetrahedral configuration to accomplish uniform heating and sample rotation suppression. (3) Cage Exchangeability: In order to meet the various user requirements and refreshing electrodes, the chamber consists of the duplex structure made from the chamber itself and the exchangeable sample cage. (4) High-speed Sample Position Sensing: High-speed sample position sensing system makes

1kHz (maximum) interval sample position control is provided. The parabolic flight test and some ground tests using the Bread Board Model have been performed in the current development phase.

10:50 AM
ATENñA Multi-Purpose Furnace for Material Science Research on the International Space Station: *C. Casgrain*[1]; L. Misener[2]; [1]Canadian Space Agency, St-Hubert, Quebec J3Y 8Y9 Canada; [2]Millenium Biologix Inc., Kingston, Ontario K7M 7G3 Canada

Canada is a partner of the International space station (ISS), providing the Mobile servicing system, a sophisticated robotic arm used to assemble and maintain the station. In exchange for its contribution the Canadian scientists will have the right to use 2.3% of the non-Russian portion of the International Space Station for research over the fifteen years of its expected life. For the Microgravity Science Program (MSP) and its scientific community, this includes the continuous use of one double middeck locker. One important mandate of MSP is to develop state-of-the-art payloads to support world class research as well as a facility to improve the microgravity environment quality. An effective use of MSPís double locker on ISS is to devote the bottom part to the isolation system against vibrations (MIM Base Unit) and the top part for small exchangeable payloads. We are presently developing the fourth generation of the CSAís locker-sized furnace payload called ATEN (Advanced Thermal ENvironment). ATEN will be the first MSP payload to fly on the MIM Base unit. The ATEN project started in February 1999 with a survey of the scientific community and four feasibility studies. In January 2001, Millenium Biologix Inc in Kingston Ontario was selected to design and build the ATEN furnace in a two-year timeframe. The ATEN furnace is designed to meet a wide range of scientific requirements. It will allow investigators to do fundamental studies in diffusion, Ostwald ripening and particle pushing as well as improving material processing techniques to grow semi-conductors, ceramics and glasses of better quality. Therefore ATEN will provide different modes of operation such as isothermal, gradient heating and melt zone heating. ATEN will be able to process specimens up to 10 mm in diameter and 80 mm in length. The furnace will operate in isothermal mode at temperatures varying from 100∫C to 1300∫C and in temperature gradient mode with a gradient varying from 5∫C/cm to 50∫C/cm. The facility will also provide melt zone mode. The temperature accuracy will be within ±0.25% at high temperature with a repeatability of ±1∫C. One of the keys to successful experiments in space is extensive ground preparation. The great advantage of ATEN is that it is a compact payload and Ground Units (GU) will be made available to scientists for mission preparation and experiment fine-tuning. As mentioned, ATEN will fit into the Microgravity vibration Isolation Mount Base Unit (MIM Base Unit) enclosure. The MIM Base Unit will provide all necessary mechanical and electrical interfaces, power, cooling, data processing capabilities, data storage and communication interface for tele-operation in addition to providing isolation from vibrations. In December 2001, ATEN successfully underwent its Preliminary Design Review (PDR) at CSA headquarters in St-Hubert. The CDR is planned for November 2002 and the final delivery is planned for the summer of 2003 while the first flight opportunity is foreseen in 2005. The aim of the present paper is to present the ATEN payload technical features as well as the major challenges undertaken during the design of the payload.

11:10 AM Cancelled
German Facility Developments for Microgravity Materials Sciences: *Rainer Kuhl*[1]; Horst Binnenbruck[1]; [1]German Aerospace Center, Space Mgmt., DLR, Microgravity Rsrch. & Life Scis., Bonn 53227 Germany

11:30 AM
Determination of Nucleation Kinetic Parameters of Metallic Melts Using Electrostatic Levitation Techniques: Melissa J. Wert[1]; *William H. Hofmeister*[1]; Robert J. Bayuzick[1]; Jan Rogers[2]; Thomas Rathz[2]; Glenn Fountain[2]; Robert Hyers[3]; [1]Vanderbilt University, Cheml. Eng., Box 1604, Sta. B, Nashville, TN 37235 USA; [2]NASA, Marshall Space Flight Ctr., MC SD47, Huntsville, AL 35812 USA; [3]University of Massachusetts, Dept. of Mechl. & Indl. Eng., Amherst, MA 01002 USA

Electrostatic levitation is an elegant way of processing metals in a high vacuum environment and is extremely useful in obtaining undercooling data required to determine nucleation kinetic parameters of metallic melts. Bulk zirconium samples (~2 mm diameter) of varying oxygen content are levitated at vacuum levels in the range of 1 x 10-8 Torr, minimizing contamination by container walls or residual gasses in the system. Numerous undercooling cycles (>100) are obtained for each sample, generating an undercooling distribution representative of the sampleís kinetic behavior. A statistical analysis is performed on the undercooling distributions of each sample, resulting in values for the kinetic prefactor, K_v, and work of formation of a critical nucleus, ΔG^*, using the Spaepen-Turnbull nucleation rate equation. Glow discharge mass spectrometry is used to determine oxygen content of each sample. Nucleation kinetic parameters are found to increase with increasing sample purity.

11:50 AM
Preparation of Al-Li Alloy with Electric Magnetic Force Simulation Facility: *Da Daoan*[1]; Hao Weixin[1]; Geng Gruihong[1]; [1]Lanzhou Institute of Physics, Gansu 730000 China

By adopting the usual smelting technology, serious segregation will occur when the content of Li exceed 3%. We discovered that the impact of gravity can be counteracted by Lorentz force generated by the electric magnetic force simulation facility. The contents of Li in the Al-Li alloy can be increased to 10% with density of 2.0g/cm, of which the non-uniformity of Li and Al element distribution at lower than 3%. The hardness measurement result of Al-Li alloy with 7% Li content shows that Vickers hardness can reach to 103.9.

Aluminum Reduction Technology: Pot Control

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

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

Session Chair: Gary Tarcy, Alcoa Inc., Hall Process Improv. Alcoa Techl. Ctr., Alcoa Ctr., PA 15069-0001 USA

8:30 AM
Aluminum Fluoride Control Strategy Improvement: Abdelhamid Meghlaoui[1]; *Najeeba Hassan Aljabri*[1]; [1]Dubai Aluminum Company, Ltd., Process Control & Dvlps., Jabel Ali, Dubai UAE

The AlF_3 is an important control parameter that has a considerable impact on the cell performance. An AlF_3 control strategy is developed on the basis of Entner model using the fuzzy logic to predict the daily AlF_3 concentration required for calculating the AlF_3 additions. Entner model is based on the AlF_3 dynamic reactive mass balance taking in consideration the time lag reaction. The main model input-parameters are the AlF_3 concentration in the bath and the historical additions. Because of economical considerations, the bath analysis is not performed daily but estimated from bath temperature daily measurements using fuzzy logic. This paper presents the control algorithm and the methodology to select adequately the model input-parameters as well as the fuzzy logic membership functions using a plant superheat data. Results from different cell technology show that fuzzy Entner based method improves dramatically the cell performance compared to the plant existing control strategy.

8:55 AM
Reduction Cell Noise Analysis: *Larry Banta*[1]; Congxia Dai[1]; [1]West Virginia University, Mechl. & Aeros. Eng., PO Box 6106, Morgantown, WV 26506 USA

In most reduction cell control schemes, anode position is a compromise between maintaining the lowest possible cell voltage and prevention of unacceptable levels of ìnoiseî. Exactly what constitutes noise varies from controller to controller, however, and there are many metrics used to quantify the noise level in a pot. It has long been known that cell voltage noise has a variety of sources, and at least some cell controllers make no attempt to distinguish one type of noise from another in determining control actions. This paper describes work underway at WVU to discriminate different noise sources by analysis of the types of noise signals they generate, and to suggest appropriate control strategies based on noise type as well as noise levels.

9:20 AM
Neural Network Qualifier of Noises of Aluminium Reduction Cells: Alexander I. Berezin[1]; *Peter V. Poliakov*[2]; Oleg O. Rodnov[1]; Igor V. Mezhubovsky[1]; Igor V. Gonebnyy[1]; [1]Mayak PKF, Ltd., Bograda st. 108, Krasnoyarsk 660021 Russia; [2]STC ìLight Metalsî, Vavilova st. 60, Krasnoyarsk 660025 Russia

The raw voltage signals from the aluminium reduction cells contain fluctuations, named in industrial practice by ìnoiseî, which can be used to identify problems with the cell operation. It is known, that each type of technology disturbances is characterized by unique noise. We

THURSDAY AM

have developed the Neural Network Qualifier of noises of aluminium reduction cells for use in control systems. Qualifier allows to distinguish noises caused by technology disturbances, and, thus, to identify them at an early stage of occurrence. Before the beginning of work the Qualifier is training for adaptation to noises of a concrete cell designs. For this purpose the network studied the images of noises, which were registered on cells with known disturbances. Trained Qualifier is used for the analysis of noises on working cells. Seeing similarity between known examples and real images of noise, the network identifies 6 technological ìillnessesî of cells.

9:45 AM
Analysis and Correction of Heat Balance Issues in Aluminum Reduction Cells: *Philip Biedler*[1]; Larry Banta[1]; [1]West Virginia University, PO Box 6106, Morgantown, WV 26506 USA

The heat balance in an aluminum reduction cell is a complex process composed of interrelated variables including power input, bath composition, bath and metal pad depth, cover depth, material properties and cell geometry. Because the process is dynamic, cells often drift from the targeted heat balance. When this happens, actions must be taken to correct the problem. However, the most appropriate action and the consequences of that action are not always known. To address these issues, a 2-D finite element model was developed to model the thermal, electrical, and chemical processes occurring in the cell. This model was used to study the interrelation of the variables affecting the heat balance and to analyze the effect of corrective actions taken to rectify several heat balance issues.

10:10 AM Break

10:20 AM
Fully Distributed Computer Control System for Aluminum Electrolysis: *Fengqi Ding*[1]; *Jie Li*[1]; Yinggang Jiang[1]; Wengen Zhang[1]; Zhong Zou[1]; Yexiang Liu[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

A fully distributed computer control system for aluminum electrolysis was developed. In the system, the supervision level made up of workstations equipped in the computer station and workshop sections and the direct control level consisting of cell controllers were connected with CAN bus to form a control network, with every workstation being able to supervise the cell controllers in its permitted supervision range; on the other hand, every workstation on the supervision level was linked into the plant LAN to meet the needs of setting up plant MIS. The cell controller was also designed in the form of network structure in which several intelligentized modules were connected with inner CAN bus to realize their data exchange and multi-CPU cooperation. Till now about 2000 cell controllers of this kind have been successfully applied at more than 20 aluminum plants in China.

10:45 AM
Increased Current Efficiency and Reduced Energy Consumption at the TRIMET Smelter Essen Using 9 Box Matrix Control: *Torsten Rieck*[1]; Martin Iffert[1]; Peter White[2]; Renuka Rodrigo[3]; Rik Kelchtermans[2]; [1]Trimet Aluminium AG, Aluminiumallee 1, Essen D-45356 Germany; [2]Heraeus Electro-Nite International NV, Centrum-Zuid 1105, Houthalen B-3530 Belgium; [3]Heraeus Electro-Nite Company, 9901 Blue Grass Rd., Philadelphia, PA 19114 USA

Energy consumption is one of the key issues in the production of primary aluminium. It is therefore a great challenge for aluminium smelters worldwide to increase current efficiency and at the same time reduce energy consumption. At the Essen Smelter a new type of energy and mass balance control algorithm has been developed and successfully implemented on an industrial scale. Central to this new strategy are superheat measurements. Supporting measurements include bath temperature and cathode voltage drop. Measurement data are input into a newly developed 9 box matrix control algorithm. The concept utilizes pot voltage adjustments as the primary control tool to drive the bath temperature into a predefined control band. As a secondary control, aluminium fluoride additions are based upon the liquidus trend. Through combined stabilization of energy and mass balance a stable cell operation is achieved, leading to the benefits of higher current efficiency and reduced energy consumption.

11:10 AM
Estimation of a Technological Condition of the Aluminium Reduction Cells on the Basis of its Daily Energy Balance: Oleg O. Rodnov[1]; *Peter V. Poliakov*[2]; Alexander I. Berezin[1]; Pavel D. Stont[1]; Igor V. Mezhubovsky[1]; [1]Mayak PKF, Ltd., Bograda st. 108, Krasnoyarsk 660021 Russia; [2]STC ìLight Metalsî, Vavilova st. 60, Krasnoyarsk 660025 Russia

It is necessary for smelters to have expert systems of analysis, which will permit to conduct deep evaluation of the technology of aluminium electrolysis with the purpose to determine the reasons, which provoke technology disturbances, to investigate and optimize operations. We offer methodology to analyze of the statistical data of electrolysis parameters permitting to determine daily an energy balance of the cell. The technique establishes interrelation of technological parameters of electrolysis by means of the introducing of new technological criterion, specially designed for the solution of put problems. In activity is submitted the conclusion of criterions describing interrelation of technological parameters of electrolysis and permitting to identification ìhealthî and ìillî of the cell. The conclusion of technological criterions bases on a theory of similarity and dynamical model of the aluminium reduction process.

11:35 AM
A Supervised System for Aluminum Reduction Cell: *Zeng Shuiping*[1]; [1]North China University of Technology, Inst. of Automatic Control, Jiyanzhang Rd. 5, Shijingshan Dist., Beijing 100041 China

This paper studied a online supervised system for aluminum reduction cell, which include the sample of data related with the process, the real-time display of some operation parameters and technical remarks, the control command for the process, alarm of abnormal condition and production management. This system can also analyze the operation techniques, judge the system conditions and give out the evaluation for the present status. The system is made with the tool of Visual Basic and the tool of Structured Query Language in database control. With the open architecture and the existing interface of I/O, the software can realize the special tasks required by individual operator, which performed well in actual production.

Cast Shop Technology: Foundry Technology II

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

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

Session Chairs: Daryoush Emadi, National Research Council of Canada, Matls. Techl. Lab., Ottawa, ON K2B 8L4 Canada; John Grandfield, CSIRO Australia, Preston, Victoria 3072 Australia

8:30 AM Cancelled
Thermal Two-Phase Finite Element Flow Model for Materials in the Semi-Solid State: *FrÈdÈric Pineau*[1]; [1]National Research Council Canada, Process Modlg. & Instrumentation, 75 de Mortagne Blvd., Boucherville, QuÈbec J4B 6Y4 Canada

8:55 AM
Hydrogen Gas Pick-Up of Al-Alloy Melt During Lost Foam Casting: *Seung-ryoul Shin*[1]; Zin-hyoung Lee[1]; [1]KAIST, Matls. Sci. & Eng., 373-1, Kusung-dong, Taejon, Yusung-gu 305-701 Korea

The hydrogen gas pick-up problem that can occur during LFC (Lost Foam Casting) was investigated. The gas content of the melt was measured by reduced pressure test and the proper test pressure was determined. The hydrogen pick-up increased with the increased amount of polystyrene that was replaced by melt. The hydrogen pick-up depended on the initial hydrogen content of the melt and the contact time of the melt with the decomposed gas phase. The effects of evacuating the chamber containing the mold, pouring system and coating thickness were investigated on hydrogen concentration and flow length of melt. Also the solidification of the melt and the gas formation were monitored using a high speed camera through the quartz window mount on the mold during the process.

9:20 AM Cancelled
Vacuum Die-Casting of Wrought 7050 & 7150 Aluminum Alloys: *J. T. Staley*[1]; *Greg N. Colvin*[1]; [1]Alcoa Inc., Howmet Rsrch. Corp., 1500 S. Warner St., Whitehall, MI 49461 USA

9:45 AM
Numerical Modeling of Squeeze Casting Magnesium Alloy AM50 with Natural Convection: *Alfred Yu*[1]; *Henry Hu*[1]; *Naiyi Li*[2]; [1]University of Windsor, Mechl., Matls. & Auto., 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Ford Motor Company, Mfg. Sys.

Dept., Ford Rsrch. Lab., Rm. 2349, MD3135, 2101 Village Rd., Dearborn, MI 48124 USA

Natural convection has received less attention than conduction in metal solidification processes. It may play an important role in solidification due to its potential influence on solutes distribution in melts and grain structure. A coupled mathematical model has been developed to simulate the solidification phenomena of squeeze cast magnesium alloy (AM50). An analysis based on a Control-Volume Finite Difference approach and an Enthalpy Transforming Model was employed to understand the heat transfer and fluid flow in a cylindrical casting during solidification. The effect of natural convection on the total solidification process of squeeze casting AM50 was examined under various processing parameters, and comparison results are presented and analyzed.

10:10 AM Break

10:20 AM

Directional Solidification of Aluminum-Copper Alloys: Alicia Esther Ares[2]; Rubens Caram[3]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Scis., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]CONICET/University of Florida, Dept. of Matls. Sci. & Eng., 131 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA; [3]State University of Campinas, Dept. of Matls. Eng., Campinas, Sao Paulo 6122 Brasil

The columnar to equiaxed transition (CET) was studied in Al-2wt%Cu, Al-4wt%Cu, Al-10wt%Cu, Al-20wt%Cu, Al-33.2wt%Cu alloys, which were solidified directionally from a chill face. The main parameters analyzed include the temperature gradients, solidification velocities of the liquidus and solidus fronts, and grain size. The transition was observed to occur when the temperature gradient in the melt decreased to values between -3.89∞C/cm to 1.25∞C/cm. In addition, there is an increase in the velocity of the liquidus front faster than the solidus front, which increases the size of the mushy zone. The size of the equiaxed grains increase with distance from the transition, an observation that was independent of alloy composition. The results are compared with both, obtained previously in lead-tin alloys and obtained by other authors.

10:45 AM

Analysis of Grain Size Obtained During Directional Solidification of Aluminum Based Alloys: Alicia Esther Ares[2]; Rubens Caram[3]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Scis., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]CONICET/University of Florida, Dept. of Matls. Sci. & Eng., 131 Rhines Hall, PO Box 116400, Gainesville, FL 32611-6400 USA; [3]State University of Campinas, Matls. Eng. Dept., Campinas, Sao Paulo 6122 Brasil

In the present work measurements of columnar and equiaxed grain size were carried out. The measurements were made from the macrography of aluminum alloys samples such as Al-Cu, Al-Mg, Al-Zn, Al-Si, Al-Si-Cu unidirectionally solidified and which presented a columnar to equiaxed transition. The results of the analysis are compared with those obtained in a previous work in alloys with low melting point, Pb-Sn. The length of the columnar grains and the size of the equiaxed grains are correlated with dynamic parameters derived from temperature measurements, such as, cooling rate, temperature gradients and velocity and position of the liquidus and solidus fronts. The results obtained are presented and discussed.

11:10 AM

Spheroidization of Silicon and its Influence on the Mechanical Properties of the Eutectic Al-12% Si Alloy: *Ruyao Wang*[1]; Weihua Lu[1]; Hsienyang Yeh[1]; Henry H.E. Yeh[1]; [1]Donghua University, Dept. of Mechl. Eng., 1882 W. Yanían Rd., Shanghai 200051 China

A procedure has been developed to vary the morphology of silicon in eutectic Al-12%Si alloy (US patent pending). This technology consists of two steps: firstly, adding small amount of designed modifier into melt, then heating the treated alloy at temperature as in solution treatment. Depending on the holding time at soaking temperature, the silicon morphology varies from connected flake to bar-like shape to, finally, nodule. Experiment showed that the heating temperature designated is much lower than that required in conventional Al-Si alloy, not resulting in coarsening or clustering of silicon particles and making spheroidization of silicon possible. The separated bar-like silicon is capable of remarkably reinforcing the matrix of Al-Si alloy, raising the tensile strength by 30% compared to that in Al-Si alloy with connected silicon. However, the high level of spheroidization of silicon particle doesnít greatly increase tensile strength and ductility, but dramatically raises the wear resistance by 150%.

11:35 AM Cancelled

Hypereutectic Al-Si Alloys with a Completely Self-Modified Eutectic Structure: *Ruyao Wang*[1]; Weihua Lu[1]; [1]Donghua University, Shanghai China

Computational Phase Transformations: Engineering Applications

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Materials Processing & Manufacturing Division, ASM/MSCTS-Thermodynamics & Phase Equilibria Committee, MPMD-Computational Materials Science & Engineering-(Jt. ASM-MSCTS), Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee, Phase Transformation Committee-(Jt. ASM-MSCTS)

Program Organizers: Yunzhi Wang, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA; Perry Leo, University of Minnesota, Department of Aerospace Engineering and Mechanics, Minneapolis, MN 55455 USA; Ralph E. Napolitano, Iowa State University, Ames Laboratory, Department of Materials Science and Engineering, Ames, IA 50011 USA; Vidvuds Ozolins, Sandia National Laboratories, Livermore, CA 94551-0969 USA; Wolfgang Windl, The Ohio State University, Department of Materials Science and Engineering, Columbus, OH 43210 USA

Thursday AM — Room: 11B
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: J. P. Simmons, Air Force Research Laboratories, Dayton, OH 45433 USA; Zi-Kui Liu, Pennsylvania State University, Matls. Sci. & Eng., University Park, PA 16802 USA

8:30 AM Invited

PrecipiCalc: An Efficient Numerical Precipitation Code for Materials and Process Design: *G. B. Olson*[1]; [1]Northwestern University, Dept. Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The Langer-Schwartz theoretical framework for precipitation kinetics is extended to multicomponent systems by a numerical code built on the Thermocalc/DICTRA system for multicomponent thermodynamics and diffusion. Under the DARPA-AIM program on Accelerated Insertion of Materials the code is linked to finite-element simulations of heat treatment of turbine discs to simulate the spatial variation of gí precipitation in IN100 and Rene88DT superalloy discs, with model validation by high resolution microanalytical studies. The code is being tested on several precipitation systems, including the devitrification of metallic glasses under the DARPA-SAM initiative on Structural Amorphous Metals.

9:00 AM

The Modelling of Phase Transformations in the Context of a Generalised Materials Property Capability: *Nigel Saunders*[1]; Xiuqing Li[2]; Alfred Peter Miodownik[1]; Jean-Philippe Schille[2]; [1]Thermotech, Ltd., Surrey Technology Ctr., The Surrey Rsrch. Park, Guildford, Surrey GU2 7YG UK; [2]Sente Software, Ltd., Surrey Tech. Ctr., The Surrey Rsrch. Park, Guildford, Surrey GU2 7YG UK

As part of the development of a new software programme (JMatPro) for calculating general materials properties, work has been undertaken on phase transformations for Al-, Fe-, Ni- and Ti-alloys. Current capability exists for the calculation of non-equilibrium solidification, TTT/CCT diagrams, Ms, gammaí/gammaî coarsening, etc. Examples will be presented on how information from a variety of other subject areas (e.g. thermodynamics, physical and mechanical property modelling) has been combined with phase transformation modelling to provide a more general materials property capability. Examples will include, the combination of solidification models with physical properties calculations, to provide density, volume changes, thermal conductivity etc. and the calculation of gamma/gammaí lattice mismatch as a function of temperature in Ni-based superalloys, which is controlled not only by thermal expansion differences but also by potential changes in gamma/gammaí equilibrium. Further extensions will be discussed including the effect on secondary creep rate of both coarsening of gammaí and the time dependent depletion of heavy elements in gamma due to formation of TCP phases such as sigma.

9:20 AM Invited

On Modeling the Microstructure and Coarsening Kinetics of γí Precipitates in Ni-Base Alloys: *Alan J. Ardell*[1]; [1]University of

California-Los Angeles, Matls. Sci. & Eng., 6531-G Boelter Hall, Los Angeles, CA 90095-1595 USA

The γí (ordered Ni_3Al) precipitate microstructure and its evolution during coarsening in aged binary Ni-Al alloys is a favorite son of the computational modeling community. Many features of the precipitate microstructure have been captured beautifully, e.g. precipitate alignment along elastically soft <001> directions, and their cuboidal shapes. However, several aspects of coarsening have not been investigated by computational methods. These include the anomalous decrease of the coarsening rate with increasing volume fraction, f; the suggestion that γí precipitate shapes might depend on f when f is small, and the concave interfaces of large individual γí precipitates when f is small (< 0.04). Furthermore, many computations use generic values of physical parameters, and therefore cannot reproduce differences among experimentally observed precipitate microstructures; e.g. why Ni_3Ge and Ni_3Al precipitates differ so in shape and spatial distributions. Useful targets for future efforts of computational modeling will be discussed in this presentation.

9:50 AM
Coarsening of Misfitting Particles: Ostwald Ripening and Coalescence: *K. Thornton*[1]; Norio Akaiwa[2]; P. W. Voorhees[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA; [2]National Research Institute for Metals, Tsukuba Japan

We present the results from two-dimensional simulations of Ostwald ripening of second-phase particles in an elastically anisotropic, homogeneous system with cubic symmetry. We utilize advanced numerical methods such as the fast multipole method to include a large number of particles to provide sufficient statistics. Calculated microstructures exhibit experimentally observed features like particle alignment and four- and two-fold symmetric particles. We characterize the microstructure evolution through analyses of the particle morphology, the size distribution, and the spatial distribution. Although the evolution of the system is not self similar, there is strong evidence that the system, including its kinetics, is approximately characterized by the ratio between the elastic energy and the interfacial energy. At high volume fractions, particle separations become so small that coalescence is possible. We discuss the influence of coalescence on the evolution of large systems of misfitting particles undergoing coarsening.

10:10 AM Break

10:25 AM Invited
Phase Field as an Engineering Tool for Heat Treatment Applications: *Jeff P. Simmons*[1]; Youhai Wen[2]; Chen Shen[3]; Yunzhi Wang[3]; [1]Air Force Research Laboratory, AFRLMILLMP, Matls. & Mfg. Direct., WPAFB, Dayton, OH 45433 USA; [2]UES, Inc, Dayton, OH 45432 USA; [3]The Ohio State University, Dept. of Matls. Sci. & Eng., Columbus, OH 43210 USA

The Phase Field model has been demonstrated to have a very wide range of applications to microstructural modeling phenomena, with applications that include both diffusional and shear transformations as well as grain growth phenomena. Like all microstructure simulation techniques, it produces all microstructural information, including particle size distributions, interparticle spacings, particle morphologies, as well as unusual (far from mean) events. In order to develop the Phase Field model into an engineering tool, two practical considerations must be made: (1) how to perform simulations under conditions of concurrent nucleation and growth and (2) quantitative comparison with microstructural descriptors such as volume fraction vs. time, coarsening rates, and particle size distribution effects. This presentation summarizes progress to date towards developing the Phase Field model into a tool suitable for engineering shop practice.

10:55 AM
Phase Field Method and Quantitative Microstructure Modeling: *C. Shen*[1]; Q. Chen[1]; Y. Wang[1]; Y. Wen[2]; J. P. Simmons[3]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [3]Air Force Research Laboratory, AFRL/MLLM, WPAFB, Dayton, OH 45433 USA

Phase field method has been developed for decades. It uses spatially distributed (field) variables to describe kinetic evolutions of material systems containing spatial non-uniformity in material properties. Its capabilities to describe complex microstructural patterns make it attractive for engineering applications. In this presentation we discuss a number of issues related to quantitative applications of the phase field method to engineering systems, including the incorporation of thermodynamic and mobility databases and necessary scaling of energy, length and time. Examples will be given on the development of γ/γí microstructures in Ni-base superalloys by nucleation, growth, and coarsening under isothermal and non-isothermal conditions.

11:15 AM
Modeling of Microstructure Evolution During the Beta to Alpha Transformation in Ti-6Al-4V Alloy: *Qing Chen*[1]; Chen Shen[1]; Kaisheng Wu[1]; Yunzhi Wang[1]; [1]The Ohio State University, Dept. Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

A combination of phase field method and classic nucleation theory has been developed to simulate the formation of grain boundary and Widmanst‰tten alpha structures during the beta to alpha transformation in Ti-6Al-4V alloy. The dependence of non-equilibrium chemical free energy on composition, temperature and long-range order parameter was established on the basis of available CALPHAD thermodynamic description. Newly assessed diffusion mobility data for alpha precipitates and beta matrix have been used in the phase field simulation. The roles of elastic strain energy, interfacial energy and interface mobility in the formation of Widmanst‰tten alpha plates have been investigated. This work is supported by AFRL under MAI contract.

11:35 AM Cancelled
Prediction of Phase Transformation Kinetics and of Ferritic Microstructures in Low-Carbon Steels: *Matthieu Kandel*[1]; [1]IRSID, Physl. Metall. Dept., Voie Romaine, BP30320, MaiziËres-les-Metz 57283 France

11:55 AM
The Quaternary Ni-Al-Mo-Ta Thermodynamic Database for Ni-Base Superalloys: *Shihuai Zhou*[1]; Longqing Chen[1]; Zi-Kui Liu[1]; [1]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., Steidle Bldg., University Park, PA 16802 USA

The thermodynamic database of the Ni-Al-Mo-Ta system is developed for the Ni-base superalloys with special attention to the stability of the topologically close-packed phases. With the CALPHAD technique using a computerized optimization procedure, the thermodynamic descriptions of the compounds δ-NiMo in Ni-Mo system, the ternary compounds in the ternary Ni-Al-Mo, Ni-Al-Ta and Ni-Mo-Ta systems are evaluated. The thermodynamics of the γ and γí phases are modeled with a single Gibbs energy function taking into account the crystallographic relation between these two phases. Various phase diagrams calculated with the current database will be presented and compared with experimental data.

Friction Stir Welding and Processing II: Friction Stir Processing

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

Thursday AM Room: 7B
March 6, 2003 Location: San Diego Convention Center

Session Chair: Rajiv S. Mishra, University of Missouri-Rolla, Metallurgl. Eng., Rolla, MO 65409-0340 USA

8:30 AM
Microstructure Evolution During Friction Stir Processing of an As-Cast Nickel Aluminum Bronze Material: *Terry R. McNelley*[1]; Asunta M. Cuevas[1]; Douglas L. Swisher[1]; [1]Naval Postgraduate School, Mechl. Eng., 700 Dyer Rd., Monterey, CA 93943-5146 USA

The evolution of microstructure during friction stir processing (FSP) of an as-cast Nickel Aluminum Bronze material (Cu-9Al-5Ni-4Fe), which is widely utilized for cast components in marine applications, has been evaluated by orientation-imaging microscopy (OIM) as well as other microanalysis methods. FSP is a novel method for surface modification of deformable metals by severe plastic deformation of the surface layers of the material using a specially designed, rotating tool. The deformation is accompanied by adiabatic heating and so the use of FSP on a cast metal allows the microstructure of near-surface regions to be converted from a cast to a wrought and annealed condition. The process results in large local strains (~10) and pronounced strain, strain rate and temperature gradients, with corresponding microstructure and property gradients that depend on material, tool and processing conditions. At the boundary between base metal and the thermo-mechanically affected zone (TMAZ) there is a characteristic region of grain elongation and lattice rotation. A {111} tends to align with the plane of shear at this boundary. Further on inside the TMAZ, there are regions of grain refinement (~5mm grain/subgrain size) accompanied by microstructural homogenization, followed by regions characterized by recrystallization and grain growth at locations still closer to the centerline of the TMAZ. These regions will be described and the extent and location of each will be related to the tool geometry and FSP conditions.

8:55 AM

Friction Stir Processing for Microstructural Modifications of Aluminum Casting: Z. Y. Ma[1]; *R. S. Mishra*[1]; M. W. Mahoney[2]; [1]University of Missouri, Dept. of Metallurgl. Eng., 218 McNutt Hall, Rolla, MO 65409 USA; [2]Rockwell Scientific, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA

Friction Stir Processing (FSP) can be used to locally tailor the microstructure for enhancement of a specific set of properties. FSP has been applied to cast aluminum plates to modify the microstructure for enhancement of mechanical properties. The influence of FSP parameters on microstructural changes and microstructure-mechanical property correlations will be presented. FSP leads to homogeneous microstructure and eliminates porosity completely. This results in significant enhancement of mechanical properties. This concept can be used to locally enhance the properties of castings.

9:20 AM

Using Grain Growth as an In-Situ Flow Marker During Friction Stir Welding: *William J. Arbegast*[1]; [1]South Dakota School of Mines and Technology, Adv. Matls. Procg. Ctr., 501 E. Saint Joseph St., Rapid City, SD 57701 USA

Friction Stir Welds in aluminum alloys have shown a tendency to undergo abnormal grain growth when subjected to high temperature thermal treatments after FSW processing. This can result in significant reduction in formability during subsequent cold and hot working operations. Experiments were conducted to evaluate the effect of time and temperature on this abnormal grain growth behavior. One Autogenous 2195 FSW and two Hybrid (4043 and 2319 Filler) FSW were prepared and subjected to temperatures ranging from 750°F to 1050°F for various times. Several distinct zones of abnormal grain growth were observed with the degree of growth a function of exposure time and temperature. The FSW nugget zone composition also had an effect on the rate of growth, but did not seem to influence the distinct origination locations. The results of these experiments are presented with discussions regarding the cause of the distinct origination points of the abnormal grain growth.

9:45 AM

Over-Stirring Hole Defects During Thermomechanical Stir Processing: *Saptarshi Mandal*[1]; Keith M. Williamson[1]; Gene J.-W. Hou[1]; [1]Old Dominion University, Dept. of Mechl. Eng., Rm. 238, Kaufman Hall, Norfolk, VA 23529 USA

This paper investigates the critical size of a hole-defect that can be resolved by a stir tool without compromising the mechanical properties of the weld. Hole-defects may occur during tool breakage or from solidification impurities in a work-piece. Results from these experiments show that hole-defects up to 12.5% of the nib diameter are virtually invisible to the thermomechanical stir process. This confirms the robustness of the stir process and suggests that an empirical relationship exists between joint fit-up and the diameter of the nib. These experiments were carried out on lap welds in Aluminum 1100 and 3000 series alloys by ìover-stirringî machined holes of various diameters. The study also determined that the critical hole depth was also a function of nib geometry; however, the full effect of the hole length is more difficult to quantify. Similar results were obtained, when the same experiment was carried out on defective stir welds with cracks of the same scale as the machined holes.

10:10 AM Break

10:20 AM

Effect of Microstructural Evolution on Mechanical and Corrosion Behavior of Friction Stir-Processed Aluminum Alloys: *P. R. Subramanian*[1]; Paul Sudkamp[2]; Alpesh K. Shukla[3]; Lisa M. Young[1]; David P. Mika[1]; Paul L. Dupree[1]; Nirm V. Nirmalan[1]; [1]General Electric, Global Rsrch. Ctr., PO Box 8, Schenectady, NY 12301 USA; [2]General Electric Aircraft Engines, 1 Neumann Way, Cincinnati, OH 45215 USA; [3]Rensselaer Polytechnic Institute, Matls. Rsrch. Ctr., 110 8th St., Troy, NY 12180 USA

Friction stir welding (FSW) is an enabling solid-state joining process for fabrication of low-cost lightweight structures. The microstructural evolution in FSW-processed alloys is closely connected to the local thermomechanical cycles imposed through the combination of frictional heating and mechanical working by the tool material. The mechanical and corrosion properties of the resultant joints, in turn, are intimately linked to the evolved microstructures. In an effort to understand these relationships, FSW simulations were conducted on monolithic aluminum alloy 2195 plates. Thermal fields produced during FSW were determined using infrared (IR) imaging and embedded thermocouples. This presentation will cover the link between the thermal fields and the resultant microstructural evolution in the various FSW zones. The influence of these microstructural features on tensile, fatigue, and corrosion behavior will be highlighted. (This work was sponsored (in part) by the Air Force Office of Scientific Research, USAF, under grant number F49620-01-1-0300).

10:45 AM

The Effect of Friction Stir Processing (FSP) on the Spatial Heterogeneity of Discontinuously-Reinforced Aluminum (DRA) Microstructures: *Jonathan E. Spowart*[1]; Zong-Yi Ma[2]; Rajiv S. Mishra[2]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]University of Missouri-Rolla, Dept. of Metallurgl. Eng., Rolla, MO 65409 USA

In order to introduce different levels of microstructural heterogeneity into an experimental DRA material, three 6061/SiC/25p DRA extrusions were produced, using established P/M techniques. Each contained F-600 grade SiC particles (median diameter, d_{50} = 13.4 µm) however, the median matrix particle size was varied in a controlled manner, by careful screening of the 6061-Al powder stock. The range of matrix particle sizes that were chosen (26.4 µm, 42.0 µm and 108.6 µm) introduced increasing levels of spatial heterogeneity into the DRA microstructures, as quantified using the Multi-Scalar Analysis of Area Fractions (MSAAF) technique. The DRA materials were further processed, using FSP, in order to ascertain the effect of FSP on the homogeneity of the as-extruded microstructures. It was found that FSP introduces a significant amount of solid-state mixing which reduces the amount of microstructural spatial heterogeneity, even in the worst DRA specimens. In addition, mini-tensile tests were carried out on the as-extruded and FSP DRA materials, and these results are presented in the context of the effects of microstructural heterogeneity on tensile mechanical properties.

11:10 AM

Finite Element Simulation of Plane-Strain Thick Plate Bending of Friction Stir Processed 2519 Aluminum: *Michael P. Miles*[1]; Tracy W. Nelson[1]; Murray W. Mahoney[2]; Rajiv S. Mishra[3]; [1]Brigham Young University, Eng. & Tech., 265 CTB, Provo, UT 846002 USA; [2]Rockwell Scientific, A-25, Thousand Oaks, CA 91360 USA; [3]University of Missouri-Rolla, Dept. of Metallurgl. Eng., B37 McNutt Hall, Rolla, MO 65409-0340 USA

Friction stir processing (FSP) is used to modify surface microstructures to enhance the ability to bend thick plate 2519-T8 aluminum. Plate, 25.4 mm thick and 50.8 mm wide, is friction stir processed across the pre-tensile surface 6.3 mm deep. The plates are then bent at 200C, still below the precipitate dissolution temperature, over a punch with radius 38.1 mm into a v-shaped die to progressively greater angles, until the outer surface of the material begins to crack. Parent metal bending results are compared to material with the surface microstructure modified by FSP. Experimental and predicted results are compared with the plate bending process modeled using an elasto-viscoplastic finite element program. The mesh is divided into two zones, i.e., one zone for the unaffected parent material and another zone for the FSP material. The purpose of this initial study is demonstrate the enhanced formability associated with FSP and to validate the modelís accuracy to predict strain for a given bend angle.

11:35 AM

Fatigue Life Improvement of an Aluminum Alloy FSW with Low Plasticity Burnishing: *N. Jayaraman*[1]; Paul S. Prevey[1]; Murray Mahoney[2]; [1]Lambda Research, 5521 Fair Ln., Cincinnati, OH 45227 USA; [2]Rockwell Science Center, 1049 Camino dos Rios, Thousand Oaks, CA 91360 USA

Friction stir welding provides a new technology for solid state joining a wide variety of aluminum alloys that cannot be joined with conventional fusion welds. However, recent work has shown that significant tensile residual stresses are developed in the stirred region with local tension maxima at the transition between the stir and heat-affected zones. Residual tension at the edges of the stir zone has been associated with stress corrosion cracking and corrosion fatigue crack initiation. This fatigue debit has been overcome using low plasticity burnishing (LPB) to introduce a deep surface layer of compressive residual stress. LPB processing after friction stir welding has increased the high cycle fatigue endurance of aluminum alloy FSW by 80%. However, the LPB processing parameters have not yet been optimized to produce the maximum achievable fatigue life. A linear elastic fracture mechanics approach is applied to calculate the fatigue crack growth rates and fatigue lives of friction stir welded 2219-T8751 aluminum with initiation from salt fog pitting. The analysis is performed with and without the including of the deep compressive residual stresses produced by low plasticity burnishing (LPB). Calculated fatigue lives are compared to fatigue data developed in four-point bending at R=0.2 following 100 hr. salt fog pitting corrosion. The results indicate that the improved fatigue life achieved with LPB after friction stir welding can be explained by retardation of crack initiation and growth in the deep compressive layer produced by LPB. The fatigue crack growth analysis provides a theoretical basis for understanding the improved fatigue life realized with LPB and for estimating the residual stress distribution that will provide the highest achievable fatigue strength.

High Temperature Alloys: Processing for Properties: Alternate Materials

Sponsored by: Structural Materials Division, SMD-High Temperature Alloys Committee

Program Organizers: Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA; Jacqui B. Wahl, Cannon-Muskegon Corporation, Muskegon, MI 49443-0506 USA

Thursday AM — Room: 13
March 6, 2003 — Location: San Diego Convention Center

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

8:30 AM
Processing, Characterization and Properties of Laser Deposited Compositionally Graded Titanium Alloys: *Peter C. Collins*[1]; Rajarshi Banerjee[1]; Hamish L. Fraser[1]; [1]The Ohio State University, Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

An upcoming technology for laser deposition of full-density near-net shape components from a powder feedstock is the Laser Engineered Net Shape (LENSô) process. One of the benefits of the LENSô process is its ability to re-create a designed compositional gradient within a single component, and thus fabricate functionally graded materials. Thus, using this processing technique coupled with concepts of combinatorial materials science, it is possible, to explore the functional relationships between microstructure and properties in a variety of alloys of metallurgical importance such as titanium alloys. This study focuses on the processing and characterization designed graded titanium alloys. These composition gradients have been designed around commercially relevant compositions. Additionally, the change in properties will be addressed. Finally, it will be demonstrated that while simple thermodynamic and kinetic models can be used to predict aspects of the microstructural development, a more detailed understanding of competing phase transformations in these titanium alloy systems is required in order to accurately predict the properties.

8:50 AM
Reaction Forging of Iron AluminidesñA Lower Energy Approach to Processing: J. Rodriguez[1]; J. Wall[1]; S. Moussa[1]; *K. Morsi*[1]; [1]University of Missouri, Mechl. & Aeros. Eng., E3411 Eng. Bldg. E., Columbia, MO 65211 USA

The work presents a new low-energy forging approach which can be used to process a range of important aluminide intermetallics and their composites (e.g. titanium aluminides, nickel aluminides, iron aluminides and niobium aluminides). Reaction forging is combines synthesis (RS) with traditional forging technology to process intermetallics with minimal external energy inputs. The effect of varying processing parameters including green density, strain, and forging temperature on the developed microstructure and hardness is presented. Results confirm the feasibility of this new approach to forge iron aluminides at operating temperatures significantly lower than conventionally used.

9:10 AM
Thermal Conductivity of Uniaxially Cold-Pressed Fe2Mo Powders: Seshadri Seetharaman[1]; [1]Royal Institute of Technology, Matls. Sci. & Eng., Brinellvagen 23, Stockholm SE-100 44 Sweden

Fe2Mo intermetallic powders have been produced by H2 gas reduction of Fe2MoO4 at 973 and 1173K to obtain powders having two different average grain sizes. The powders have been pressed uniaxially between 500 and 2000MPa to obtain pellets having different porosities. The thermal diffusivity of the pellets having a relative density (1-porosity) between 0.515 and 0.710 has been measured at room temperature using the laser flash technique. An identical relationship has been found between the thermal diffusivity and the relative density regardless of average grain sizes. In order to understand the relationship betweeen the thermal conductivity and the relative density, a 5 resistor 2-D Ohmís law model has been formulated. It was found that the 5 resistor 2-D Ohmís law model successfully explains the experimental results in both the numerical and conceptual sense.

9:30 AM
Fabrication of TiC-(Al,Ni) Cermets by Reaction Assisted Infiltration for Application at Elevated Temperature: *Naum Frage*[1]; Hayim Prigogine[1]; Moshe P. Dariel[1]; [1]Ben-Gurion University, Dept. Matls. Eng., Beer-Sheva 85103 Israel

TiC based cermets with an intermetallic matrix (Ni3Al or Al3Ni) are known for their physical properties and wear resistance at elevated temperatures. The fabrication of such cermets by free infiltration with a liquid intermetallic phase requires temperatures above 1350¢™C. The aim of the present work was to determine ways for reducing the fabrication temperature by reaction-assisted infiltration. The preforms for infiltration were compacted from TiC and Ni powder mixtures (10, 15, 20wt% Ni) and sintered to different relative densities. The sintered preforms were infiltrated with liquid Al at 1150-1200¢™C under vacuum 10-4 torr. The interaction between the Ni particles and liquid Al leads to the intermatallic phase formation in the course of the infiltration stage. Several cermets with different compositions were fabricated and characterized. The mechanical properties of the cermets of various compositions will be discussed and related to their microstructure.

9:50 AM Break

10:10 AM
Effects of Fine a2-Ti3Al Precipitates on Quasi-static and Dynamic Deformation Behavior in Ti-6Al-4V Alloy: *Dong Geun Lee*[1]; Sung Hak Lee[1]; Chong Soo Lee[1]; [1]Pohang University of Science & Technology, Matls. Sci. & Eng., San 31, Hyoja-dong,, Nam-gu, Pohang, Kyungbuk 790-784 S. Korea

The effects of Ti3Al(·2) phases precipitation in · phase on the tensile and dynamic fracture characteristics were investigated in this study. Overaging heat treatments can precipitate the nano sized Ti3Al phases inside · phase of Widmanstatten and equiaxed. The important microstructural parameters of two microstructures were analyzed and compared those ëbeforeí with those ëafterí ·2 phases precipitation. Dynamic torsional tests were conducted using a torsional Kolsky bar for Widmanst tten and equiaxed microstructures precipitated by ·2 phases. And then the test data were compared with those of the microstructures without ·2 phases. By ·2 phases precipitation, critical dynamic fracture toughness(Ec) and the resistance to the dynamic shear fracture were increased. The possibility of the adiabatic shear band formation was analyzed from the energy and the difference of the void initiation behavior, which showed the increasing the resistance of dynamic fracture and adiabatic shear band formation.

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

Sponsored by: Materials Processing and Manufacturing Division, MPMD-Shaping and Forming Committee

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

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

Session Chairs: Thomas R. Bieler, Michigan State University, Dept. of Cheml. Eng. & Matls. Sci., E. Lansing, MI 44824-1226 USA; Kenji H. Higashi, Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Sakai, Osaka 599-8531 Japan; Gorti B. Sarma, Oak Ridge National Laboratory, Compu. Sci. & Math. Div., Oak Ridge, TN 37831-6359 USA

8:30 AM
Lateral Spread, Temperature and Microstructure Evolution in Hot Flat Rolling of Aluminium Alloys: *Xinjian Duan*[1]; Terry

Sheppard[1]; [1]Bournemouth University, Sch. of Design, Eng. & Compu., 12 Christchurch Rd., Bournemouth, BH1 3NA England

Hot breakdown rolling is a complicated thermomechanical process. There is shape change, temperature variation and microstructural evolution. These three factors affect each other. In this communication, a three-dimensional FEM program, FORGE3®, is used to simulate a four-pass laboratory rolling pass schedule. The computed histories of temperature and subgrain size fit well with the measurements taken from the literature. The temperature change during water quenching is also simulated. The computed width variations agree with those predictions from an empirical formula which was proposed by the present authors. The prediction of recrystallisation is also conducted for this pass schedule by the use of JKAK equation. A new approach is proposed on the calculation of the Zener Hollomon parameter. The discussion focuses on the determination of friction coefficient and the heat transfer coefficient.

8:50 AM
The Effect of Dislocation Structure on Precipitation Behavior in Al-Mg and Al-Mg-Si Alloys: *Reza Shahbazian Yassar*[1]; David P. Field[1]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Spokane St., PO Box 2920, Pullman, WA 99164 USA

The precipitation behavior of a heat treatable Al-Mg-Si alloy (AA6022) and a non-heat treatable Al-Mg alloy (AA5657) were investigated by DSC, SEM and TEM. Selected samples were subjected to deformation before aging and the dislocation structures were characterized. The effect of deformation and aging treatment on precipitation kinetics and precipitate morphology was studied. Also the correlation between the DSC thermal events and SEM-TEM results is discussed.

9:10 AM
Microstructural Control During Hot Deformation Processing of Aluminum 7075 with Rapidly Engineered Dies: *Deepanker Mathur*[1]; *Kuldeep Agarwal*[1]; *Rajiv Shivpuri*[1]; [1]The Ohio State University, Dept. of Industl. & Sys. Eng., 1971 Neil Ave., 210 Baker Sys., Columbus, OH 43210 USA

The controlling driver in the aerospace industry is the manufacturing cost reduction for all components used to fabricate airframe structures, either for commercial or military purposes. The microstructure, texture and mechanical properties of precipitation hardenable Aluminum alloys undergo dynamic changes during thermomechanical processing (TMP). Hot forging of Al 7075 with dies made of stainless steel and bronze mixture with conformal cooling lines, produces a recrystallised microstructure. These dies are manufactured from innovative 3-D printing technology. Changing process conditions such as temperatures, speed of deformation, pressure loads gives flexibility in controlling the microstructure. Tests for determination of heat transfer coefficient are carried out on this material system. Scaled down forging tests representing rib web structures are performed on aluminum 7075 samples with these engineered dies and various cooling rates to study the influence of heat transfer on the microstructural evolution and properties in such a deformation.

9:30 AM
Hot Fracture Behavior of Al-Mg Alloy During Rolling: *Paul T. Wang*[1]; Zhe Jin[1]; [1]Alcoa Inc., Process Tech., Alcoa Techl. Ctr., Alcoa Ctr., PA 15668 USA

Al-Mg alloys were tested under tension and torsion conditions, and rolled at a lab-scale reversing mill to determine hot fracture behavior under various temperatures and strain rates. Two fracture modes were observed, ductile fracture and hot shortness. The corresponding surface morphology representing these modes is analyzed at a local level to reveal potential fracture mechanisms. A fracture criterion applicable to hot deformation is proposed to account for these two fracture modes. Its applications to fracture of free surface during hot rolling are discussed.

9:50 AM
Deformation and Failure Mechanisms in Commercial AA5083 Materials: *Mary-Anne Kulas*[1]; Paul E. Krajewski[2]; Terry R. McNelley[3]; Eric M. Taleff[4]; [1]The University of Texas at Austin, Matls. Sci. & Eng. Prog., MC C2201, Austin, TX 78712 USA; [2]General Motors Corporation, R&D Ctr., 30500 Mound Rd., Warren, MI 48090 USA; [3]Naval Postgraduate School, Dept. of Mechl. Eng., 700 Dyer Rd., Monterey, CA 03043 USA; [4]The University of Texas at Austin, Dept. of Mechl. Eng., MC C2200, Austin, TX 78712 USA

AA5083 aluminum sheet materials have been studied for the determination of deformation and failure mechanisms over a range of temperatures and strain rates. In addition to relatively standard methods of deformation mechanism determination, the characterization of transient creep behaviors has been used for the identification of deformation mechanisms. Both grain-boundary sliding and solute-drag creep are found to contribute to deformation within the range of temperatures and strain rates evaluated. The mechanisms of tensile failure observed include cavitation-induced ductile fracture and cavitation-induced ductile fracture accelerated by neck growth. The failure mechanisms of samples elongated in tension have been quantitatively evaluated using a new parameter, Q. This parameter provides a measure of the contributions to failure from necking and cavitation and is found to provide insight into the deformation mechanisms active prior to failure in the AA5083 materials.

10:10 AM
Constituent Particle Break-Up During Hot Rolling of AA 5182: *Julian H. Driver*[1]; Antoine Baldacci[1]; Annabelle Bigot[2]; Helmut Klocker[1]; [1]Ecole des Mines de St Etienne, SMS, 158 Cours Fauriel, St. Etienne 42023 France; [2]Pechiney, CRV, 725 rue Aristides Berges, Voreppe 38341 France

The break-up of large (> 100 µm3) intermetallic particles during reversible hot rolling has been investigated by experimental studies of particle evolution during hot deformation and by Finite Element micromechanical analyses. The experiments involve controlled hot plane strain compression tests and industrial hot rolling up to strains of 2 or 3. The particle sizes and shapes have been characterized at various strains both by SEM observation after selective dissolution of the Al matrix and, quantitatively, by 3-D synchrotron X-ray tomography. The two types of constituent particles typical of this alloy (Al-Fe-Mn and Mg2Si) are shown to exhibit different behaviours; e.g. the iron-rich particles break up more quickly but also develop wider size distributions. The influence of particle morphology has also been examined by 3-D FE analysis of the stress/strain distributions around complex, ramiform particles. It is shown that the branches of the particles should break off rapidly (e<<1) from the outer branches inwards. The influence of strain rate on particle break-up is also evaluated.

10:30 AM
Hot Deformation and Fracture of an Al-Zn-Mg-Cu Alloy: Christopher Roberts[1]; *Zhe Jin*[1]; [1]Alcoa Technical Center, Alcoa Ctr., PA 15069 USA

A systematic characterization of hot-torsion-deformed samples of a cast Al-Zn-Mg-Cu aluminum alloy was carried out to understand the hot deformation and hot fracture behavior of the alloy over temperatures ranging from 650F to 900F and strain rates from 0.0025/s to 5/s. The torsion tests were conducted using Gleeble machine and samples were quenched immediately after the completion of deformation. The grain structure after hot deformation was found to vary depending on temperature and strain rate. The soluble constituents existing in the starting microstructure were also seen to change primarily depending on the test temperature. A transition from a transgranular failure to a brittle intergranular failure was observed as temperature increases at high strain rates. At low strain rates, however, the fracture appeared to be dominated by decohesion along the recovered subgrain boundaries, which was observed to be independent upon the temperatures studied.

10:50 AM
Mechanical Behavior of Open-Cellular Al Foams at Ambient and Intermediate Temperatures: *I. Nieves*[1]; F. Arceo[1]; T. G. Nieh[2]; J. C. Earthman[1]; [1]University of California-Irvine, Dept. of Cheml. Eng., Biocheml. Eng. & Matls. Sci., Irvine, CA 92695 USA; [2]Lawrence Livermore National Laboratory, Matls. Sci. Div., Livermore, CA 94551-0808 USA

Several open-cellular 6101-T6 Al foams (Duocell) were tested to failure in tension and compression at 293K, 423K, and 523K. These tests were used to examine the effects of temperature, density and macroporosity on the yield stress and elastic compression modulus and to access the effectiveness of density compensation model in predicting elevated temperature cellular behavior. In general, the yield strength values agreed with model predictions at 293K but were noticeably greater than expected at 423K and 523K. The experimental elastic modulus values also agreed with the room temperature predictions but were considerably lower at elevated temperatures. Both yield and modulus data correlated with the SEM results, which revealed increased ductility at elevated temperatures. These results suggest that density compensation alone is inadequate for predicting the mechanical behavior of cellular materials at intermediate temperatures. Accordingly, the researchers have developed new models whose predictions are in better agreement with the experimental results. These improved predictions are compared with those of the Gibson-Ashby density compensation model and the reasons for the discrepancies explained. The new predictions are further used to elucidate the mechanisms governing cellular behavior at intermediate temperatures.

11:10 AM
Structure Property Relationships of Al-(Sc,Zr) Alloys at 24 and 300°C: *Christian B. Fuller*[1]; David N. Seidman[1]; David C. Dunand[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

A study of the relationship between the microstructural and mechanical properties of Al-(Sc,Zr) alloys containing < 0.01% of $Al_3Sc_{1-X}Zr_X$ precipitates is presented. Classical precipitation-strengthening deformation mechanisms are observed at 24°C, where a transition between precipitate shearing and Orowan looping was calculated to occur at an average precipitate radius of 2-3 nm. For alloys aged above 300°C, Zr additions produce longer lifetimes, but have no effect on the magnitude of the peak hardness. Creep experiments of Al(Sc,Zr) alloys at 300°C yield high stress exponents (27-50), and threshold stress values between 0.06 and 0.33 of the Orowan stress. The climb controlled threshold stress is dependent on the average precipitate radius, which is in agreement with Al(Sc) alloys and a model describing the interaction of dislocations and coherent precipitates. This research is supported by the US Department of Energy.

11:30 AM
The Effect of Microstructure on the Properties of AA5182 Alloy: *Zhengdong Long*[1]; [1]University of Kentucky, Ctr. for Aluminum Tech., 1505 Bull Lea Rd., Lexington, KY 40511 USA

The AA5182 alloy is widely used as beverage can end materials due to its excellent strength and formability. The Al-Mg alloy is traditionally believed as non-heat treatment, solid solution strengthened Aluminum alloy. Some investigations indicated that there are different phases existed in this alloy. In this paper, the phases in this alloy were investigated by SEM and TEM. The different heat treatments were applied to obtain different microstructure, and the effects of these treatments on the mechanical properties of hot band and cold rolled sheet were also further studied. The results indicated that the mechanical properties are greatly affected by microstructure and heat treatments.

11:50 AM
Effect of Forming and Treatment Parameters on Mechanical and Fatigue Properties of Al-Li Extrusions: *K. Sperlink*[1]; V. Ocenasek[2]; [1]AIE CR, Novotneho lavka 5, 116 68 Prague Czech Republic; [2]Research Institute for Metals, Panenske Biezany, 250 70 Odolena Voda Czech Republic

Since 1990, research and development of production and treatment technologies of several types of high strength Al-Li extruded shapes have been carried out in cooperation with Russian partners. The paper presents the results of extrusion experiments performed in real industrial conditions together with the results of mechanical, fatigue and fracture toughness testing. Extruded shapes produced from the alloys Al-Mg-Li (1420), Al-Cu-Mg-Li (1441) and Al-Cu-Li (1450) were prepared and tested. In several cases, the properties of Al-Li extrusions were compared with the properties of commonly used AA2124 and AA7075 extrusions. Very extensive work has been done in the investigation of the heterogeneity and anisotropy of large extrusions used in aircraft constructions. The results of property testing were completed with the results of extrusion texture analysis. Both the effect of extrusion conditions (application of direct or indirect method, extrusion ratio) and the effect of the parameters of thermo-mechanical treatment (small plastic deformation after quenching, temperature and duration of artificial ageing) were evaluated. The optimum conditions of the extrusion and consequent treatment of the studied alloys were determined according to these experimental results.

International Symposium on Gamma Titanium Aluminides: Advanced Alloy Design

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, ASM/MSCTS-Materials & Processing, SMD-High Temperature Alloys Committee, SMD-Titanium Committee
Program Organizers: Young-Won Kim, UES, Inc., Materials & Processes Division, Dayton, OH 45432 USA; Helmut Clemens, GKSS, Institute of Materials Research, Geesthacht D-21502 Germany; Andrew H. Rosenberger, Air Force Research Laboratory, Materials & Manufacturing Directorate, Wright-Patterson AFB, OH 45433-7817 USA

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

Session Chairs: Helmut Clemens, GKSS-Research Centre, Inst. for Matls. Rsrch., Geesthacht D-21502 Germany; David G. Morris, CENIM/CSIC, Physl. Metall., Madrid 28040 Spain

8:30 AM Invited
Recent Developments of TiAl Alloys Towards Improved High-Temperature Capability: *Fritz Appel*[1]; [1]GKSS Research Centre Geesthacht, Inst. for Matls. Rsrch., Max-Planck-Str. 1, Geesthacht D-21502 Germany

Demands for higher strength coupled with good oxidation resistance have led to the development of a new family of gamma -TiAl alloys with the base-line composition (at.%) Ti-45Al-(5-10)Nb+X, with X designating modest amounts of several other metallic and non-metallic elements. Experimental data concerning strength, creep resistance and defect structures of such alloys have been analysed to validate their potential for engineering applications. Under high-temperature exposure, microstructural changes are perhaps more likely to occur in this more complex alloy system because quaternary or higher elements are likely to modify the various phase stabilities. To overcome this problem requires a tight optimisation of alloy composition and processing conditions. Engineering alloys can be identified that exhibit excellent creep and oxidation resistance at 700-800∞C and that are capable of carrying tensile stresses of 1000 MPa at room temperature combined with plastic elongations of 1-2%.

9:00 AM
Designing Gamma TiAl Alloys for Enhanced High-Temperature Performance: *Young-Won Kim*[1]; [1]UES, Inc., Matls. & Processes, 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Gamma TiAl alloys are emerging as a pervasive structural materials technology. For the last several years, ever-increasing need of higher temperature materials has resulted in worldwide efforts in the development of such gamma alloys. The main thrust was to enhance resistance to high cycle fatigue deformation and failure, creep deformation and oxidation, and much of this has been accomplished through the refinement of lamellar structures/grains, microalloying, chemistry modification, process development, and/or their combinations. These improvements have led to the introduction of a series of gamma alloys, which are to be used at temperatures up to 800∞C under application stress conditions. Another alloys having higher-temperature capability are expected to emerge from an extended effort in identifying more stable dispersoids and more oxidation-resistant alloying additions. Technological challenges ahead are enormous, however, and they are: fixing a few engineering alloy compositions, applying them in various application-specific product forms, and developing industry scale, cost-effective processing practice. This paper discusses the status of current alloys, advances made in wrought processing, and the design methodology and future outlook of emerging high-temperature alloys.

9:30 AM Invited
A Potential γTiAl-Deduced High Temperature Intermetallic Alloy Based on a New Intermetallic Compound γ1 in TiAl+Nb System: *Guoliang Chen*[1]; Xiaodong Ni[1]; Jingwen Xu[1]; Yanli Wang[1]; Xiping Song[1]; [1]University of Science & Technology-Beijing, State Key Lab. for Adv. Metals & Matls., Beijing 100083 China

Nb atom in TiAl is proved to be substitutional atom of Ti and preferentially occupies on the Ti sublattice by experimental and theoretical methods such as ALCHEMI technique. For the case of low Nb content the site occupation of Nb atoms is random in the Ti sublattice. As increasing Nb content the distribution of Nb atoms in the sublattice become ordering. This is a continuous ordering process occurred in the

TiAl + Nb system with increasing Nb and Al contents. The development of the continuously ordering finally lead to form a new ternary compound g1. The molecular formula of g1 phase was determined to be Ti4Nb3Al9. The unit cell of g1 phase contains 16 atoms and is 4 times of the g-TiAl phase with L1 0 structure. The relationship of lattice parameters between g and g1 phase is: ag1= ÷2 ag cg1=2cg The typical atom occupation in the unit cell as well as the reciprocal lattice of g1 phase has been shown in the paper. The x-ray diffraction pattern of stoichimetrical g1 phase, Ti4Nb3Al9, is shown in the paper too. The space group of the lattice is P4/mmm. Alloy based on the g1 phase contains in the g1 and h-(NbTi)Al3 two phases or in the g, g1and h-(NbTi)Al3 three phase region exhibit attractive high temperature mechanical properties. As an example, the high temperature compressive yield stress in air reaches 1100 C ~700 MPa at strain rate ?=10-2/s and 200 MPa at strain rate ?=10-5/s.

10:00 AM Invited

Microstructure Control Using β-Ti Phase for Wrought Gamma TiAl Based Alloys: *Masao Takeyama*[1]; Satoru Kobayashi[1]; Takashi Matsuo[1]; [1]Tokyo Institute of Technology, Dept. Metall. & Ceram. Sci., 2-12-1 Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Ti-Al-M ternary systems (M: β stabilizer for Ti) exhibit an unique composition range where thermodynamically stable β phase exists at higher and lower temperatures but not in between: β+α->α->β+γ. The presence of β phase at higher temperatures is useful for hot workability, whereas the β phase formed at low temperatures is effective in toughening the materials since the transformation of α->β+γ can produce fully lamellar structure having fine β particles within lamellae. Thus, the microstructure control involving β-Ti phase in this composition range makes it possible to develop a new class of wrought gamma alloys to be used at around 900 K. In this study, the alloying effects on the phase equilibria and kinetics of microstructure evolution along the pathway (α->β+γ) in ternary systems have been presented. The knowledge is extended to quaternary systems, and a design concept for development of multi-component wrought gamma alloys will be discussed.

10:30 AM Invited

High Temperature Properties of TiAl Alloys Produced by Elemental Powder Metallurgy: Y. Wu[1]; *S. K. Hwang*[1]; S. W. Nam[2]; N. J. Kim[3]; [1]Inha University, Matls. Sci. & Eng., 253 Yonghyun-Dong, Nam Gu, Incheon 402-751 Korea; [2]KAIST, Daejeon 305-701 Korea; [3]CAAM, POSTECH, Pohang 790-784 Korea

Through the fundamental research works in the last decade, it has been realized that elemental powder metallurgy (EPM) is a viable approach to produce sound TiAl alloys of dependable quality. Alloy design efforts resulted in a variety of chemical compositions such as Ti-46.6Al-1.4Mn-2Mo with assorted minor additions of C or Y. Fully lamellar microstructures with extremely fine lamellar thickness could be obtained and were shown to have about 3% in tensile elongation and close to 700MPa in tensile yield strength at room temperature. Furthermore, in high temperature air tests, the alloys exhibited a rather low secondary creep rate as well as a good oxidation resistance at 800?aC. The attractive properties have been attributed to the fine-scale lamellar microstructure, precipitation hardening and the formation of a surface oxide phase impervious to oxygen penetration.

11:00 AM Invited

Processing, Microstructure and Mechanical Properties of Directionally Solidified TiAl Based Alloy Reinforced by Al2O3 Particles: *Juraj Lapin*[1]; Mohamed Nazmy[2]; [1]Slovak Academy of Sciences, Inst. of Matls. & Machine Mechanics, Racianska 75, Bratislava SK-831 02 Slovak Republic; [2]Alstom Power, Haselstrasse 16, Baden 5401 Switzerland

The processing, microstructure and mechanical properties of intermetallic Ti-46Al-2W-0.5Si (at%) alloy directionally solidified (DS) in alumina moulds were studied. After directional solidification the microstructure consisted of regular a2- and g-lamellae, elongated B2 particles, Al2O3 particles and fine Ti5Si3 precipitates. Various volume fractions of Al2O3 particles and mean interlamellar spacings in DS ingots were achieved by variations of the growth rate, reaction time and cooling rate. The ingots with constant volume fraction of Al2O3 particles were prepared at a constant growth rate and various mean interlamellar spacings were achieved by subsequent solution annealing followed by cooling at constant rates. The volume fraction of Al2O3 particles increased with increasing temperature of the melt and reaction time. The mean interlamellar spacing for both DS and heat-treated (HT) ingots decreased with increasing cooling rate. In DS ingots, microhardness, compression strength, yield strength and deformation to fracture increased with increasing cooling rate. In HT ingots, microhardness and yield strength increased and compression strength and deformation to fracture decreased with increasing cooling rate. The yield stress increased with decreasing interlamellar spacing and increasing volume fraction of Al2O3 particles. A simple model including the effect of interlamellar spacing and volume fraction of Al2O3 particles was proposed for the prediction of the yield stress.

11:30 AM Invited

Development of a Hot Forged TiAl Alloy and Manufacturing of Components: *Toshimitsu Tetsui*[1]; [1]Mitsubishi Heavy Industries, Nagasaki R&D, 5-717-1 Fukahori-machi, Nagasaki 851-0392 Japan

In order to enlarge parts size and reduce material cost of gamma TiAl alloy, a new hot forgeable TiAl alloy has been developed. A composition of this alloy is Ti-42Al-5Mn (at%) and a specific gravity of 4, and a microstructure shows fine-grained lamellar + beta + gamma structure. In the manufacturing process a cast ingot is heated to the beta + alpha region, subsequently hot forged. After hot forging heat treatment of near 1200°C is applied for stress relief and softening. Due to the effect of the beta phase that does not appear in ordinary gamma TiAl alloy, the machinability of this alloy is improved and complicated shape can be machined without chipping. In this paper, properties of this alloy such as tensile, fatigue, fracture toughness and creep will be reported and also manufacturing of components such as blades and special structural parts will be demonstrated.

12:00 PM

Microstructure and Mechanical Properties of Extruded Gamma Met PX: *Susan L. Draper*[1]; Gopal Das[2]; Ivan E. Locci[1]; J. D. Whittenberger[1]; H. Kestler[3]; J. L. Smialek[1]; [1]NASA Glenn Research Center, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [2]Pratt & Whitney, 400 Main St., E. Hartford, CT 06108 USA; [3]Plansee AG, Reutte A-6600 Austria

The potential of Gamma Met PX, a high strength alloy based on TNB developed by GKSS, Germany, to be utilized as a compressor blade material is being assessed. The microstructure and mechanical properties of VAR cast Ti-45Al-X(Nb,B,C) (at.%) gamma TiAl hot extruded above the alpha transus temperature were evaluated. Heat treatments of the as-extruded rods have led to the development of a duplex microstructure at 1290∞C and a fully lamellar microstructure at 1340∞C, suggesting that the alpha transus temperature for the alloy is lower than 1340∞C. Both tensile and compression behavior of as-extruded and heat treated specimens were studied in the temperature range of RT to 871∫C. In general, the yield stress, ultimate tensile strength and the elastic modulus decreased with increasing deformation temperature. Compression creep testing between 727 and 1027∫C revealed that the properties were reproducible and predictable; while strengths reached superalloy-like levels at fast strain rates and lower temperatures, def ormation at slower strain rates and/or higher temperature indicated significant weakening. Microstructural evolution during heat treatment, identification of various phases, and deformation mechanisms for tensile, compression and creep processes will be discussed in terms of the current understanding of phase transformation and mechanical behavior in gamma TiAl alloys.

12:20 PM

Processing, Properties, and Microstructure of Gamma-TiAl Alloys with Niobium Ranging from 4 to 10 at. %: *Vinod Kumar Sikka*[1]; Tadeu Carneiro[2]; Edward A. Loria[3]; [1]Oak Ridge National Laboratory, Metals & Ceram., PO Box 2008, Oak Ridge, TN 37831-6083 USA; [2]Reference Metals Company, Inc., 1000 Old Pond Rd., Bridgeville, PA 15017 USA; [3]Consultant, 1828 Taper Dr., Pittsburgh, PA 15241 USA

The γ-TiAl-based alloys have been investigated extensively in literature. However, there is still a need for further development of γ-TiAl-based alloys with a unique combination of low-temperature ductility and high-temperature strength and oxidation resistance. The purpose of this study is to investigate the effect of varying niobium from 4 to 10 at. % for a base 46 at. % Al composition. The alloy compositions investigated are included in Table 1. The alloys for this study were prepared by nonconsumable-arc melting and extruding them to a reduction ratio of 16:1 at a temperature above α-transus of 1350∞C. The α-transus temperature was measured to vary from 1300 to 1315∞C for alloys with 4 to 10 at. % Nb.

12:40 PM

TiAl G4 Alloy: Investment Cast Processing and Mechanical Properties: *Grange Marjolaine*[1]; Thomas Marc[2]; Raviart Jean-Louis[2]; Belaygue Philippe[3]; Recorbet David[4]; [1]Snecma Moteurs, Service YKO, Ctr. de Villaroche, Moissy, Cramayel 77550 France; [2]ONERA, DMMP, 29 Ave. de la Division Leclerc, BP72, Chatillon, Cedex 92322 France; [3]TurbomÈca, DÈpt. MatÈriaux, Bordes, Cedex 64511 France; [4]Centre díEssais AÈronautique de Toulouse, Div. S, 23 ave. Henri Guillaumet, Toulouse, Cedex 31056 France

Over the last years, Snecma Moteurs has devoted extensive research effort in an advanced cast gamma TiAl french program, aimed at optimizing investment cast structure and at demonstrating the manufacturing of near net-shape turbine blades. In an attempt to improve the quality of cast products, a Ti-47Al-1Re-1W-0.2Si (at%) alloy, namely G4, has been used for this program in order to promote beta equiaxed-type solidification instead of the conventional alpha columnar-type solidification. Current status of investment casting process and of microstructure/property relationships of alloy G4 is highlighted in the present paper. Improved mechanical properties and environmental resistance of alloy G4 with respect to conventional near-gamma alloys have been assessed. In contrast to the commonly practiced subtransus heat treatment applied to conventional gamma aluminides to achieve nearly lamellar microstructure, a modified heat treatment condition leading to a duplex microstructure was found to considerably increase the creep resistance and the fatigue behaviour of this alloy.

International Symposium on Intermetallic and Advanced Metallic Materials - A Symposium Dedicated to Dr. C.T. Liu: Intermetallics VII–High Temperature Intermetallics II

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

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

Thursday AM	Room: 8
March 6, 2003	Location: San Diego Convention Center

Session Chairs: David P. Pope, University of Pennsylvania, Matls. Sci. & Eng., Philadelphia, PA 19104 USA; Y. Austin Chang, University of Wisconsin-Madison, Dept. of Matls. Sci. & Eng., Madison, WI 53706 USA

8:30 AM Invited
Damage Tolerance in Refractory Mo-Mo3Si-Mo5SiB2 Silicides at Temperatures up to 1300∞C: *R. O. Ritchie*[1]; H. Choe[1]; J. H. Schneibel[2]; [1]Lawrence Berkeley National Laboratory, Matls. Scis. Div., Berkeley, CA 94720 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Oak Ridge, TN 37831-6115 USA

There has been considerable interest of late in multiphase Mo-Si-B refractory alloys for potential higher-temperature advanced engine systems due to their high melting temperatures (> 2000°C) and relatively good oxidation resistance. In this study, we investigate the damage-tolerant properties, i.e., fracture toughness and fatigue-crack propagation behavior, of several such multiphase Mo-Si-B intermetallics. Specifically, the alloys Mo-12Si-8.5B, Mo-16.8Si-8.4B, and Mo-10Nb-12Si-8.5B (at.%), consisting of alpha-Mo, Mo3Si, and Mo5SiB2 (T2) as their primary phases, are examined at temperatures from ambient to 1300°C, with the objective of discerning salient toughening mechanisms. It is found that Mo-12Si-8.5B (at.%) alloy in particular displays relatively high intrinsic (crack-initiation) toughness both at ambient and elevated temperatures, which is attributed to a crack-trapping mechanism at coarse alpha-Mo particles. Moreover, both fracture toughness and fatigue-crack growth properties in this alloy actually improve as temperature increases up to 1300°C. This is principally due to the enhanced ductility of alpha-Mo phase at elevated temperatures, which promotes some degree of extrinsic toughening, specifically crack bridging, which in turn results in rising resistance-curve behavior at 1300°C.

8:50 AM Invited
Iridium Alloys for Electric Generators in Space: *E. P. George*[1]; C. G. McKamey[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6093 USA

Interplanetary spacecraft need onboard electric power that is reliable and stable over many years. Solar cells and fuel cells can be used for near-Earth missions but, for deep space missions, radioisotope thermoelectric generators (RTGs) remain the only viable choice. RTGs work by converting the heat generated during the radioactive decay of plutonia directly to electricity with the help of Si-Ge thermoelectric elements. Most recently, they were used onboard the Cassini mission to Saturn. This talk will describe C. T. Liuís pioneering contributions to the development of the iridium alloy that is currently used to contain the plutonia fuel in RTGs. He made the key discovery that alloying with ëppm levelsí of Th suppresses grain-boundary fracture and dramatically improves the high-temperature impact ductility of iridium. We will discuss the physical mechanisms responsible for the Th effect, including grain refinement and enhanced grain-boundary cohesion. The talk will conclude by describing current research at ORNL that builds on Liuís early work and is aimed at improving the weldability and impact ductility of iridium alloys as well as understanding the effects of trace impurities on mechanical properties. Research sponsored by the Office of Space and Defense Power Systems and the Division of Materials Sciences and Engineering at the Oak Ridge National Laboratory managed by UT-Battelle, LLC, for the US Department of Energy under contract DE-AC05-00OR22725.

9:10 AM
Effects of Nb on the Yield Strength and Ductility of the C11b Structure of MoSi2: *Adel A. Sharif*[1]; Amit Misra[2]; Terence E. Mitchell[2]; [1]California State University, Los Angeles, Col. of Eng., Compu. Sci., & Tech., Dept. of Mechl. Eng., 5151 State Univ. Dr., Los Angeles, CA 90032 USA; [2]Los Alamos National Laboratory, MST-8: Struct. Property Relations, MS G755, Los Alamos, NM 87545 USA

The effects of alloying with 1 at% Nb on the mechanisms of deformation of MoSi2 are investigated from room temperature to 1600°C. Nb alloying lowered the brittle to ductile transition of MoSi2 from about 900°C to room temperature and increased its yield strength at 1600°C by an order of magnitude. The dislocation substructure investigations are utilized to elucidate the source of concurring low-temperature solid solution softening and high-temperature solid solution hardening observed in the presence of Nb in the C11b structure of MoSi2.

9:25 AM Invited
Effects of Microstructure on the High Temperature Mechanical Properties of Al_3Ti-Mo_3Al Two-Phase Intermetallic Alloys: *Seiji Miura*[1]; Hiroyuki Shimamura[1]; Juri Fujinaka[1]; Tetsuo Mohri[1]; [1]Hokkaido University, Matls. Sci. & Eng., Kita 13, Nishi 8, Kita-Ku, Sapporo, Hokkaido 060-8628 Japan

We present the results of the study on the high temperature deformation behavior of a two-phase intermetallic-based alloy composed of the $D0_{22}$-Al_3Ti and A15-Mo_3Al phases with an equi-axed structure or a lamellar structure. It was found that the equi-axed structure formed by an isothermal heat treatment was very stable even after a long-term heat treatment and, moreover, the microstructure was retained even after a heavy deformation at high temperatures. Although the stress exponent is rather lower a steady state deformation was attained, and compressive deformability was found to be extremely high with the equi-axed structure. On the other hand, a lamellar structure was deterious after compression tests. The stress exponent of the specimens with a lamellar structure is rather high, and the resulting microstructure strongly suggested that the deformation was concentrated at boundaries between lamellar colonies.

9:45 AM
Controlling the Thermal Expansion Anisotropy of Mo_5Si_3 and Ti_5Si_3 Silicides: *Joachim H. Schneibel*[1]; Claudia J. Rawn[1]; E. Andrew Payzant[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, MS 6115, Oak Ridge, TN 37831-6115 USA

The silicides Mo_5Si_3 and Ti_5Si_3 are potential high-temperature structural materials with melting points in excess of 2100°C. Unfortunately, the ratio of their coefficients of thermal expansion in the crystallographic a and c directions, CTE(a)/CTE(c), is very high with values of 2.0 and 2.7, respectively. This pronounced anisotropy results in severe microcracking of polycrystals of these materials. Guided by the ab-initio model of Fu et al. [Phil. Mat. Lett. 80 (2000) 683-690)], ternary and quaternary alloying additions capable of reducing the CTE anisotropy were identified. Thermal expansion coefficients of promising alloys were determined by powder x-ray diffraction using a precisely controlled high-temperature stage. In addition, the site occupation of the ternary alloying additions, which is of crucial importance for interpreting the CTEs, is under investigation. Alloys with anisotropy values as low as 1.16 exhibiting dramatically reduced microcracking were obtained. This work was sponsored by the Division of Materials Sciences and Engineering, Office of Basic Energy Sciences, and the Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program, Oak Ridge National Laboratory. ORNL is operated by UT-Battelle, LLC, for the USDOE under Contract No. DE-AC05-00OR22725.

10:00 AM

Multi-Phase Microstructure Design Coupled with Thermodynamic Modeling in the Mo-Si-B-Ti System: *Y. Yang*[1]; Y. A. Chang[1]; [1]University of Wisconsin-Madison, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706-1595 USA

Based on the critically assessed literature information and our own experimental data, all ternary systems in the Mo-Si-B-Ti system have been thermodynamically modeled. A consistent thermodynamic description for the Mo-Si-B-Ti system is thus obtained via the descriptions of the low order systems. A few key quaternary alloys were made to validify the thermodynamic description. The calculations show good agreements with experimental observations. By means of this set of thermodynamic description, a reaction scheme at 1600∞C in metal-rich side is presented, the reaction scheme aims to serve as a guide for multi-phase microstructure design at extremely high temperature structural applications.

10:15 AM Break

10:30 AM Invited

Phase Equilibria in the Ternary Fe-Rh-Ti System: Jozef Balun[1]; Luiz Tadeu Fernandes Eleno[2]; *Gerhard Inden*[1]; Claudio Geraldo Schoen[2]; [1]Max-Planck-Institut f¸r Eisenforschung GmbH, Physl. Metall., Max-Planck-Str. 1, D¸sseldorf D-40237 Germany; [2]Escola Politecnica da Universidade de Sao Paulo, Computatl. Matls. Sci. Lab./Dept. Metall. & Matl. Eng., Av. Prof. Mello Moraes 2463, Sao Paulo-SP CEP 05508-900 Brazil

No information is available about phase equilibria in the ternary Fe-Rh-Ti system. In all three binary subsystems a strong atomic ordering tendency exists between unlike atoms leading to a very stable bcc B2 ordered structure around the equiatomic composition. It is thus expected that a wide B2 phase field should exist in the ternary system with continuous solubility across ternary sections between binary stoichiometric B2 phases. Experiments were carried out at 1273K and 1073K to determine the isothermal sections. Both diffusion couples and individual alloys with compositions within two- or three-phase fields were analysed. The resulting sections show a large ternary miscibility gap. Calculations were performed using the Cluster Variation Method in the irregular tetrahedron approximation in order to model the experimental observation. Pair and tetrahedron interactions were used to calculate prototype diagrams guiding towards a set of interaction parameters which provides a fairly good description of the experimental findings.

10:50 AM Invited

Microstructure and Oxidation Behavior of Mo-Si-B Intermetallic Alloys: Keith J. Leonard[2]; Brian Kowalski[1]; Madan G. Mendiratta[3]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; [2]Oak Ridge National Laboratory, Metals & Ceram. Div., Bldg. 4500S, MS 6116, Oak Ridge, TN 37831-6116 USA; [3]UES, Inc., Air Force Rsrch. Lab., WPAFB, Bldg. 655, Dayton, OH 45433 USA

There is growing interest in refractory intermetallic alloys based on the Mo-Si-B system for very high temperature structural applications. These alloys is characterized by a multiphase microstructure consisting of α-Mo, Mo_3Si and Mo_5SiB_2 (T2) phases. In the present study, thermal effects on microstructure evolution in a Mo-7.44Si-8.51B (at.%) alloy were studied and the microstructures characterized by XRD, SEM, TEM and hardness measurements.. The results indicate that it is possible to exert some control over microstructure and properties by very high temperature heat treatments. Significant changes in volume fraction of α-Mo, Mo_3Si and T2 phases occur at temperatures ≥1700∞C. In addition, the cyclic oxidation behavior in air at temperatures between 800-1100∞C were studied in a three-phase Mo-12Si-12B (at.%) and near-single T2 phase Mo-12.5Si-25B alloy. The evolution of the oxidation process and products during the early, transient period was also studied and the microstructural changes characterized by XRD and SEM. The results indicate that catastrophic oxidation occurs in both alloys at/below 800-900∞C; performance and oxidation protection is better at ≥1000∞C. A porous, non-protective borosilicate/B-SiO_2 layer forms at low temperatures, which permits easy oxygen diffusion and increased weight loss through volatilization of Mo as MoO_3 gas. A stable, dense silica scale forms at/above 1000∞C, which provides protection from oxidation and reduced weight loss. The authors are thankful for financial support of this work by AFOSR (Grant # F49620-00-1-0080, Dr. Craig. S. Hartley, Program Monitor).

11:10 AM

Influence of Alloying Elements on the Mechanical Properties of FeCo-V Alloys: A Review: R. S. Sundar[1]; S. C. Deevi[1]; [1]Chrysalis Technologies, Inc., Rsrch. Ctr., Richmond, VA 23237 USA

Among the soft magnetic materials, ordered intermetallic alloys based on FeCo exhibit highest saturation magnetization, high Curie temperature, good permeability, low magnetic anisotropy and reasonable strength. The recent interest to design a imore electric aircraftî sparked interest in improving the high temperature mechanical properties of FeCo-V alloys while maintaining the excellent magnetic properties. FeCo-2V alloys offer the highest saturaization magnetization but do not meet the more electric aircraft design requirements of the yield strength and creep resistance. The aim of this paper is to review the influence of alloying elements and select the best possible alloying elements to enhance the strength and creep resistance without sacrificing the electrical and magnetic property requirements. In addition, we review the physical metallurgy of FeCo-V alloys and suggest the strengthening mechanisms based on which strength and creep resistance can be improved. We will also present limited experimental data indicating that the design requirements can be met without deteriorating the electrical and magnetic properties of FeCo-2V alloys.

11:25 AM

Microstructure of Nb Substrates Coated with Mo(Si, Al)$_2$-Al$_2$O$_3$ Composite and B-Doped Mo$_5$Si$_3$ Layers by Spark Plasma Sintering: *Takashi Murakami*[1]; Shinya Sasaki[1]; Kazuhiro Ito[2]; Haruyuki Inui[2]; Masaharu Yamaguchi[2]; [1]National Institute of Advanced Industrial Science and Technology, Trib. Grp., Inst. of Mechl. Sys. Eng., AIST Tsukuba E., 2-1, Namiki 1-Chome, Tsukuba, Ibaraki 305-8564 Japan; [2]Kyoto University, Dept. of Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan

Nb alloys have attracted much attention as high temperature structural materials. However, their high-temperature oxidation resistance has yet to be much improved. In this study, Nb substrates were coated with Mo(Si, Al)$_2$-Al_2O_3 composite, B-doped Mo_5Si_3 and NiAl by spark plasma sintering, and microstructure and thermal stability of these coated specimens were examined. When thin Al foil was inserted between Mo(Si, Al)$_2$-60wt% Al_2O_3 powder and Nb substrate before sintering, Nb substrate was found to be coated with a thin Al_2O_3 interlayer and a Mo(Si, Al)$_2$-60wt% Al_2O_3 outlayer without forming any cracks. The Al_2O_3 interlayer is believed to be formed by partial oxidation of Al foil during sintering in a vacuum of 10Pa. Nb substrates can be coated with B-doped Mo_5Si_3 although a thin reactive layer was formed between the substrates and B-Mo_5Si_3. The results of coating Nb substrates with NiAl will be also shown in this presentation.

11:40 AM

Physical and Mechanical Properties of Single Crystalline $Mo_5X_{3+\alpha}$ (X=Si, B, C) and Related Mo Based Multiphase Compounds: *Kazuhiro Ito*[1]; Taisuke Hayashi[1]; Michiaki Kumagai[1]; Keisuke Ihara[1]; Katsushi Tanaka[1]; Masakuni Fujikura[2]; Masaharu Yamaguchi[1]; [1]Kyoto University, Matls. Sci. & Eng., Sakyo-ku, Kyoto 606-8501 Japan; [2]Japan Ultra-High Temperature Materials Research Center, Ltd., 3-1-8 Higashi-cho, Tajimi, Gifu 507-0801 Japan

$Mo_5X_{3+\alpha}$ (X=Si, B, C) intermetallic compounds such as Mo_5SiB_2 ($D8_l$), Mo_5Si_3 ($D8_m$) and Mo_5Si_3C ($D8_8$) have a great potential for ultra-high temperature applications. Anisotropy of the coefficient of thermal expansion for Mo_5SiB_2 (α_c / α_a=1.2-1.6) is significantly reduced from about 2 of other two $Mo_5X_{3+\alpha}$. Thermal conductivity of Mo_5SiB_2 at room temperature is about 0.3W/cmK, which is a quarter lower than that of Mo due to complex structure. Of interest is that Mo_5Si_3C shows relatively lower thermal conductivity (0.098W/cmK) and higher electrical resistivity (150μΩcm). The creep strength of Mo_5SiB_2 is superior to those of $MoSi_2$ and Si_3N_4 based structural ceramics. Direct access to Mo_5SiB_2+ Mo_{ss} eutectic microstructure was succeeded in synthesis using an optical floating zone method at a growth rate of less than 5mm/h. The room temperature fracture toughness of the alloys is on the order of 16 MPa√m and substantially improved from that of the monolithic Mo_5SiB_2.

11:55 AM Concluding Remarks by Organizers

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

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

Thursday AM　　Room: 15B
March 6, 2003　　Location: San Diego Convention Center

Session Chairs: K.-N. Tu, University of California-Los Angeles, Matls Sci. & Eng., Los Angeles, CA 90095 USA; Ray Fournelle, Marquette University, Matl. Sci. & Eng. Prgm., Milwaukee, WI 53201 USA

8:30 AM Invited
Effect of Intermetallic Compound Layer Development on Mechanical Strength of Lead Free Solder Joints: *Kikuo Kishimoto*[1]; Masaki Omiya[1]; Masazumi Amagai[2]; [1]Tokyo Insitutute of Technology, Dept. of Mechl. & Control Eng., 2-12-1 O-okayama, Meguro-ku, Tokyo 152-8552 Japan; [2]Texas Instrument Japan, Ltd., Pkg. Dvlp., 4260, Kawasaki, Hiji-machi, Oita 879-1595 Japan

A mechanical reliability of the solder joints is a serious concern. Intermetallic compounds are formed during reflow process between solder and electrical pads and gradually grow in service. Due to its brittle manner, the reliability of solder joints are supposed to degenerate. By using the Cu-plates on which Cu or Ni or Ni/Au plating was deposited, the specimens of solder joints were fabricated with Sn-Ag-based lead free solders. After aging it in an isothermal chamber, tensile tests were performed and the relationships between solder joint strength and aging period were considered. From SEM microscope observation and EDX microprobe analysis, the growth and components of the intermetallic compounds layer were examined. To investigate the stress condition on the solder joint interface, the finite element analysis was also carried out.

8:55 AM
Mechanical Properties of Intermetallic Compounds Formed in Pb-Free Solder Joints During Isothermal Aging: H. Rhee[1]; F. Guo[1]; J. P. Lucas[1]; K. N. Subramanian[1]; [1]Michigan State University, Cheml. Eng. & Matls. Sci., E. Lansing, MI 48824-1226 USA

The intermetallic compound (IMC) layer that forms during reflow can play pivotal roles on reliability and failure of solder joints irrespective of whether failure occurs within the IMC or in the solder region adjacent to IMC. To understand the role of IMCs in small solder joints, common in microelectronics, their micro-mechanical properties were probed primarily by nano indentation testing (NIT). Fracture and flow properties were determined for interfacial IMCs and constituent IMC particles present in solder in unaged and isothermally-aged conditions in base and composite Pb-free solder alloys. Roles of hardness, modulus and creep property data obtained from such studies on the solder joint reliability will be presented. Project funded by National Science Foundation under grant No. NSF-DMR-0081796.

9:15 AM
Effect of Microelements Addition on the Interfacial Reaction Between Sn-Ag-Cu Solders and Cu Substrate: *Chiang Ming Chuang*[1]; Kwang Lung Lin[1]; [1]National Cheng-Kung University, Dept. of Matls. Sci. & Eng., Tainan 701 Taiwan

Two kinds of Sn-Ag-Cu lead-free solders, Sn-3.5Ag-0.7Cu and Sn-3.5Ag-0.5Cu-0.07Ni-0.01Ge (in wt%), were selected to explore the effect of microelements (Ni and Ge) on the interfacial reaction between solder and Cu substrate. After same time dipped, the thickness of the interfacial intermetallics of the Ni and Ge addition solder is several times as that of the Sn-3.5Ag-0.7Cu solder. Besides, the additional microelements would transfer the feature of interfacial intermetallics from pebble shape to worm shape. However, the negligible difference in XRD data of these two alloys suggests that both interfacial intermetallics have the same crystal structure. The major interfacial intermetallic formed with Sn-3.5Ag-0.7Cu solder is Cu6Sn5, while it is (Cux, Ni1-x)6Sn5 with Sn-3.5Ag-0.5Cu-0.07Ni-0.01Ge. The analytic results of Electron Probe Microanalyzer (EPMA) show that the aggregating Ni into the interfacial intermetallics plays the influential role on the difference of interfacial reaction rate and the morphology of interfacial intermetallics.

9:35 AM
Mechanical Properties of Sn-Based Intermetallics Measured by Nanoindentation: *Richard R. Chromik*[1]; Sarah L. Allen[1]; Michael R. Notis[1]; Rick P. Vinci[1]; [1]Lehigh University, Matls. Sci. & Eng., 5 E. Packer Ave., Bethlehem, PA 18103 USA

In solder joints prepared with Sn-based solders, one finds intermetallic compounds present as distributed phases in the bulk of the solder alloy, and as discrete layers present at the interface between the solder and the metallization or substrate. These intermetallics are typically metal-Sn alloy compounds, such as Ag_3Sn, Cu_6Sn_5, $AuSn_4$, and Ni_3Sn_4. In order to examine the role of intermetallic phases in determining the ultimate mechanical strength and lifetime of a solder joint, the first step is to characterize the mechanical properties of the intermetallics themselves. We report on our study using nanoindentation to characterize elastic and plastic behavior of many common intermetallic alloys. The measured mechanical properties will be used as a database for future finite element modeling (FEM) that takes into account the presence of the intermetallics and the experimentally observed microstructure of solder joints.

9:55 AM
Thickening Kinetics of Interfacial Cu_6Sn_5 and Cu_3Sn Layers During Reaction of Liquid Tin with Solid Copper: *Robert A. Gagliano*[1]; Morris E. Fine[2]; [1]Ondeo Nalco, Metall. Dept., 1 Ondeo Nalco Ctr., Aurora, IL 60563 USA; [2]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

Thickening behavior of interfacial η (Cu_6Sn_5) and ε (Cu_3Sn) phase intermetallic layers was investigated in liquid tin/solid copper reaction couples over reaction times from 1 minute to 100 minutes and temperatures from 250°C to 300°C. Scanning electron microscopy (SEM) was used to quantify the interfacial microstructure at each processing condition. The η was always found to develop with a scalloped growth morphology, while the ε always grew as a somewhat undulated planar layer in phase with the ε. The thickness of each phase was quantitatively evaluated from SEM micrographs using imaging software. Thickening kinetics of the η and ε compounds were modeled using a time and temperature dependent empirical power law. From the model, values for the kinetic exponent, rate constant, and activation energy were established for each intermetallic layer. Measured values for the kinetic exponents and activation energies suggest that thickening of the η is controlled by a grain boundary diffusion mechanism, and growth of the ε occurs by simple solid state diffusion.

10:15 AM Break

10:30 AM Invited
Compound Formation for Electroplated Ni and Electroless Ni in the Under Bump Metallurgy with 42Sn-58Bi Solder During Aging: *Jenq Gong Duh*[1]; Bi Lian Young[1]; [1]National Tsing Hua University, Dept. of Matls. Sci. & Eng., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan

Ni-based under bump metallurgy (UBM) is of focused interests due to slower reaction rate with Sn-rich solders compared to Cu-based UBM. In this study, several UBM schemes using Ni as the diffusion barrier are investigated. Joints of Sn-58Bi/Au/electroless nickel (EN)/Cu/Al2O3 and Sn-58Bi/Au/electroplated nickel/Cu/Al2O3 were aged at 110∞C and 130∞C for 1~25 days to study the interfacial reaction and microstructural evolution. Sn-Bi solders react with the Ni-based multi-metallization, and form ternary Sn-Ni-Bi IMC during aging at 110∞C. Compositions of ternary IMC were Sn79Ni16Bi5, Sn78Ni16Bi6 and Sn80Ni12Bi8 for Sn-58Bi/Au/Ni-5.5wt%P/Cu, Sn-58Bi/Au/Ni-12wt%P/Cu and Sn-58Bi/Au/Ni/Cu, respectively. Elevated aging at 130∞C accelerates IMC growth rate and results in the formation of (Ni, Cu)3Sn4 and (Cu, Ni)6Sn5 adjacent to the ternary Sn-Ni-Bi IMC for the Sn-58Bi/Au/Ni-12wt%P/Cu and Sn-58Bi/Au/Ni/Cu joints, respectively. Cu contents in the (Cu, Ni)6Sn IMC are six times of those in the (Ni, Cu)3Sn4. Electroplated Ni fails to prevent Cu diffusion toward the Ni/solder interface as compared to EN-based joints. It is more favorable to employ Ni-12wt%P for the Sn-58Bi/Au/EN/Cu joint. Electroless nickel with a higher phosphorous content of 12wt%P is a better effective diffusion barrier during aging. In addition, P enrichment occurs near the interface of EN/solder and the degree of P enrichment is enhanced with the aging time.

10:55 AM
Solid-State Reactions Between the Au/Ni Surface Finish and the SnAgCu Lead-Free Solders with Different Cu Concentrations: L. C. Shiau[1]; Y. L. Lin[1]; W. C. Luo[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Cheml. & Matls. Eng., Chungli City 32054 Taiwan

The SnAgCu series of solders are very promising lead-free replacements for the Sn-37Pb solder, especially for reflow soldering applications. For industrial uses, a 0.2% uncertainty in composition is generally considered acceptable. However, our recent study revealed that, during reflow, the Cu concentration had a very strong effect on the reactions between SnAgCu solders and Au/Ni surface finish. With increasing Cu concentration in solder, the reaction product at the interface after reflow changed from a Ni3Sn4-based compound to a Cu6Sn5-based compound. In this study, we would like to extend our earlier study to investigate whether this strong concentration dependency also occurs during the solid-state aging of the solder joints. We aged solder joints at the solid-state at several different temperatures for time as long as 1000 hours. The solder compositions studied include Sn3.5Ag0.2Cu, Sn3.5Ag0.3Cu, Sn3.5Ag0.4Cu, Sn3.5Ag0.5Cu, Sn3.5Ag0.6Cu, Sn3.5Ag0.7Cu, and Sn3.5Ag1.0Cu. Analysis techniques used include optical microscope, SEM, EPMA, and XRD. It turned out that after solid-state aging, the dependence in Cu concentration disappeared. Detailed mechanism will be presented for this interesting phenomenon.

11:15 AM
Study of the Reaction Mechanism Between Electroless Ni-P and Sn and its Effect on the Crystallization of Ni-P: *Yoon Chul Sohn*[1]; Yu Jin[1]; Sung K. Kang[2]; Won Kyoung Choi[2]; Da-Yuan Shih[2]; [1]KAIST, Matls. Sci. & Eng., 373-1 Kusong-dong, Yusong-gu, Taejon 305-701 Korea; [2]IBM T. J. Watson Research Center, PO Box 218, Rt. 134, Yorktown Heights, NY 10598 USA

The reaction mechanism between electroless Ni-P and Sn has been investigated to understand the effects of Sn on the solder reaction-assisted crystallization at low temperatures as well as the self-crystallization of Ni-P at high temperatures. Ni_3Sn_4 starts to form in a solid-state reaction before Sn melts. Heat of reaction for Ni_3Sn_4 is measured during the Ni-P and Sn reaction, 241.2 J/g. It is found that solder reaction not only promotes crystallization at low temperatures by forming Ni_3P in P-rich layer but also facilitates self-crystallization of Ni-P by reducing the transformation temperature and heat of crystallization. The presence of Sn reduces the self-crystallization temperature of Ni-P by about 10°C. The heat of reaction for the self-crystallization decreases with an increased Sn thickness.

11:35 AM
Development of Electroplate Under Bump Metallization for Flip Chip Lead-Free Solder Bumping: *Su-Hyeon Kim*[1]; Jong-Yeon Kim[1]; Jin Yu[1]; [1]Korea Advanced Institute of Science and Technology, Ctr. for Elect. Pkgg. Matls., Matls. Sci. & Eng., 373-1 Guseong-dong, Yuseong-gu, Daejon 305-701 Korea

The recent transition from lead-bearing to lead-free solder bumping in flip chip packages requires the replacement of the conventional under bump metallization (UBM). In the midst of recently suggested UBM schemes, electropated UBM is still commonly used for electroplate solder bumping. However, when we use electroplated thick UBM in the chip side, the stress problem should be taken into account as well as the interfacial reaction with solders. In the present research, Cu/Ni-Cu alloy/Cu multi-layered UBM was newly developed. The Cu bottom layer is designed as a cushion layer to reduce the stress, the Ni-Cu alloy layer acts as a diffusion barrier, and the Cu finish can promote the solderability. We utilized a simple process to deposit both Cu and Ni-Cu alloy layers by electroplating in a single bath. The plated Cu/Ni-Cu alloy/Cu UBMs were characterized in terms of stress, interfacial reaction with lead-free solder, and electroplating character.

11:55 AM
Metallurgical Reaction Between Ni/Cu UBM and Lead-Free Sn-Ag Flip Chip Solder Bump: *Chien Sheng Huang*[1]; Jenq Gong Duh[1]; Yen Ming Chen[2]; [1]National Tsing Hua University, Dept. of Matl. Sci. & Eng., 101, Sect. 2 Kuang Fu Rd., Hsinchu 300 Taiwan; [2]National Chiao Tung University, Dept. of Matls. Sci. & Eng., 1001 Ta Hsueh Rd., Hsinchu 300 Taiwan

Recently, there are several international legislations to ban the use of Pb for the environmental concern. The eutectic Sn-Ag solder is one of strong candidates to replace the conventional Sn-Pb solder due to its excellent mechanical property. In this study, interfacial reaction between eutectic Sn-Ag and Ni/Cu under bump metallization (UBM) was investigated with a joint assembly of 96.5Sn-3.5Ag/Ni/Cu/Ti/Si3N4/Si multiplayer structure. After the first reflow, only one scallop-type (Ni1-x, Cux)3Sn4 IMC was found between solders and Ni. The thickness of IMC was about 1 mm. The concentration of Cu in (Ni1-x, Cux)3Sn4 was around 1.5 at.%. During multiple reflows, both thickness of IMC and Cu content in (Ni1-x, Cux)3Sn4 increased. In comparison, the thickness of (Ni1-x, Cux)3Sn4 formed in the Sn-Pb system remained almost the same despite the numbers of reflow. Correlations between the IMC morphology and Cu diffusion behavior in the joint assembly will be investigated with respect to the microstructural evolution due to interfacial reaction between the solder and the UBM.

Magnesium Technology 2003: Magnesium General Sessions

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

Thursday AM — Room: 2
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: Eric Nyberg, Pacific Northwest National Laboratory, Matls. Procg. Grp., Richland, WA 99352 USA; Zi-Kui Liu, Pennsylvania State University, Matls. Sci. & Eng., State College, PA 16082-5006 USA

8:30 AM
New Refractory Lining Concept at Dead Sea Magnesium Electrolysis Cells: *Zeev Goshen*[1]; Edward Anton Snajdr[2]; Peter Nebgen[3]; Manfred Rosch[4]; Eli Aghion[5]; Zeev Rubinowitz[6]; [1]Minerals & Refractories, Ltd., PO Box 106, 43 Haíazmauth St., Yehud 56100 Israel; [2]Vesuvius USA, Bettsville Rsrch. Ctr., 495 Emma St., PO Box 392, Bettsville, OH 44815 USA; [3]IKB GmbH, Rennweg/Klingelswiese 2, Postfach 1417, Andernach D-56604 Germany; [4]Refraline (pty), Ltd., 82 Bell St., Meadowdale Ext. 1, PO Box 8393, Germiston Edenglen 1613 S. Africa; [5]Dead Sea Magnesium, Magnesium Rsrch. Div., Potash House, PO Box 75, Beer Sheva 84100 Israel

Refractory lining in primary magnesium electrolysis cell is exposed to aggressive environment, therefore, needs to be periodically replaced and re-built. This constant refurbishment is costly, time consuming and halts production for the turnaround duration. The project (called ìTAMARî) objective was to completely redesign the cell refractory lining concept, materials, structure, and installation method. During the project, many advanced (but not necessarily expensive) refractory products were tested and selected for better durability. The cell refractory lining structure was re-designed to incorporate mostly pre-fab units, make use of up-to-date monolithic products, and enable quicker installation. Thus, trying to achieve: lower refurbishment costs, longer life expectancy, and shorter cell turnaround (idle) time. ìTAMARî Project took about two years. The international team, headed by Minerals & Refractories, Ltd. from Israel, included Vesuvius USA, IKB - Germany, Dead Sea Magnesium - Israel, and Refraline - South Africa. The project was partially funded by the BIRD Foundation. Recently, a prototype cell was installed at Dead Sea Magnesium and now operating and monitored.

9:00 AM
Explosion Weld Clad for Magnesium Melting Furnace Crucibles: *George A. Young*[1]; John G. Banker[1]; [1]DMC Clad Metal Division, 5405 Spine Rd., Boulder, CO 80301 USA

Large magnesium melting and alloying crucibles, referred to as mag pots, are typically constructed of cast iron or low carbon steel. The steel interior is resistant to attack by the molten magnesium. However, the exterior of the steel pot is subject to high temperature oxidation. Cleanliness and control of iron oxide accumulation is critical for safe operations. Clad pots consisting of an exterior layer of high temperature stainless steel or nickel alloy and a low carbon steel interior provide a number of operational benefits. These include improved safety, increased dimensional stability, and considerably longer service life, especially for gas fired furnaces. The explosion cladding technology has been used for clad pot manufacture since the mid-1980ís. The cladís metallurgical bond is not deleteriously affected by long term operation at high temperatures or the stresses of cyclic temperature exposure typical during mag pot operation. Test data indicate long term stability at up to 1000∞C. The presentation d iscusses the various metal alloys which are being used in magnesium pot applications, their performance attributes and limitations, clad fabrication techniques, and inspection methods. Pot design issues as relates to both performance and economics are discussed.

9:30 AM

Scale-Up of Magnesium New Rheocasting from a Laboratory Level to an Industrial Process: *Werner Fragner*[1]; Christian Peterlechner[1]; Ralph Potzinger[2]; [1]ARC Leichtmetall-kompetenzzentrum Ranshofen GmbH, Shaped Parts, AMAG-FVA-Gebaeude, Postfach 26, Ranshofen A-5282 Austria; [2]TCG Unitech AG, Lab. Mgr., Quality Mgr., Steiermaerker Strafle 49, Kirchdorf an der Krems A-4560 Austria

New Rheocasting (NRC) is a novel semi solid casting process for high quality castings. Originally designed for aluminum, laboratory trials on a 350 ton squeeze casting machine gave promise to introduce NRC to the magnesium market. However, scaling up to an industrial 800 ton plant demands consideration of several factors that differ from aluminum: Heat loss and alloy composition affect cycle time and process window. Process parameters such as casting speed, pressure, and temperature influence mechanical properties and microstructure. Varying composition in the Mg-Al-Zn system (AZ alloys) changes the NRC process window, which affects the ideal fraction liquid of the slug, i.e. castability. To provide the cast shop with the optimal casting temperature, a numerical approach was developed based on calculated phase diagrams. With proper machine and process modifications it is shown that Mg-NRC is a feasible way to produce sound Mg components on an industrial scale.

10:00 AM

Advances in Thixomolding Magnesium Part II: *Randy S. Beals*[1]; Steve LeBeau[2]; Yanzhong Wu[1]; Oscar E. Roberto[3]; P. Shashkov[1]; Simon Dewey[4]; [1]DaimlerChrysler Corporation, Matls. Eng. Dept. 5810, 800 Chrysler Dr., CIMS 481-01-41, Auburn Hills, MI 48326 USA; [2]Thixomat, 620 Technology Dr., Ann Arbor, MI 48108 USA; [3]Henkel Surface Technologies, Light Metals Grp./ANOMAG, 32100 Stephenson Hwy., Madison Heights, MI 48071 USA; [4]Keronite, Ltd., PO Box 700, Granta Park, Great Abington, Cambridge CB1 6ZY England

This report is a continuation of a previously published paper regarding ThixomoldingÆ magnesium alloys. The previous report discussed the characteristics of thixomolding magnesium alloys with low and high fraction percent solids. The effect of the amount of prior fraction solid on the physical properties of selected samples are further explored, with the percent solids in both AZ-91D and AM-60 B alloys evaluated over a range from 0% to 20%. The corrosion resistance of test plates that had been exposed to the Anomag and Keronite surface finishing technologies before and after 240 hour salt spray corrosion testing was examined in more detail than previously published. The surface features, cavitation and corrosion mechanics of the tested panels were analyzed and the results are discussed. The corrosion penetration at the interface between the scribe, coating and the material was also investigated. The corrosion behavior of the different coatings are discussed.

10:30 AM Break

10:45 AM

Low Cost SiCp/AZ91 Mg Composites Prepared by RCM: *Shae K. Kim*[1]; Hoon Cho[1]; Hyung-Ho Jo[1]; Young-Jig Kim[2]; [1]KITECH, Adv. Matl. R&D Ctr., 472, KaJwa 4 Dong, Seo-Ku, Incheon 404-254 Korea; [2]Sungkyunkwan University, Dept. of Adv. Matl. Eng., 300, ChunChun-Dong, JangAn-Ku, Suwon, GyeongGi-Do Korea

Magnesium metal matrix composites are gaining increased importance for transport and electronics applications where low inertia is required, with their improved stiffness, wear resistance, and elevated-temperature properties. Although the use of ceramic particle instead of fibrous reinforcements has help to reduce the over-all material cost, however, the processing-related cost should also be considered. This paper describes a novel low-cost molten metal mixing method, RCM (Rotation-Cylinder method), which has been developed for preparing SiC particle reinforced magnesium composites, with the aim of rapid incorporation and homogeneous distribution of reinforcement particles in an ambient atmosphere. RCM claims to significantly reduce the time required for incorporation and particle agglomerations by the U-shaped melt surface with the Rankine vortex. Sound magnesium composites can be produced in conjunction with subsequent investment casting or thixoforming processes. The development background, characteristic vortical motion, and on-going research strategy of RCM will be described.

11:15 AM

A Thermodynamic Database for Magnesium Alloys: *Marie Piche*[2]; Arthur Pelton[2]; Christine Brochu[1]; [1]Noranda, Noranda Tech. Ctr., 240 Hymus, Montreal, Quebec H9R1G5 Canada; [2]…cole Polytechnique de MontrÈal, Matls., MontrÈal H3C 3A7 Canada

A thermodynamic database for the Mg, Al, Mn, Fe, Be system as been developed at the CRCT (Centre for Research in Computational Thermochemistry), a research center based at the …cole Polytechnique de MontrÈal, in collaboration with Noranda Technology Centre for the MÈtallurgie Magnola, Inc. foundry. The goal of this work is to have a better understanding the chemistry of the alloying process of magnesium. The software used to make optimisations of the systems and to built the solutions and compound database is FACTSAGE 5.0. It permits to define the activity of each element of the system in the liquid and the solid phases and to calculate complex chemical equilibrium in a multicomponent system. The binary systems Al-Fe, Al-Mg, Al-Mn, Fe-Mg, Fe-Mn and Mg-Mn are taken from the COST 507 project, a thermochemical database for light metal alloys, and the binary systems with beryllium (Al-Be, Be-Fe, Be-Mg and Be-Mn) are evaluated in this work. The evaluation is made with literature data of phase equilibria, activitie s and thermodynamic properties of magnesium rich end data. For the multicomponent systems, they are interpolated from the binaries and optimised the same way. With the magnesium database, the software FACTSAGE 5.0 can plots phase diagrams, simulates the alloying process of magnesium for temperature between 500°C and 900°C and predicts the phase formation at each step. Magnesium producers can improved alloying method and increased the element recovery by calculating the distribution of the element between the magnesium liquid and the sludge, the heat required to maintain the furnace at constant temperature and the kind of intermediate phases formed during element addition.

11:45 AM

Orientation Dependence of Fatigue Crack Propagation in Magnesium Single Crystals: *Shinji Ando*[1]; Hideki Tonda[1]; [1]Kumamoto University, Dept. of Mechl. Eng. & Matls. Sci., Fac. of Eng., 2-39-1 Kurokami, Kumamoto 860-8555 Japan

The fatigue crack growth behavior of pure magnesium single crystals has been investigated in laboratory air at room temperature. Four type of compact specimens with different notch orientations were prepared from magnesium single crystal. Fatigue crack propagation behavior of each specimens were different related to notch orientation. In the case of A-specimen which notch plane and direction is (1210)[1010], a fatigue crack propagates parallel to notch plane. In B-specimen with (1010)[1210] notch, a fatigue crack propagates along to [1010] which inclined 30° to notch plane. In D-specimen with (1210)[0001] notch, a crack propagated to [0001] at low ΔK. The crack are inclined to [1210] gradually with increasing ΔK. In F-specimen with (0001)[1120] notch, a crack propagates parallel to basal plane with {1012} twin. Fatigue crack growth rate is lowest at B-specimen, and fastest at F-specimen.

Measurement and Interpretation of Internal/ Residual Stresses: SXRD on Composites

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, SMD-Mechanical Behavior of Materials-(Jt. ASM-MSCTS), MPMD-Shaping and Forming Committee

Program Organizers: Craig S. Hartley, Air Force Office of Scientific Research, Arlington, VA 22203 USA; Mark A.M. Bourke, Los Alamos National Laboratory, Neutron Science Center, Los Alamos, NM 87545 USA; Bimal K. Kad, University of California, Ames Laboratory, La Jolla, CA 92093-0085 USA

Thursday AM — Room: 17B
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: Mike Prime, Los Alamos National Laboratory, Los Alamos, NM 87545 USA; Bjorn Clausen, Los Alamos National Laboratory, LANSCE-12, Los Alamos, NM 84545 USA

8:30 AM Invited

The Use of High Energy X-Rays from the Advanced Photon Source to Study Stresses in Materials: *Dean R. Haeffner*[1]; [1]Argonne National Laboratory, APS, 9700 S. Cass Ave., Bldg. 431-A008, Argonne, IL 60439 USA

High-energy, third-generation synchrotrons, such as the Advanced Photon Source (APS), are well suited to produce very high fluxes of x-rays with energies above 40 keV. These so-called ìhigh-energyî x-rays are particularly well suited for studies of internal stresses and texture in materials. The low absorption of high-energy x-rays by most materials allows probing to depths comparable to that done with neutrons, but with much better spatial resolution and with much more beam intensity. The SRI-CAT high-energy x-ray program at the APS 1-ID beamline has been developing optics and experimental techniques for high-energy x-rays since the onset of the APS operations in 1995. Stress studies have been carried out on a variety of systems, including duplex steels, thick coatings, bulk metallic glasses, and composites. Examples from this research will be shown and plans for a future dedicated high-energy beamline (HEX-CAT) at the APS will be presented. Use of the Advanced Photon Source was supported by the US Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. W-31-109-Eng-38.

9:00 AM Invited

High Spatial Resolution Stress Measurements Using Synchrotron Based Scanning X-Ray Microdiffraction with White or Monochromatic Beam: *Nobumichi Tamura*[1]; Bryan C. Valek[2]; John C. Bravman[2]; Ralph Spolenak[3]; Wendel A. Caldwell[1]; Rich S. Celestre[1]; Alastair A. MacDowell[1]; Howard A. Padmore[1]; Jim R. Patel[1]; [1]Lawrence Berkeley National Laboratory, MS 2-400, 1 Cyclotron Rd., Berkeley, CA 94720 USA; [2]Stanford University, Dept. Matls. Sci. & Eng., Stanford, CA 94305 USA; [3]Max-Planck-Institut f¸r Metallforschung, Heisenbergstr. 3, Stuttgart D-70569 Germany

Scanning X-ray Microdiffraction (µ-SXRD) combines the use of high brilliance synchrotron sources with the state-of-the-art achromatic X-ray focusing optics and fast large area 2D detector technology. Using either white or monochromatic beams, it allows for orientation and strain/stress mapping of polycrystalline thin films with submicron spatial resolution. As an example of applications of the technique, the evolution of inter and intragranular stresses in micron size individual grains of a polycrystalline Al thin films deposited on Si, was monitored during an in-situ thermal cycling experiment. Strong stress and orientation variations were measured between grains and inside individual grains, which could be explained in terms of yield stress distribution and grain-to-grain interactions. If the µ-SXRD data are averaged over a macroscopic range, results show good agreement with macroscopic texture and stress measurements.

9:30 AM

Multi-Scale Characterization of Deformation Heterogeneities in Mo Single Crystal: *Erica Thea Lilleodden*[1]; Nobumichi Tamura[2]; David Lassila[3]; J. W. Morris[1]; [1]Lawrence Berkeley Laboratory, Matls. Sci., 1 Cyclotron Rd., MS 66-200, Berkeley, CA 94720 USA; [2]Lawrence Berkeley Laboratory, Adv. Light Source, 1 Cyclotron Rd., Berkeley, CA 94720 USA; [3]Lawrence Livermore National Laboratory, Eng., PO 808, L-113, Livermore, CA 94550 USA

High brilliance synchrotron sources used in conjunction with improved focusing optics have furthered the spatial resolution of x-ray diffraction measurements. A beamline at the Advanced Light Source at Lawrence Berkeley Laboratory, which is dedicated to microdiffraction experiments, is used in scanning mode to map out Laue diffraction patterns across the surface of a sample with a lateral resolution of 1 micron. These measurements are then analyzed in order to quantify the distribution of residual stress and strains in the sample. Furthermore, the distribution of dislocations in the sample can be elucidated from such measurements in conjunction with other scales of characterization. Here, we present preliminary findings on the spatial distribution of dislocations in a compression loaded Mo single crystal, and discuss the importance of X-ray microdiffraction in the hierarchy of multi-scale characterization of deformation behavior.

9:50 AM Break

10:05 AM

Microdiffraction Study of the Strains at Domain Walls in BaTiO3: *Robert C. Rogan*[1]; Ersan Ustundag[1]; Geoff Swift[1]; Nobumichi Tamura[1]; [1]Lawrence Berkeley National Laboratory, Adv. Light Source, Berkeley, CA 94720 USA; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA

The local strain fields around ferroelectric 90∫ domain walls in BaTiO3 have been investigated for the first time using the emerging technique of polychromatic scanning X-ray microdiffraction. A tetragonal BaTiO3 single crystal was scanned with a sub-micrometer X-ray beam to collect Laue patterns from regions around an individual domain. These patterns yielded information about orientation relationships across domain boundaries as well as the three-dimensional strain tensor associated with the domain. The results suggest the presence of significant residual elastic strain fields around domains in a nominally stress-free BaTiO3 crystal. The diffraction data will also be compared to and interpreted with micromechanical models.

10:25 AM

X-Ray Stress Analysis of Damage Evolution in Ti-SiC Composites: *Jay C. Hanan*[1]; Ersan Ustundag[1]; Irene J. Beyerlein[2]; Geoffrey A. Swift[1]; Jonathan D. Almer[3]; Ulrich Lienert[3]; Dean R. Haeffner[3]; [1]California Institute of Technology, Matls. Sci., 1200 E. California Blvd., MC 138-78, Pasadena, CA 91125 USA; [2]Los Alamos National Laboratory, Theoretl. Div., Los Alamos, NM 87545 USA; [3]Argonne National Laboratory, Adv. Photon Source, Argonne, IL 60439 USA

High energy X-ray microdiffraction was used to determine the elastic lattice strains of both phases in a Ti-SiC composite providing the in-situ load transfer under applied tensile stress at the scale of the microstructure. To understand the damage evolution, the measured strains were compared to those predicted by a modified shear lag model. Comparisons between the model and the data demonstrated the importance of accounting for the matrix axial and shear stiffness, provided an optimal stiffness ratio for load transfer and planar interpretation of the geometry in the composite, showed the matrix within and around the damage zone sustained axial load, and highlighted matrix yielding observed in the composite. It was also shown that an area detector is essential in such a study as it provides multiaxial strain data and helps eliminate the ìgraininessî problem.

10:45 AM

Internal Stresses and Orientation-Changes in Directional Solidification Microstructures: *Bernard Billia*[1]; Joseph Gastaldi[2]; Nathalie Bergeon[1]; Haik Jamgotchian[1]; Henri Nguyen Thi[1]; [1]L2MP, UMR CNRS 6137-University Aix-Marseille 3, Faculte des Sciences Saint-Jerome, Case 142, 13397 Marseille, Cedex 20 France; [2]CRMC2/CNRS, Campus de Luminy Case 913, 13288 Marseille, Cedex 9 France

In the processing of structural materials, all along alloy solidification from its melt internal stresses are generated in the solid by the composition variations that accompany microstructure formation. Directional solidification, which allows independent control of growth parameters, is used as model configuration. By means of in situ white-beam synchrotron X-ray topography, internal strains and stresses, and orientation-changes, created in the process of cellular/dendritic interface formation are followed in real time during directional solidification of thin Al-based binary alloys. With X-ray topography comprehensive information can be obtained by considering different reflecting planes. It follows that: -elastic strains giving specific contrasts precede visible interface corrugation, -cells are stressed due to non-uniform solute concentration in the solid, and -grain polygonisation results from changes in cell-body orientation, either suddenly or in a somewhat coherent manner. Based on mechanical shearing and bending, the phenomena can be analyzed, and modeling proposed.

11:05 AM

Distribution of Residual Microstresses: *Yu. Perlovich*[1]; M. Isaenkova[1]; H. J. Bunge[2]; [1]Moscow Engineering Physics Institute, Kashirskoe shosse 31, Moscow 115409 Russia; [2]TU Clausthal, Clausthal-Zellerfeld 38678 Germany

X-ray diffractometers of last generations allow to combine texture and microstress measurements. By use of the scanning regime or a position-sensitive detector, the X-ray line profile is registered for each point of the texture pole figure, so that additionally generalized pole figures of peak position 2q and line broadening b can be constructed. Though first attempts to obtain these pole figures were undertaken long ago, construction of distributions 2q(y,j) and b(y,j) became a routine procedure only recently, as both measurements and data treatment were completely automated. In the given work systematic studies of residual deformation effects in textured materials resulted in determination of main principles, controlling the above distributions. The distribution of elastic microstrains depending on grain orientation was studied in details as applied to various rolled metal materials (steels, Nb, Mo, Cu, Zr-alloys, Ti, single crystals Ti-Ni) having textures of different types. For the sake of the effective systematization of obtained experimental data, measured values of the Bragg angle 2qhkl(y,j) were recalculated into values of the interplanar spacing dhkl and then - into values [dhkl(y,j) - dav)/dav, that is the relative deviation of interplanar spacing along the direction (y,j) from the average weighted level dav. The average level is admitted to be conditioned by elastic macrostresses (stresses of 1st kind) acting within the irradiated volume, whereas local fluctuations about this level are connected with elastic microstresses (stresses of 2nd kind) of opposite signs. By the definition, residual stresses of the 2nd kind, or elastic microstresses, are equilibrated within a volume of the group of neighbouring grains, which to a first approximation corresponds to the volume participating in X-ray diffraction by the texture measurement. The analysis of experimental data shows, that the actual variant of stress equilibrium in rolled metals is connected with crystallographic orientations of grains and with their position relative to texture maxima and minima as well as to the planes of symmetry of the pole figure. Most often the distribution of elastic microstresses shows a cross-wise character, consisting in alteration of the predominant sign (≤+≤ or ≤-≤) of microstresses, when passing from one quadrant of the pole figure to another. As a result, elastic microstresses of opposite signs prove to be equilibrated about both longitudinal and transverse planes. In cases of simple rolling textures the distribution of residual microstrains acquires some additional features, which seem to be a remarkable manifestation of the tendency to stress equilibrium: - when the rolling texture contains a single component (for example, in the case of the rolled single crystal Ti-Ni), this component breaks in half, that is in two close subcomponents, which differ in signs of the elastic strain along the same direction; - when the rolling texture consists of two crystallographically equivalent components, the equilibrium of elastic microstresses is ensured by the fact, that deformation tensors for their crystallites prove to be mutually opposite; - in the case of the rolling texture with a significant axiality the stretched zones of elastic compression and extension are situated at opposite slopes of texture maxima.

Mercury Management: Remediation and Fundamentals

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

Thursday AM — Room: 1B
March 6, 2003 — Location: San Diego Convention Center

Session Chairs: Jerry Downey, Hazen Research Inc., Golden, CO 80403-1848 USA; Charles Gale, Summit Valley Engineering and Equipment, W. Bountiful, UT 84087 USA

8:30 AM Invited
Process Development Strategies for Mercury Remediation: *Jerome P. Downey*[1]; Guy L. Fredrickson[1]; [1]Hazen Research, Inc., 4601 Indiana St., Golden, CO 80403-1848 USA

The technologies developed within the field of extractive metallurgy to recover, concentrate, and purify metals and industrial minerals from ores, have served as the foundation for all of the subsequent environmental remediation, materials recycling, and waste management technologies. This paper discusses the application of classical extractive engineering principles to the development of a thermal process for the recovery of mercury from solid wastes. Process development is presented as a series of logical stages, which begin with a thorough characterization of the site-specific waste, move on to bench and pilot scale testing of the process options, and end with a functional process flowsheet. This paper also presents thermodynamic data on many mercury species of interest to the minerals, power, and chemical industries. These data can be used to model the speciation and recovery of the mercury as a function of various process parameters.

9:00 AM Invited
Selection and Demonstration of a Process for Mercury Remediation in Soils: A Case Study: *Charles W. Kenney*[1]; [1]Hazen Research, Inc., 4601 Indiana St., Golden, CO 80403-1848 USA

Mercury contamination of soil at an old chemical plant surrounded by a residential community and adjacent to a river has provided unique challenges for the current owner. Inorganic and organic mercury compounds accumulated over the past 50 years to levels that require remediation. Hazen Research, Inc. conducted a multi-phased program to characterize contaminants and their occurrences, evaluate various treatment technologies, and demonstrate the selected technology at bench and pilot scales. A combined physical cleaning and chemical treatment was demonstrated to meet treatment goals for the site. This paper discusses the methods of process selection, and results of the bench- and pilot-scale testing.

9:30 AM Invited
Treatment of Mercury-Contaminated Soil, Mine Waste and Sludge Using Silica Micro-Encapsulation: *Amy Anderson*[1]; [1]KEECO, Lynnwood, WA USA

The Silica Micro-Encapsulation (SME) Technology has been utilized on three separate projects focused upon the stabilization of mercury-contaminated materials. At the Mother Lode Mine in Oregon approximately 450 cubic yards of soil were amended with the chemical using a backhoe and nominal amounts of water to promote the reaction. Results collected via EPA Method 1311 TCLP, exhibited leachable mercury concentrations below the treatment goal of 200 parts per billion (ppb); actual concentrations ranged from 0.8 ppb to 2.6 ppb. In a laboratory study jointly sponsored by the EPA Mine Waste Technology Program and Superfund Innovative Technology Evaluation (SITE) Demonstration Program the SME Technology, an inorganic sulfide chemical and a generic phosphate treatment were tested for their ability to treat mine waste from the Sulfur Bank Mercury Mine. Technical and economic performance of the silica-based technology exceeded the two competing technologies, achieving an 88% reduction in total leachable mercury and a greater than 99% reduction in the particulate-associated mercury as compared to the control samples. Costs of treatment were estimated at $18.26 per metric ton. At a former chloralkali plant in British Columbia, dredged lagoon sludge was amended with the SME chemical to reduce leachable mercury concentrations to 0.150 ppm or less, based on EPA Method 1311 TCLP. Results from laboratory and field tests established an optimum chemical dosage rate of 3% chemical by dry weight of sludge. Samples were evaluated during field tests for weight, temperature, mercury vapor and leachable mercury. Results exhibited a 75% reduction in leachable mercury as compared to the control and satisfied the project treatment goals. Operating costs were estimated at $15.00 per metric ton.

10:00 AM Break

10:15 AM Invited
Utilization of an Equilibrium Calculational Program to Demonstrate the Hydrometallurgical Treatment of Mercury Wastes: *Hsin H. Huang*[1]; Larry G. Twidwell[1]; Courtney A. Young[1]; [1]Montana Tech of the University of Montana, Sch. of Mines & Eng., 1300 W. Park, Butte, MT 59701 USA

Equilibrium calculational software programs can be effectively utilized to model and to understand the behavior of mercury. The equilibrium calculation program STABCAL will be demonstrated. The illustrated diagrams include Eh-pH, activity or concentration as a function of pH, Eh or additional ligand concentration. The resulting diagrams and simulations will be presented to demonstrate the fundamentals of geological deposition processes, pyro- and hydrometallurgical and environmental treatments and recovery, transpositon between aqueous and gaseous phases and mercury dispersal in the environment.

10:45 AM Invited
Mercury, Lead and Cadmium Removal from Aqueous Streams Using Silica Polyamine Composites: *Edward Rosenberg*[1]; Robert Fischer[1]; Carolyn Hart[1]; Purity Systems, Inc.[2]; [1]University of Montana, Dept. of Chem., Missoula, MT 59812 USA; [2]3116 Old Pond Rd., Missoula, MT 59802 USA

New composite materials have been developed that are particularly effective for the removal of lead, cadmium and mercury from low levels (i.e. National Sanitary Foundation recommended challenges: 150, 30 and 6 micrograms/L respectively) to the allowable release levels (15, 6 and 2 micrograms/L respectively). The synthesis and physical characteristics of these materials will be described. Data on idle column leaching will be presented and the various methods attempted for regeneration of the columns will be discussed. Conditions for the application of these materials to the recovery and remediation of these metals will be recommended.

11:15 AM
Mercury Spill Response: *Lise Mercier*[2]; *Neal Langerman*[1]; [1]Advanced Chemical Safety, 7563 Convoy Ct., San Diego, CA 92111 USA; [2]EPS Chemicals, Inc., 161-145 Tyee Dr., Pt. Roberts, WA 98281 USA

Mercury use, in thermometers, switches, floating contacts, or other applications, results in spills. These spills pose a long-term toxicity hazard to employees, their families, and the environment. Prompt response and efficient clean-up is essential. Spills from thermometers and switches are generally small and can be efficiently handled by area workers using a MERCONô Spill Kit. As the amount of mercury spilled increases, the knowledge and protection for the responders increase and the use of more sophisticated clean-up and decontamination tools is required. Responders to a large mercury spill must be trained under the OSHA ìHazardous Waste Operations and Emergency Responseî (29 CFR 1910.1209q) standard. In addition to personal protective equipment, responders must have a mercury vacuum, a mercury vapor detector, and an adequate supply of MERCON-Xô. The safe and effective use of these procedures and tools will be illustrated with actual examples.

Microstructural Processes in Irradiated Materials: Fusion Reactor Materials

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

Thursday AM Room: 11A
March 6, 2003 Location: San Diego Convention Center

Session Chairs: David Gelles, Pacific Northwest National Laboratory, Richland, WA 99352 USA; Robin Schaeublin, EPFL, CRPP, Villigen AG 5232 Switzerland

8:30 AM
Irradiated Microstructrures in Fusion Reactor Materials: *Steven J. Zinkle*[1]; [1]Oak Ridge National Laboratory, Metals & Ceram. Div., PO Box 2008, Oak Ridge, TN 37831 USA

Microstructural studies are indispensable for determining fundamental physical processes that occur in materials during energetic particle irradiation. The major materials systems of interest for fusion reactor applications include body centered cubic metallic alloys (ferritic/martensitic steels, vanadium alloys, Mo alloys), SiC/SiC and carbon composites, and ceramic insulators. Cu alloys and austenitic stainless steel are also of interest, in particular for near-term proposed next-step burning plasma experiments such as ITER. This presentation will review recent electron microscopy results on irradiated materials, with an emphasis on the fluence and temperature-dependent accumulation of defect clusters, and in-situ and post-deformation observations of irradiated and deformed metals. Intrinsic differences between the defect accumulation behavior of BCC and FCC metals will be highlighted. Recent experimental observations on flow localization due to plastic deformation of quenched and irradiated metals (in particular, twinning and dislocation channeling), and studies of the detailed dislocation-defect cluster interaction mechanisms will be summarized.

9:15 AM
A Comparison of Helium Implanted Iron and EUROFER97: Effects of He Dose: *Morten M. Eldrup*[1]; Bachu N. Singh[1]; Peter Jung[2]; [1]Risoe National Laboratory, Matls. Rsrch., Roskilde DK-4000 Denmark; [2]Forschungszentrum J¸lich, Association EURATOM-FZJ, Institut f¸r Festkˆrperforschung, J¸lich D-52425 Germany

This work is part of an effort to investigate, experimentally as well as theoretically, the role of He in cavity nucleation in neutron irradiated iron and steel. We have already reported results on cavity formation at low temperatures and at low neutron doses in iron and steel without He-implantation. Pure iron and EUROFER97 steel will be homogeneously implanted with He at 50C and 350C to doses of 1, 10 and 100 atppm and subsequently neutron irradiated at the implantation temperatures. The microstructure after He implantation and neutron irradiation will be investigated, as well as the annealing behaviour of some selected specimens. Positron annihilation spectroscopy (PAS), which is sensitive to cavities in the size range from single vacancies to several nano-metres, will be used as the main characterisation technique. The present contribution will describe and discuss the results of the studies of the as-implanted samples.

9:35 AM
Post-Irradiation Deformation of Ferritic Steels: *David S. Gelles*[1]; [1]Pacific Northwest National Laboratory, P8-15, PO Box 999, Richland, WA 99352 USA

In order to understand the processes of post-irradiation deformation in ferritic steels, the microstructures of deformed tensile specimens of Fe-9Cr and F82H have been examined by transmission electron microscopy in order to study dislocation behavior during and after channel deformation. It is found that channel deformation occurs in Fe-9Cr after irradiation to between 10 and 40 dpa at ~400∫C, whereas no channel deformation is found in F82H (Fe-7.5Cr-2W-0.1C) following irradiation to 2.6 dpa at 330∫C. Microstructural studies reveal that a<100> loops formed by irradiation in Fe-9Cr can easily be removed by intersection with moving individual a/2 <111> dislocations, whereas irradiation produced loop damage in F82H is very limited and has little effect on moving slip dislocations. The dislocation interaction mechanisms will be carefully described in order to provide a basis for modeling post-irradiation deformation behavior.

9:55 AM Break

10:25 AM
Dislocation-Defect Interactions in Materials: *Joshua S. Robach*[1]; Athanasios Arsenlis[2]; Ian M. Robertson[1]; Brian D. Wirth[2]; [1]University of Illinois, Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA; [2]Lawrence Livermore National Laboratory, Matls. Sci. & Tech. Div., PO Box 808, L-353, Livermore, CA 94551 USA

In order to develop predictive models of the mechanical response of irradiated materials it is necessary to understand the fundamental physical processes controlling the deformation. This is particularly important near yielding where local defect interactions, which cannot be averaged, may dominate the behavior. Samples of a range of materials containing various densities and distributions of stacking-fault tetrahedra and small dislocation loops were examined using the in-situ TEM straining technique. In this paper, the dynamics of the interaction of dislocations with these defects are presented. Defect annihilation mechanisms as well as the conditions required to produce defect-free channels are proposed. The experimental results are compared to atomistic simulations of unit interactions. Based on these new observations an improved continuum model is presented.

10:45 AM
Irradiation Induced Stacking Fault Tetrahedra in fcc Metals: *Robin Sch‰ublin*[1]; Nadine Baluc[1]; Maximo Victoria[1]; [1]Swiss Federal Institute of Technology Lausanne, CRPP Fusion Tech.-Matls., Villigen PSI 5232 Switzerland

Irradiation can induce the formation of stacking fault tetrahedra (SFTs) in a number of fcc metals, such as stainless steel and pure copper. In order to understand the role of the materialís parameters on this formation, pure Cu, Ni, Pd and Al, having a stacking fault energy of about 70, 140, 180 and 200 mJ/m2, have been irradiated with high energy protons to doses ranging from 10-4 to 10-1 dpa at room temperature. The irradiation induced microstructure has been investigated using transmission electron microscopy. It appears that all irradiated metals but Al present SFTs. Their formation energy as a function of size has been calculated using elasticity of the continuum, with respect to the formation of a number of other possible defect configurations. It appears that the key parameters are the stacking fault energy and the shear modulus. Their implication on the formation of the SFTs under irradiation is discussed.

11:05 AM
Effects of Irradiation on the EUROFER 97 RAFM Steel: *Nadine L. Baluc*[1]; Robin E. Sch‰ublin[1]; Philippe Sp‰tig[1]; Max Victoria[1]; [1]Centre of Research in Plasma Physics, Fusion Tech. Matls., Swiss Federal Inst. of Tech.-Lausanne, Assoc. EURATOM-Swiss Confederation, Villigen-PSI, AG 5232 Switzerland

Series of Charpy and tensile flat specimens have been prepared from a plate of the reduced activation ferritic/martensitic (RAFM) steel EUROFER 97 (Fe-9CrWVTa). A part of them were irradiated with 590 MeV protons in the Proton Irradiation Experiment (PIREX) facility at the Paul Scherrer Institute. The irradiations were performed at ambient temperature (300-320 K), 523 K and 623 K to doses ranging between 0.3 and 2 dpa. Charpy impact tests, tensile deformation experiments were performed over a wide range of temperatures on the unirradiated and irradiated specimens. The defects associated with irradiation were imaged in transmission electron microscopy by using the bright/dark field and weak beam techniques. The whole set of results will be presented in comparison with those previously obtained for the F82H and OPTIMAX series of RAFM steels.

11:25 AM
Characterization and Modeling of Helium Bubbles in Self-Irradiated Plutonium Alloys: *Adam J. Schwartz*[1]; Mark A. Wall[1]; Charlene M. Schaldach[1]; Wilhelm G. Wolfer[1]; [1]Lawrence Livermore National Laboratory, CMS, L-355, 7000 East Ave., Livermore, CA 94550 USA

The characterization of microstructural changes in Pu-Ga alloys resulting from aging phenomena is an important technical challenge to the nuclear Stockpile Stewardship program. We have identified at least two age-related phenomena that may occur in Pu alloys, dimensional changes due to the initial transient, helium accumulation, and void swelling, and phase instability. The initial transient is a well-known effect that results from the initial cascade damage. This form of dimensional change tends to saturate within approximately two years. A second contributor to dimensional change is the build-up of helium as a result of the alpha decay. Helium is generated at a rate of approximately 40 parts per million per year as a result of alpha decay.

Transmission electron microscopy observations indicate a high density of small nanometer sized bubbles. We will describe a model for the nucleation and growth of the He bubbles with simple rate equations. This work was performed under the auspices of the US Department of Energy by the University of California, Lawrence Livermore National Laboratory under Contract No. W-7405-Eng-48.

Science and Technology of Magnetic and Electronic Nanostructures: Nanoscale Probes

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Jt. EMPMD/SMD-Chemistry & Physics of Materials Committee
Program Organizers: Ramamoorthy Ramesh, University of Maryland, Department of Materials and Nuclear Engineering, College Park, MD 20742 USA; Y. Austin Chang, University of Wisconsin-Madison, Department of Materials Science & Engineering, Madison, WI 53706-1595 USA; Robert D. Shull, NIST, Magnetic Materials, Gaithersburg, MD 20899-8552 USA

Thursday AM — Room: 15A
March 6, 2003 — Location: San Diego Convention Center

Session Chair: ChangBeom Eom, University of Wisconsin, Dept. of Matls. Sci. & Eng., Madison, WI 27708-0300 USA

8:30 AM
Control of Carbon Nanotube Configuration for Nano-Device Applications: *Sungho Jin*[1]; [1]University of California-San Diego, La Jolla, CA 92130-0411 USA

Carbon nanotubes are fascinating new nano materials with many interesting physical, chemical and electronic properties and potential new applications. They have a graphitic tubular structure with nanometer-scale diameter and very large aspect ratio. For most of the possible technical applications, a well-aligned growth structure, rather than a random, tangled configuration, is desired. While a dense, forest-like alignment is relatively easily obtained, the growth of low-density yet aligned carbon nanotubes with a specific diameter and length at specific nano-scale locations is not easy. Various possible approaches of nucleation control for growth of such desired configuration of carbon nanotubes will be discussed in relation to some potential electronic applications.

9:15 AM
Opportunities in Nanomagnetism: *Samuel D. Bader*[1]; [1]Argonne National Laboratory, Matls. Sci. Div., Argonne, IL 60439 USA

Magnetism is one of the oldest fields in science but it is also at the forefront of the nanotechnology revolution. This talk draws on historical roots to provide a modern experimental perspective on recent issues and challenges in the field. Highlighted areas include the quest for magneto-electronics and the importance of novel interfacial magnetic coupling phenomena. Examples of how nanotechnology provides new strategies to realize composite magnetic materials with exceptional properties and interesting physics will be presented.

10:00 AM
Nanoscale Piezoelectric Imaging and Piezoelectric Nanostructures: *Marin Alexe*[1]; [1]Max Planck Institute of Microstructure Physics, Halle D-06120 Germany

One of the challenging target of the ferroelectric community is to demonstrate that high density devices based on ferroelectric oxides are feasible and there are no technological or basic physical phenomena which will restrict the downscaling of the ferroelectric oxides toward the nanoscale level, i.e. dimensions in the range of 20-100 nm. The present talk addresses simultaneously both issues of patterning of crystalline oxide structures and characterization of ferroelectric at nanoscale levels. Patterning of ferroelectric structures down to several tens of nanometers using both ìtop-downî and ìbottom-upî approaches based on electron-beam lithography and self-assembly methods, respectively will be presented. Ferroelectric, piezoelectric and switching measurements on nanoscale structures and thin films using scanning probe microscope (SPM) working in piezoresponse mode will be also broadly discussed.

10:45 AM
Magnetic Force Microscopy of Nanostructured Materials: *R. D. Gomez*[1]; [1]University of Maryland, Dept. of Electl. & Compu. Eng., College Park, MD 20742 USA

In its invention in 1987, magnetic force microscopy (MFM) has become a dominant tool for magnetic characterization at the ten-nanometer length scales. It is a robust instrument, operable in air, and uses very little surface preparation. Additionally, MFM images obey a straightforward instrument response function, from which the field producing component of the magnetization can be readily extracted. In comparison with other high-resolution imaging techniques, MFM has had the highest impact towards understanding magnetism at the local regime. Magnetic nanostructured materials, which I define as artificially synthesized particles or particle arrays under hundred nm in lateral dimensions, is a field that is greatly influenced by MFM. Among the many conceived applications of these nanoparticles, the technologically vigorous areas include ultrahigh density data storage, magnetic random access memory devices, and biological tagging and detection. In these applications, the keys to successful device development are the understanding of magnetic domains and the behavior with applied fields, the distribution of switching fields, the influence of particle-particle interactions, and spin polarized electron transport. In this talk, I will review the basics of MFM as well as discuss some of the recent advances we developed to enhance its functionality. I will present some of our work on imaging in in-situ applied fields, in ultra-high vacuum, at low temperatures, as well as, driving domain walls using high speed electronic pulses. I will present specific examples and highlight some new insights and processes on small magnetic particles.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Advances in Materials Processing Technologies III: Casting and Aluminium Processing

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors
Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Thursday AM — Room: Point Loma
March 6, 2003 — Location: San Diego Marriott Hotel

Session Chairs: W. D. Cho, University of Utah, Dept. of Metallurgl. Eng., Salt Lake City, UT 84112 USA; V. M. Kevorkijan, Independent Researching Slovenia

8:30 AM
Modeling of Melting Behavior of Aluminium Metal in Molten Salt and Metal Bath: *B. Zhou*[1]; Y. Yang[1]; M. A. Reuter[1]; [1]Delft University of Technology, Dept. of Appl. Earth Scis.,, Mijnbouwstraat 120, Delft 2628 RX The Netherlands

During secondary aluminium recovery, complex aluminium scraps are melted and refined often in a rotary melting furnace, and the aluminium metal has to pass through a molten salt layer and melt down in a bottom aluminium bath. An experimental study and industrial observations have indicated that salt shell formation and re-melting on the metal solids in the early stage plays a critical role in the overall melting process of the scrap. In the present paper, mathematical models are developed to simulate the process of salt shell formation and its re-melting, as well as aluminium melting in the molten melt. Influence of the salt layer properties, the residence time of the metal in the salt layer, the particle size and shape, and the temperature of melt were investigated. Reasonable agreement with the measurement data was reached. The developed melting model will be used to construct a population balance model of the scrap melting behaviour for a rotary melting furnace, taking into account the distributed characteristics of the scrap. Finally, an overall process model based on Computational Fluid Dynamics (CFD) for the rotary furnace will be constructed, coupling with the population balance model for the scrap melting.

8:55 AM
Electrical Noise Analysis of Industrial Aluminium Smelting Cells: *A. Agnihotri*[1]; [1]Jawaharlal Nehru Aluminium Research Development and Design Centre, Wadi, Nagpur 440023 India

One of the interesting research endeavours in the aluminium reduction industry has been resistance/voltage variation in aluminium reduction pots. Measurement of cell voltage and current simultaneously were conducted in actual plant with high-speed data acquisition system. It was found that the amplitude, frequency, etc. of cell resistance provided valuable information of anode bottom, metal rolling, cell temperature, etc. Special measurements were made in cells at various states of disturbances viz. uneven anode bottom, before anode effect, during anode effect, after anode effect. The resistance/voltage data was analysed at these cell states and electrical noise patterns were studied. The study provided valuable information on cell state by means of data, which is generally averaged out in normal plant operation. Statistical analysis of data obtained provides further scope in evaluating the status of process and detecting operational problems like uneven anode bottom, hydro-dynamic instability, etc. Data analysis deserves more attention as they can be used in predicting the cell stability and operating parameters at very early stage. Details of these are presented in this paper.

9:20 AM
Theoretical Bases of High-Efficient Purification of Molten Aluminum and Purification Practice: *Gaosheng Fu*[1]; Jixing Kang[1]; Wenzhe Chen[1]; Kuangwu Qian[1]; [1]Fuzhou University, Dept. of Mechl. Eng., Fuzhou 350002 China

In order to obtain aluminum products with higher quality, the harm of metallurgical defects such as inclusions (Al2O3) and hydrogen must be eliminated or weakened before pouring molten aluminum into mold, because their presence will have direct or indirect influences on strengths, deformation properties and performances of aluminum products. The effective purification treatment of molten aluminum is the key to improving melt cleanness of aluminum. In this paper, based on the analyses of the essences of behavior of inclusions and hydrogen in molten aluminum and their interactive relationship, which are the theoretical bases of high-efficient purification of molten aluminum and are of the utmost importance in determining the technology of purification of molten aluminum, the interactive ìparasitic mechanismî between inclusions and hydrogen in molten aluminum is put forward. At the same time, aimed at the currently existing problems in the methods of purification, the principle for purification of molten aluminum, that is, ìremoving inclusions is the basis for eliminating hydrogen and, therefore, more attention should be firstly paid on removing inclusionsî is firstly put forward, thus breaking through the trammel of traditional ideas of purification i.e., more attention is firstly paid on eliminating hydrogen. The principle has been tested and verified in authorsí researches and practices. According to this principle above, a new method of purification by filtrating with flux and a corresponding high-efficient flux for removing inclusions have been developed, which has been successfully applied to productions of some high-performance aluminum sheets such as aluminum sheet used for pressure can, etc. It is found that with special flux(CJ-5) for removing inclusions and filtration technology, and proper addition amount and melting temperature, the rate of removing inclusions and the extent of lowering porosity for different grades commercial purity aluminum could amount to about 70-82% and 60-88% respectively, and mechanical properties of this material were improved remarkably, especially the relative increase of elongation(d) was about 70% in comparison to that of no filtration.

9:45 AM
Original Industrial Application of Two Numerical Models in Concasting Technology: Frantisek Kavicka[1]; *Josef Stetina*[1]; [1]Brno University of Technology, Fac. of Mechl. Eng., Technicka 2, Brno 616 69 Czech Republic

Solidification and cooling of a continuously cast slab and simultaneous heating of a crystallizer is a very complicated problem of transient heat and mass transfer. Nowadays, the solving of such a problem is impossible without numerical models of the temperature field not only of the slab itself, while it is being processed through the whole concasting machine (CCM), but of the crystallizer as well. Two original numerical models have been developed and used in the investigation of a continuously cast steel slab. The first (one of two) 3D model of the temperature field of a concasting is capable of simulating the temperature field of a caster. Experimental research and data acquisition have to be conducted simultaneously with the numerical computation-not only to confront it with the actual numerical model, but also to make it more accurate throughout the process. After computation, it is possible to obtain the temperatures at each node of the network, and at each time of the process. The utilization of the numerical model of solidification and cooling of a concasting plays an indispensable role in practice. The potential change of technology-on the basis of computation-is constantly guided by the effort to optimize, i.e. to maximize the quality of the process. The user can therefore choose any appropriate longitudinal or cross-section of a slab and display or print the temperature field in a 3D or 2D graph whenever necessary. The second numerical model of dendritic segregation of elements assesses critical points of slabs from the viewpoint of their increased susceptibility to crack and fissure. In order to apply this model, it is necessary to analyse the heterogeneity of samples of the constituent elements (Mn, Si and others) and impurities (P, S and others) in characteristic places of the solidifying slab. The numerical model, based on measurement results obtained by an electron micro-probe, generates distribution curves showing the dendritic segregation of the analysed element, together with the distribution coefficients of the elements between the liquid and solid states. The combination of both models enables the prediction of cracks and fissures in critical points of the continuously cast carbon-steel slab. Both models had been applied in the industrial investigation of a cast low-carbon-steel slab.

10:10 AM Break

10:30 AM
Study on the Energy Absorption Property of Foaming Alminium: *Yihan Liu*[1]; Guangchuen Yao[1]; Xiaoming Zhang[1]; [1]Northeastern University, Sch. of Matls. & Metall., Shenyang 110004 China

Some different density foaming aluminium samples were prepared in the semi-industrial scale experiment system. Their properties such as energy absorption characteristics, mechanical properties, heat conductivity and acoustical properties were detected. The results showed that the foaming aluminium density from 0.5 to 0.6 g/cm3 have the better energy absorption, suit to be the ensuring bar of the car, and also suit to be the cushion materials for the engine. The foaming aluminium density from 0.25 to 0.4g/cm3 have the better acoustical properties and heat conductivity resistance, suit to be the filling material for the door, the ceiling and the partition of the car engine.

10:55 AM
Study on Formation and Magnetic Susceptibility of Intermetallic Compound Containing Iron, Manganese in the Aluminum Melts: *Guangchun Yao*[1]; Linli Wu[1]; Lei Zhang[1]; [1]Northeastern University, The Key Lab. of Electromagnetic Procg. of Matls., Shenyang 110004 China

Iron and silicon impurities would form needle and branch shape intermetallic compounds in the molten aluminum when there was no manganese. It is difficult to separate them from molten aluminum by electromagnetic separation. The Fe-Si-Al and Fe-Al intermetallic compounds were transform into Mn-Fe-Si-Al or Mn-Fe-Al compounds when the manganese was added into the molten aluminum. The metallograph show that the shape of these intermetallic compound particles is lumpish and spherical shape. These impurity particles will suffer less resistance when they were migrated in the molten aluminum and it is benefit to be separated. Some $FeSiMnAl_4$ and $FeMnAl_6$ were paramagnetic materials. Their impurity particles could be migrated by the magnetic force and could be separated from the molten aluminum.

11:20 AM
Study on the Factors Affecting Separation of Iron Impurity from Molten Aluminum by Altering Induced-Magnetic Field: *Guangchun Yao*[1]; Lei Zhang[1]; Linli Wu[1]; [1]Northeastern University, The Key Lab. of Electromagnetic Procg. of Matls., Shenyang 110004 China

There are branching and needle-shape intermetallic compounds composed of iron, silicon and aluminum in the molten aluminum. When manganese metal was adding into molten aluminum in a suitable proportion, the shape of the intermallic compound particles may become lumpish and spherical. Depend on the difference of these impurity particles and molten aluminum in electric and magnetic properties, these impurity intermetallic compound particles can be separated by altering magnetic field. In this experiment the altering magnetic field was formed by a ìCî type loop which AC (500Hz) current went through. The inner vacum of the separator made of fireclay refractory materials was 25◊15◊130mm^3 with a lateral baffle inside. The separator was put into the interspace of the ìCî type loop. The experiments of separating iron-rich impurity particles were conduced in different separate time, magnetic intensity and adding manganese respectively. It was found that the longer the separating time was the larger transfe rred amount of impurities would be within three minutes, the transferred amount of impurities increased with the magnetic field intensity strengthened, the separating impurities effect from molten aluminum was increased obviously by adding manganese metal.

Yazawa International Symposium on Metallurgical and Materials Processing: Principles and Technologies: Experimental Techniques

Sponsored by: Extraction & Processing Division, EPD-Process Fundamentals Committee, EPD-Pyrometallurgy Committee, EPD-Aqueous Processing Committee, EPD-Copper, Nickel, Cobalt Committee, EPD-Lead and Zinc Committee, Jt. MPMD/EPD-Process Modeling Analysis & Control Committee; See Plenary Session for Co-Sponsors
Program Organizers: Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA; Kimio Itagaki, Tohoku University, Institute for Advanced Materials, Sendai 980-8577 Japan; Florian Kongoli, FLOGEN Technologies, Inc., Materials Technology Department, Montreal, Quebec H3S 2CS Canada; Chikabumi Yamauchi, Nagoya University, Department of Materials Science & Engineering, Nagoya 464 8603 Japan

Thursday AM	Room: Solana
March 6, 2003	Location: San Diego Marriott Hotel

Session Chairs: Roberto Parra, Universidad de ConcepciÛn, Dept. of Metallurgl. Eng., ConcepciÛn Chile; Takeshi Azakami, Saitama Institute of Technology, Mechl. Eng., Saitama Japan

8:30 AM
Recent Progress in Thermal Diffusivity Measurement of Molten Oxides by the Laser Flash Method: *Hiromichi Ohta*[1]; Shibata Hiroyuki[2]; Yoshio Waseda[2]; [1]Ibaraki University, Dept. of Matls. Sci., Hitachi 316-8511 Japan; [2]Tohoku University, Inst. of Multidisciplinary Rsrch. for Adv. Matls. (IMRAM), Sendai 980-8577 Japan

Precise thermal properties of molten materials are essentially required for analyzing the process control in metal production. These properties are also important as a clue to consider the dynamic structure of liquid materials. However, it is difficult to obtain the precise thermal diffusivity values of molten materials at high temperature because of the effect of bubble formation and the severe separation of radiative and conductive heat components. We have developed many types of laser flash technique to measure thermal diffusivity of molten oxide such as three layered cell system composed of metal plate/liquid phase/metal crucible, two layered cell system of liquid phase/metal crucible and differential data analysis to estimate the sample thickness as well as radiative heat flow. The main purpose of this paper is to present a novel experimental technique recently developed for measuring thermal diffusivity of molten oxides at high temperatures. In this new technique, the sample is melted in a metal crucibl e and heated to the desired temperature. A single laser pulse is flashed on the bottom of the crucible and infrared ray irradiated from the same bottom surface is measured for obtaining the temperature decay from which thermal diffusivity of the liquid sample can be estimated. The validity of this new laser flash technique has been confirmed by obtaining the thermal diffusivity values of silicate melts in the temperature range between 773 and 1673 K.

8:55 AM
Two Methods for the Continuous Measurement of Slag Viscosity from the Glassy to the Liquid States: *Yutaka Shiraishi*[1]; Yutaka Sakurai[1]; [1]AGNE Gijutsu Center, Ltd., Minami-Aoyama 5-1-25, Kitamura Bldg., Minato-ku, Tokyo 107-0062 Japan

Two methods were developed to measure the viscosity of slag from the glassy to the liquid states in an ascending temperature process. The first method, referred to as the sp method, employs rotating parallel plates within which is placed a solid cylindrical sample in line with a sphere. The viscosity of the glass sample is first determined from the rate of penetration of the sphere into the sample. After the sphere is fully immersed within the sample, the viscosity is determined from the creep rate of the sample between the parallel plates. In the liquid state, the viscosity is determined from the torque generated by the rotation of the plates. The second method, referred as the tp method, uses a tube which is pushed into a sample held in a rotating crucible. In this case, the viscosity is determined from the rate of penetration of the tube into the glass sample and from the torque exerted on the tube by the rotation of the liquid sample. Both methods were tested using standard glasses, including boron trioxide and NBS standard glass #711. The main difference between these two methods is in sample preparation. Comparative measurements between the sp and the tp methods were performed using a sodium boro-di-silicate glass containing 5%mol of alumina. The results showed good agreement in the mid-range of glassy viscosities, 105 to 107 Pa.s. In the liquid state, there were no significant differences between the two methods. A discussion is presented about the observed discrepancies in the results.

9:20 AM
Advanced Image Analysis of Molten Slag for Process Control: *Subagyo*[1]; G. A. Brooks[1]; [1]McMaster University, Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

The condition of slag in a metallurgical reactor is vital to ensuring control of the process. A new technique based on multivariate image analysis (MIA) of digital images processing has been developed to provide an online measurement of important slag characteristics, such as the presence of exposed metal, the presence of solid phases, and temperature. This paper shall describe the technique and provide some results from plant trials.

9:45 AM
Impurity Removal from Carbon Saturated Liquid Iron Using Lead Solvent: *Katsunori Yamaguchi*[1]; Yoichi Takeda[1]; [1]Iwate University, Fac. of Eng., Ueda 4-3-5, Morioka, Iwate 020-8551 Japan

A new technique for the removal of tramp-elements from steel scrap has been required from the viewpoint of saving energy and protection of environment. In order to obtain fundamental information on the removal of copper, tin and zinc from molten iron, the extraction method by using of phase separation in the liquid Fe-C-Pb system was investigated at 1453K. A mixture of iron, lead and carbon melted in a carbon crucible separates into two phases of lead and iron containing carbon. The compositions on the miscibility gap in the Fe-C-Pb system are 95.4%Fe-4.5%C-0.1%Pb and 99.9%Pb-0.1%Fe. The distribution ratios of copper, tin and zinc between the lead and the iron phases, LX=[mass%X]Pb/(mass%X)Fe, are 2.2, 2.1 and 1.4, respectively. Seventy percent of copper and tin from iron scrap can be eliminated with comparable lead addition.

10:10 AM Break

10:30 AM
The Criteria of Hydrogen and Oxygen Potentials in Copper Melt for Making a Sound Casting of Bar and RodñThe Guide of the Effective Use of an Electrochemical Gas Monitoring System: *Norihiko Fukatsu*[1]; Noriaki Kurita[1]; Daisuke Yamamoto[2]; [1]Nagoya Institute of Technology, Dept. Matls. Sci. & Eng., Gokiso-cho, Showa-ku, Nagoya 466-8555 Japan; [2]Mizuno Corporation, Miyahara-cho, Omiya 330-0038 Japan

The in-line monitoring of the hydrogen and oxygen potentials in molten copper has been realized by using the electrochemical sensors based on the proton and the oxide ion conducting solid electrolyte. In order to use this sensing system effectively, the criteria of the potentials for making sound casting must be known. In the present work, the copper melt equilibrated with a given hydrogen and oxygen potentials was quenched in the crucible. The relations between the holding potentials and the gas porosity in the solidified sample were investigated by measuring the density of the sample and microscopic observations. The criteria thus determined were examined with reference to the measured potentials in the practical casting machine. In tough-pitch copper casting, the problem due to gas porosity was found to be serious when the hydrogen potential increased. In the oxygen-free copper casting, the evolution of hydrogen was found not to be avoided. The gas porosity in the casting, however, was found to be eliminated when the oxygen potential was kept at a low value.

10:55 AM
Viscosities of FeO-MgO-SiO2 and FeO-MgO-CaO-SiO2 Slags: Xi Dai[1]; Xueping Gan[1]; Chuanfu Zhang[1]; [1]Central South University, Sch. of Metallurgl. Sci. & Eng., Changsha, Hunan 410083 China

In the present work,the viscosities of molten FeO-MgO-SiO2 and feO-MgO-CaO-SiO2 semi-synthetic slags at nickel flash smelting conditions were measured in the temperature range from 1523 to 1723 K using a rotational viscometer. The Fe/SiO2 ratio was maintained constant at 1.2 and the calcium and the magnesium oxide contents were varied in the range from 2 to 8 wt-% and 9 to 12 wt-%, respectively. The viscosity values are presented as a function of temperature and composition.

11:20 AM
Measurement of the Oxygen Potential of Non-Ferrous Slags with an Ex-Situ Electrochemical Device: *Nele Moelans*[1]; Bert Coletti[1]; Bart Blanpain[1]; Patrick Wollants[1]; Jaak Plessers[2]; Marc

Straetemans[2]; [1]Katholieke Universiteit Leuven, Dept. of Metall. & Matls. Eng., Kasteelpark Arenberg 44, Leuven B-3001 Belgium; [2]Heraeus Electro-Nite Int. N.V., Centrum Zuid 1105, Houthalen B-3530 Belgium

A measurement method using an ex-situ electrochemical measurement device was developed to determine the oxygen potential of non-ferrous metallurgical slags. The electrochemical cell consists of the molten slag sample, an inert Ir electrode, a stabilized ZrO2 solid electrolyte, and a Ni/NiO reference electrode. Different slags obtained from non-ferrous pyrometallurgical processes were analysed at 1100 and 1200∞C and $p(O_{/2})$ values between 2 10^{-3} and 1 10^{-12} were obtained. The measurement values were evaluated with thermodynamic calculations and using processing considerations. It was found that the reproducibility and stability of the measurement was significantly improved with the addition of carbon to the slag, especially for slags with a low oxygen potential.

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

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

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

Thursday AM — Room: Santa Rosa
March 6, 2003 — Location: San Diego Marriott Hotel

Session Chairs: Susumu Okabe, Mitsubishi Materials Corporation, Tokyo 100-8117 Japan; Florian Kongoli, FLOGEN Technologies Inc., Matls. Tech. Dept, Montreal, Quebec H3S 2C3 Canada

8:30 AM

Application of Porous Plug System in Anode Furnace, Onsan Smelter: *Sang-Su Lee*[1]; Baek-Sang Kim[1]; Sei-Phim Choi[1]; [1]LG-Nikko Copper, Inc., Daejung-Ri 70, Ulju-Gun, Onsan-Eup, Ulsan City 689-892 Korea

This paper shows the application of porous plug system in the Mitsubishi continuous copper smelting and converting process. This system has already adopted and utilized in the steel industry to enhance productivity. Recently, some copper smelter has adopted this system but the result has not been satisfactory. Porous plug system was installed at the Anode furnace in December 2001 to reduce oxidation time and decrease the oil consumption. Some good effect is that nitrogen stirring the melt through porous plugs, improved the heat transfer of the melt. As the result of enhancing heat transfer, oil consumption and inside build-up was actually decreased. But the oxidation time has not sufficiently been reduced until now. In the future, further research and local test is needed for productivity increase and cost saving.

8:55 AM

Behavior of Silver in the Electric Furnace for Copper Dross Treatment: *Takao Yasugi*[1]; [1]Toho Zinc Company, Ltd., Production Dept., 5562-1 Higashino-cho, Toyota-gun, Hiroshima-Pref. 725-0222 Japan

The majority of copper in the raw material migrates to the decopperized dross formed when crude lead produced from the blast furnace is treated by the decopperizing process. Decopperized dross is treated in the electric furnace, producing crude lead, matte, and speiss. Decopperized dross contains large amounts of silver. Therefore, it is important to improve the rate of silver migration to crude lead. For this reason, we examined the behavior of silver between the crude lead, speiss, and matte phases. At the same time, we conducted several tests to improve the percentage of silver migrating to crude lead.

9:20 AM Invited

A Process Designed for the Ancient Copper Smelting Slags: *Bora Derin*[1]; *Onuralp Y¸cel*[1]; Ercan Acma[1]; Okan Addemir[1]; [1]Istanbul Technical University, Metallurgl. & Matls. Dept., Fac. of Cheml. & Metallurgl. Eng., Maslak, Istanbul 80626 Turkey

This work carried out on the ancient copper smelting slags located in K¸re-Turkey for recovery of valuable metals. In this process, copper and cobalt were recovered as metallic and their components, where as iron was recovered as magnetic oxide or pigment from the slag. The process stages were involved carbothermal reduction in DC-Arc Furnace, leaching, chemical precipitation, selective roasting and product preparation. In this paper, the process stages were described as theoretically and experimentally where as slag and products were characterized in detail.

9:45 AM

Present and Future of Caletones Smelter: *Patricio Burchard Chacana*[1]; *Gerardo Hern·n Achurra*[1]; Julio Gatica Buchi[1]; Fernando Andres Condore[1]; [1]CODELCO, El Teniente, Caletones Smelter, Millan 1020, Rancagua, OíHiggins VI 000 Chile

During the last years, Caletones Smelter like others world copper smelter, has evolved from conventional, inefficient, pollutant and expensive technologies, to modern, environmentally friendly and efficient ones. For it and besides for improving its competitive position, in the last period this Smelter shut down their reverberatory furnace, started up two big acid plants and has developed some pirometallurgical technologies. The main facilities of Caletones Smelting and Converting Process, in the present and future, are the Teniente Converters, reactors with a high operational availability that have reached concentrate top process capacities near to 2,400 T/D. These reactors operate in most of the Chilean smelters as well as in other world smelters: Zambia, Peru and Mexico. Since 2000, the Caletones Smelting and Converting Process, has been performed in two Teniente Converters, four Slag Cleaning Furnaces and four Pierce-Smith Converters. These facilities, together with the operation of two Fluidized Bed Drye rs and two Oxygen Plants have allowed to reach concentrate smelting capacities and copper productions of 1,250 KDT/year and 380 KFT/year, respectively. Associated to El Teniente Division Expansion Plan, in a first stage, Caletones Smelter will increase its capacity to 1,440 KT/year the 2004. In the long term, Caletones has different options, from 1,600 to 2,000 KT/year, producing anodes, white metal or a mixture of them. This options are connected with CODELCOís Smelters & Refineries Strategic Plan, including Mejillones Project. The main challenges of Caletones Smelter are associated to develop and consolidate Continuos and Environmental Friendly Processes, automation and control of different processes and improve its leadership between World Copper Smelters.

10:10 AM Break

10:25 AM Invited

Thermodynamic Fundamentals of Calcium Ferrite Slag and their Application to Mitsubishi Continuous Copper Converter: *Fumito Tanaka*[1]; Osamu Iida[2]; Yoichi Takeda[3]; [1]Mitsubishi Materials Corporation, Central Rsrch. Inst., 1-297 Kitabukuro-cho, Saitama 330-8508 Japan; [2]Mitsubishi Materials Corporation Metals Company, Process & Tech. Dept., 1-5-1 Ohtemachi, Chiyoda-ku, Tokyo 100-8117 Japan; [3]Iwate University, Dept. of Matls. Sci. & Tech., 4-3-5 Ueda, Morioka, Iwate 020-8551 Japan

The Mitsubishi process is the sole pyrometallurgical process for continuous production of blister copper from copper concentrates and has been successfully operated around the world. The development of calcium ferrite slag (CaO-FeOX-Cu2O), so called ìC-slagî, has enabled continuous converting from matte to blister copper in commercial scale operation. Commercial interests in the slag led to many papers over CaO-FeOX slag. However, Cu2O-bearing slag has not been studied well due to experimental difficulties. The authors have quantified thermodynamic properties of CaO-FeOX-Cu2O system. The results have been utilized to analyze various issues related to the converting furnace, such as magnetite precipitation from slag or from blister copper, and to develop better control criteria for the continuous converting operation.

10:50 AM

Process Development, Optimization and Automation through Appropriate Thermophysicochemical Modeling and Simulation Software: *F. Kongoli*[1]; I. McBow[1]; S. Llubani[1]; [1]FLOGEN Technologies, Inc., 5757 Decelles Ave., Ste. 511, Montreal, Quebec H3S 2C3 Canada

Nonferrous and ferrous smelting industries have recently faced the unavoidable necessity of changing and/or improving the smelting technologies as a result of the use of new raw materials which are becoming available from different geographical areas. These new feed materials usually contain different ore composition and higher level of minor components, which adversely affect the smelting process. Due to the problems encountered in several processes as a result of this feed diversification some work has been undertaken in order to make uniform the feed and avoid later surprises in the smelting process. However this has proven to be very difficult and sometimes almost impossible. In this work a more viable approach is undertaken to deal with this problem. It consists of controlling not the cause of the problem i.e. the feed composition but instead the results of this feed change, i.e. the end-point of smelting technologies. It is shown that this approach, when carried out through appropriate thermophysico-chemical modeling and simulation software is not only easier and less costly but it also helps the automation of the smelting process. Several examples are given and future work is underlined.

11:15 AM
Control of Magnetite Behavior in the Mitsubishi Process at Naoshima: *Nozomu Hasegawa*[1]; Hideya Sato[1]; [1]Mitsubishi Materials Company, Ltd., Naoshima Smelter & Refinery, 4049-1 Naoshima-Cho, Kagawa-Gun, Kagawa 761-3110 Japan

The phase diagrams and microstructures of slags in the Mitsubishi Continuous Copper Smelting and Converting Process were studied for the prevention of magnetite troubles such as accretion build-up on furnace hearths and launders, melt outlet blockages, slag viscosity increases and so on. According to the result of those studies, the silica and lime content in the silicate slag at the smelting furnace and the lime content in the calcium-ferrite slag at the converting furnace were changed, and then the slag loss of copper in the discard slag has been stabilized lower than before and the cleaning- frequency of melt outlets and launders has been significantly reduced. However, those procedures may decrease the desirable amount of accretion-coating inside the furnaces and thereby reduce the furnace campaign lives. Therefore, an estimating method of those accretion amounts was developed, and slag compositions and temperatures have been controlled within the appropriate ranges to prolong furnace lives and also prevent magnetite troubles.

11:40 AM
The Behavior of Impurities at Kosaka Smelter: *Satoshi Nakagawara*[1]; Kenji Watanabe[2]; [1]Kosaka Smelting & Refining Company, Ltd., Production Mgmt. Sec., 60-1 Otarube, Kosaka, Kazuno, Akita 017-0202 Japan; [2]Kosaka Smelting & Refining Company, Ltd., Techl. Dept., Kosaka, Kazuno, Akita 017-0202 Japan

Kosaka Smelter has been changed to a custom smelter and devoting to treat the complex sulfide copper concentrates in the world. Since the mid-80s, a progressive diversification of the smelter input has taking place, impure and complex sulfide concentrate being introduced in increasing proportions compared with the period of the ìBlack Oreî treatment. And recently, Kosaka is trying to add value to its copper smelting by recovering valuable metals from non-concentrate materials (e.g.?\@recycled materials and the residue generated in the zinc smelter), and by increasing the volume of recovery. In this process, precious metals are mostly recovered and unnecessary impurities are efficiently removed. Thus, it can be said that recent operation of Kosaka Smelter depends on the treatment of impurities. This is a report on the behavior of impurities at Kosaka Smelter which performs outstanding smelting operation.

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

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Thursday AM	Room: Leucadia
March 6, 2003	Location: San Diego Marriott Hotel

Session Chairs: H. G. Kim, POSCO Research Institute Korea; Varadarajan Seshadri, Universidade Federal de Minas Gerais, Dept. of Metallurgl. Eng. & Matls., Belo Horizonte Brazil

8:30 AM Invited
Operational Improvements for RH-KTB Degasser of Companhia Siderurgica De Tubarao (CST) Steel Plant Through a Physical Modeling Study: *Varadarajan Seshadri*[1]; Itavahn Alves da Silva[2]; Carlos Antonio da Silva[2]; Roberto Parreiras Tavares[1]; Ernane M·rcio de Castro Martins[3]; Fernando Demuner da Silva[1]; Geraldo Alves Vargas Filho[4]; Paulo SÈrgio Bringhenti Lascosqui[4]; [1]Universidade Federal de Minas Gerais, Dept. of Metallurgl. Eng. & Matls., Belo Horizonte Brazil; [2]Universidade Federal de Ouro Preto, Dept. of Metallurgl. & Matls. Eng., Ouro Preto Brazil; [3]Universidade Federal de Ouro Preto, REDEMAT, Ouro Preto Brazil; [4]Companhia Sider˙rgica de Tubar„o, Vitoria Brazil

Improvements in the efficiency of the metallurgical performance of refining reactors in the metallurgical industry can be achieved through physical modeling of the unit and experimentation in the laboratory scale. In the Rh process, macroscopic parameters such as circulation time and mass transfer coefficient for the degassing step are important parameters which decide improved operational practice. In this study the effect of some operational parameters, such as diameter of snorkels, gas injection flow rate through bottom of the ladle, gas flow rate through nozzles on the circulation rate and decarburization of steel are investigated using a physical model. The object of the investigation was to improve the performance characteristics of the RH-KTB degasser of Companhia Sider˙rgica de Tubar„o (CST) steel plant.

9:00 AM
Optimization of the Injection Refining-Up Temperature Process (IR-UT) of the Companhia Siderurgica de Tubarao Steel Plant (CST) Using Physical Modeling: *Carlos Antonio da Silva*[1]; Itavahn Alves da Silva[1]; Varadarajan Seshadri[2]; Cristiano Magson de Oliveira Genelhu Silva[3]; Marcos de Paula Alves[1]; Carlos Alberto Perim[4]; [1]Universidade Federal de Ouro Preto, Dept. of Metallurgl. & Matls. Eng., Ouro Preto Brazil; [2]Universidade Federal de Minas Gerais, Dept. of Metallurgl. Eng. & Matls., Belo Horizonte Brazil; [3]Universidade Federal de Ouro Preto, REDEMAT, Ouro Preto Brazil; [4]Companhia Sider˙rgica de Tubar„o (CST), Vitoria Brazil

Steel refining reactors based on chemical heating are being used as an alternative to ladle furnaces. They show a high degree of flexibility in respect of heating rate in addition to the ease in carrying out refining reactions such as desulphurization, inclusion removal, alloying etc. IR-UT and CAS-OB are examples of such reactors. Their performance is dependent upon geometrical characteristics as well as operational conditions which determine the flow field inside the vessel. A physical model of the IR-UT process of the Companhia Sider˙rgica de Tubar‚o steel plant situated in Vitoria, Brazil was developed to assess the features of the flow field and its influence on the metallurgical behavior. Results of the physical model were used to optimize the process parameters.

9:25 AM
Effect of TiC Addition on Corrosion Resistance of MgO-C Based Refractories to Smelting Reduction Slag: *Qingcai Liu*[1]; Jing Lin[1]; Dengfu Chen[1]; Joseph W. Newkirk[2]; [1]Chongqing University, 174 Shapinba St., Chongqing 400044 China; [2]University of Missouri-Rolla, 1870 Miner Cir., Rolla, MO 65409 USA

The interaction between MgO-C-TiC refractories and slag of smelting reduction with and without iron bath was studied by rotary immersion and stationary immersion test. The effects of the TiC addition to the corrosion behavior of the MgO-C based refractories were investigated in detail. The present work highlights significant improvements in the corrosion resistance of the refractories to slag from addition TiC in the MgO-C based refractories. Petrographic and SEM analysis of the refractories after the slag test show that TiC increases the viscosity of both the glassy phase of the refractory and the slag film. This effect retards the slag penetration into refractory and the interaction between the slag film and the deterioration layer of refractory.

9:50 AM
Assessment of the Performance of the Tundish of a Six Strand Continuous Casting Unit of Companhian Siderurgica de Belco-Mineira(CSBM) Using Physical Modeling: *MÙnica Suede Santos Silva*[1]; Elvis GonÁalves da Mota[2]; Edilson CaniÁali Fracalossi[2]; Carlos AntÙnio da Silva[2]; Itavahn Alves da Silva[2]; Joaquim GonÁalves da Costa Neto[3]; Varadarajan Seshadri[4]; [1]Universidade Federal de Ouro Preto, REDEMAT, Ouro Preto Brazil; [2]Universidade Federal de Ouro Preto, Dept. of Metallurgl. & Matls. Eng., Ouro Preto Brazil; [3]Companha Siderurgica de Belgo Mineira, Jo„o Monevalde Brazil; [4]Universidade Federal de Minas Gerais, Dept. of Metallurgl. Eng. & Matls., Belo Horizonte Brazil

This work describes characterization of the performance of the tundish of a six strand continuous casting unit of the Companhia siderurgica Belgo Mineira, Jo„o Monlevade, Brazil using physical modeling. Permanent and transient conditions, due to ladle metal composition changes are considered. Pulse and step tracer addition techniques are employed to assess the control variables. For transient conditions the degree of inter-mixing and the minimum residence time are determined with a view to optimizing the process especially during the grade changes of steel.

10:15 AM Break

10:25 AM
Interfacial Area in Pyrometallurgical Reactor Design: *Geoffrey Alan Brooks*[1]; Subagyo[1]; [1]McMaster University, Dept. of Matls. Sci. & Eng., 1280 Main St. W., Hamilton, Ontario L8S4L7 Canada

Increasing interfacial area through gas injection is one of the main methods for accelerating reaction between slag, metals and gases in modern metallurgical reactors. This paper will examine the limitations of top lance and bottom tuyere blowing in terms of interfacial area generation and examine alternative reactor designs that may overcome these limitations. The effect of the quantity and size of droplets generated through gas injection on overall reactor design performance, especially relating to settling rates, will be examined in this paper.

10:50 AM
Software for Selection and Analysis of Mould Fluxes for Continuous Casting of Slabs: *Fabricio Batista Vieira*[1]; Varadarajan Seshadri[2]; Roberto Parreiras Tavares[2]; [1]Vallourec & Mannesmann Tubes, Belo Horizonte, MG 30640-010 Brazil; [2]Federal University of Minas Gerais, Metallurgl. & Matls. Eng. Dept., Belo Horizonte, MG Brazil

Mould fluxes are synthetic slags normally used in continuous casting of steels, particularly in slab casting. They should satisfy several requirements, which include: - reducing the friction between the strand and the mould walls; - controlling the heat transfer between the solidified shell and the mould. The performance of mould fluxes in satisfying the requirements above can have a significant effect on the efficiency of the continuous casting process and on the surface quality of the products. This performance is affected by various parameters including the characteristics of the mould fluxes, the quality of the steel being cast and the operational conditions of the caster. The extremely complex relationships involving the parameters mentioned above make it very difficult to have simple criteria for selecting mould fluxes for casting a certain grade of steel in a continuous casting machine. These difficulties led steelmakers and mould flux suppliers to select mould fluxes based mainly on plant trials. In t he present work, a software to help in the analysis and development of mould fluxes for continuous casting was developed. This software includes several models for prediction of mould flux viscosity and consumption. A mathematical model was also used to predict the heat flux through the mould flux. The proposed methodology was applied to continuous casting of crack sensitive steels. Some of the results given by this software are presented, discussed and compared to plant data.

11:15 AM Invited
Modeling of EAF Slag Chemistry for Optimal Slag Foaming and Refractory Service Life: *James P. Bennett*[1]; Kyei-Sing Kwong[1]; [1]USDOE, Albany Rsrch. Ctr., 1450 Queen Ave. SW, Albany, OR 97321 USA

EAF slag chemistry and its control have been recognized as important in producing quality steel at a low price for a number of decades. EAF steel producers make lime and dolomite additions to furnaces, controlling the C/S ratios and sulfur pickup from the steel. Existing phase diagram data at 1600°C for CaO, MgO, FeO, SiO2, and Al2O3 was used to write a computer model optimizing slag chemistry for slag foaming and reduced refractory wear. The model, its development, and its use in an industrial steelmaking environment will be discussed along with how the model can improve energy efficiency. The importance of EAF mass balance to account for critical materials like Si in scrap and refractory wear using the model will also be emphasized.