TMS

Become A TMS Member

TMS derives its strength from its members, who take a hands-on approach to shaping the policy, programming, and publications of the society. Guided by these volunteers, TMS serves all segments of its professional community by:

FACILITATING NETWORKING:

By sponsoring numerous annual meetings and specialty conferences, TMS maximizes the opportunities for professionals from industries, universities, and government agencies worldwide to meet face to face and exchange technical ideas and experience, offer customer/client insights, find a mentor and/or serve as one, and just plain chat with peers and colleagues.

PRODUCING JOM AND OTHER PUBLICATIONS:

Every TMS member receives a complimentary subscription to JOM. Formerly Journal of Metals, this highly respected monthly journal, explores traditional, innovative, and revolutionary issues in the minerals, metals, and materials fields. Designed to be of maximum and immediate benefit to readers throughout the world, JOM is on-line before the print version is mailed.

TMS also publishes three other journals (*Journal of Electronic Materials* and *Metallurgical and Materials Transactions A and B*), numerous conference proceedings volumes and textbooks, and videos designed to give materials scientists and engineers the latest information on scientific and applied advances in areas as diverse as electronic materials, automotive manufacture, and extractive metallurgy.

PROMOTING LIFE-LONG LEARNING:

TMS is dedicated to the education of the materials science and engineering professional as well as to cultivating an interest in the field by young people.

For the practicing professional, TMS and its five technical divisions sponsor continuing education courses, primarily technical but also nontechnical, to promote the education and development of current and future professionals.

For student members, TMS participates on both the Accreditation Board for Engineering & Technology (ABET) and the National Council of Examiners for Engineering and Surveying (NCEES) to help, respectively, maintain the highest possible standards in the accreditation of metals and materials programs in academia and in the registration of professional engineers.

—All individuals registering for the 130th Annual Meeting & Exhibition at the non-member fee will automatically receive a one-year complimentary introductory membership for 2001. Your membership will be activated upon completion of your registration form, membership application, and payment of the non-member registration fee. You will receive a membership card and new member packet immediately after the meeting.

- *Members from 77 countries and six of the world's seven continents.*
- *All new members will begin receiving a monthly subscription to JOM.*
- *New members will also be able to continue networking with a prestigious membership at future TMS meetings that fit their area of interest at a discounted member fee.*
- *Additional benefits include access to, and inclusion in the TMS Membership Directory on TMS OnLine at www.tms.org., professional development and continuing education opportunities, and group insurance programs. See the membership page on TMS OnLine for a complete list of membership benefits.*
- *Please direct any questions regarding your complimentary membership to the TMS Member Services Department via email to abartholomay@tms.org or via phone to Anne Bartholomay at (724) 776-9000 Ext. 241.*

TMS THE MINERALS, METALS & MATERIALS SOCIETY

PROMOTING THE GLOBAL SCIENCE AND ENGINEERING PROFESSIONS CONCERNED WITH MINERALS, METALS, AND MATERIALS

184 THORN HILL ROAD
WARRENDALE, PA 15086-7514
USA

TELEPHONE: (724) **776-9000**
(800) **966-4867**
FAX: (724) **776-3770**
WEB: **www.tms.org**

THE VISION OF TMS IS TO BE THE PROFESSIONAL SOCIETY OF CHOICE FOR THE WORLDWIDE MINERALS, METALS AND MATERIALS COMMUNITY.

GREAT MEMBER BENEFITS

- Five distinct technical divisions which are composed of 52 separate, highly specialized committees
- Periodicals: JOM, Metallurgical and Materials Transactions A and B, Journal of Electronic Materials
- Conference Proceedings, Monographs, and Textbooks
- TMS OnLine & the TMS Document Ordering Center
- TMS Conferences: TMS Annual Meeting & Exhibition, TMS Fall Meeting, TMS Fall Meeting for Extraction & Processing, Electronic Materials Conference, Specialty Conferences
- Professional Development and Continuing Education Opportunities
- Professional Registration
- TMS Young Leaders
- TMS Resume Referral Service
- TMS Gold or Platinum MasterCard
- Group Insurance
- TMS Membership Directory
- TMS Speakers Directory
- International Healthcare Plan
- Hertz Car Rental Discounts
- Auto and Homeowners Program
- PROinsure Program
  A Professional Liability/Errors and Omissions Program
- PRObop Program
  A Professional Business Owners Package Program
- Member Benefits Program
  Receive a 20% Rebate Buying or Selling Your Home
- Nelson Financial Services Program
- WAAIME Auxiliary Activities

ADMISSION REQUIREMENTS

FULL MEMBER
A candidate for election as full member shall be a person of integrity in activities associated with minerals extraction, processing, fabrication, or with materials applications. A candidate shall hold: (a) A baccalaureate degree in metallurgy, metallurgical engineering, materials science, or materials engineering, and at least 3 years' professional experience. (b) A baccalaureate degree in science or engineering in a discipline other than identified and at least 5 years' professional experience. (c) A baccalaureate degree from a recognized university in a discipline other than (a) or (b) and whose main activities lie in, but are not limited to, the development, management, administration, welfare, sales, or services to the minerals, metals and materials industries, with at least 7 years' experience. A credit in experience of one year for a masters degree or two years for a doctoral degree shall be granted.
Annual dues: $90.00

ASSOCIATE MEMBER
A candidate for associate member shall be a person of integrity who, while not possessing the academic or technical experience of a member, is active in fields that are sufficiently related to the advancement of, or service to, the minerals, metals or materials extraction, processing, or applications industry.
Annual dues: $90.00

The Minerals, Metals & Materials Society is a member society of the American Institute of Mining, Metallurgical and Petroleum Engineers, Inc.

LIFE MEMBER
A candidate for election as life member shall be a person who qualifies as a full member or associate member and desires to only pay dues once.
Dues: $1,350.00
Pay dues once, effective for lifetime regardless of dues increase(s).

REINSTATEMENT
Those members who may have let their dues payment lapse may reinstate in the same grade as when they left by submitting a new application and paying a reinstatement fee of $10.00 plus current dues. If original election year is desired, back dues must be paid to date (half the annual dues fee for each year of lapsed membership); otherwise, election year will be year of reinstatement.

The TMS membership year runs from January 1–December 31. Applications received January 1–September 30 will be processed for the current calendar year.

Applications received after September 30 will be processed for the remainder of the current calendar year and the entire following year. Membership benefits commence upon processing; subscriptions commence January–December of the following year.

Two weeks required for processing of complete applications submitted with full payment. Incomplete applications will not be processed. Allow eight to ten weeks for subscriptions to start.

MEMBERSHIP APPLICATION

PLEASE TYPE OR PRINT

❒ Mr. ❒ Mrs. ❒ Ms.
❒ Dr. ❒ Professor

SEND MAIL TO:
- ❒ Business Address
- ❒ Home Address

TECHNICAL DIVISION SELECTION:
- ❒ Electronic, Magnetic, & Photonic Materials Division
- ❒ Extraction & Processing Division
- ❒ Light Metals Division
- ❒ Materials Processing & Manufacturing Division
- ❒ Structural Materials Divison

THROUGH WHAT MEANS WERE YOU ENCOURAGED TO JOIN TMS?
- ❒ TMS Annual Conference
- ❒ TMS Fall Conference
- ❒ TMS Specialty Conference
- ❒ Exhibitor
- ❒ TMS Staffed Booth
- ❒ JOM
- ❒ MET TRANS, JEM, Etc.
- ❒ Publications Catalog
- ❒ TMS Mailed Brochure
- ❒ Non-TMS Advertisement
- ❒ Continuing Education
- ❒ TMS OnLine/Web
- ❒ Colleague
- ❒ Other _____

BIRTHDATE:

_____/_____/_____

Members automatically receive a monthly print subscription to JOM.

If you prefer to receive your copy only electronically, please check here. ❒

If you prefer to receive your subscription in both print and electronic formats, please check here. ❒
(You must include $8 extra charge.)

If you would like additional information about member subscriptions and discounts to JOM, Journal of Electronic Materials, or Metallurgical and Materials Transactions A and B, please check here. ❒

FOR OFFICE USE ONLY
ID
BIRTH
ELECTED
TYPE
CATEGORY
SECTION
APPROVED

NAME: _____ (LAST / FIRST / MIDDLE INITIAL)

TITLE: _____

COMPANY OR ORGANIZATION: _____

BUSINESS: _____ (STREET OR P. O. BOX / CITY / STATE / 9 DIGIT ZIP/POSTAL CODE / COUNTRY)

_____ (PHONE / FAX / TOLL FREE # / E-MAIL / WEB ADDRESS)

HOME: _____ (STREET OR P. O. BOX / CITY / STATE / 9 DIGIT ZIP/POSTAL CODE / COUNTRY)

_____ (PHONE / FAX / E-MAIL)

_____ (MONTH / DAY / YEAR)

WHAT IS THE PRIMARY ACTIVITY OF YOUR PLACE OF EMPLOYMENT? (check one)

❒ Commercial Laboratory
❒ Government/Nonprofit Laboratory
❒ Engineered Materials Producer
❒ Manufacturer of Parts/Components
❒ Manufacturer of Finished Products (OEMs)
❒ Primary Metals Producer
❒ Secondary Metals Producer
❒ Producer/Processor of Materials
❒ Educational
❒ Engineering or Consulting Firm
❒ Other _____

WHAT BEST DESCRIBES YOUR PRIMARY JOB FUNCTION? (check one)

❒ Applications/Product Development
❒ Basic Research
❒ Product Engineering and Design
❒ Technical/Lab Management
❒ Process Engineering
❒ Metallurgical Materials Selection
❒ Corporate Management
❒ R & D Engineer
❒ R & D Scientist
❒ R & D Management
❒ Manuf./Production Management
❒ Quality Engineering
❒ Marketing or Sales
❒ Consultant
❒ Educator
❒ Student
❒ Other _____

OTHER SOCIETY AFFILIATIONS: _____

EDUCATION TO DATE:

Name of School	Dates Attended Month/Year–Month/Year	Major Subject/ Engineering Field	Degree Received or Expected Graduation Date: Month/Year
B.S. ❒			
M.S. ❒			
Ph.D. ❒			

REGISTERED PROFESSIONAL ENGINEER? ❒ Yes ❒ No STATE: _____ YEAR OF REGISTRATION: _____

RECORD OF EXPERIENCE:
(List most recent record of employment. If you do not possess a qualifying degree, please include your last seven years of experience.)

From: _____ Title: _____
Company: _____
To: _____ Nature of Company's Business: _____
Total Time with Company: _____
Engineering Responsibilities: _____

TO APPLICANT

If you have been encouraged to submit this application by a current member of TMS, please complete the following information:

Member's Name _____ Member # _____

I agree, if elected, to accept election, and to abide by the TMS bylaws.

Signature _____ Date _____

PREPAYMENT IS REQUIRED (checks should be made payable to TMS in U.S. dollars drawn on a U.S. bank)

❒ Check enclosed
❒ Bill my credit card: (check one)
❒ American Express ❒ VISA ❒ MasterCard ❒ Diners Club

Credit Card # _____ Expiration Date _____

Cardholders Name _____

Signature _____

COMPLETE APPLICATION AND MAIL WITH PAYMENT TO: **184 Thorn Hill Road, Warrendale, PA 15086-7514**
Phone: 800-966-4867 or 724-776-9000 • Fax: 724-776-3770

Advance Registration Form

FOR THE TMS ANNUAL MEETING AND EXHIBITION ■ FEBRUARY 11–15, 2001 ■ NEW ORLEANS, LOUISIANA

PLEASE CHOOSE ONLY ONE OPTION FOR SENDING FORM

WEB Take advantage of the convenience of on-line pre-registration via the TMS website: **http://www.tms.org** Web registration requires credit card payment.

FAX Fax this form to TMS Meeting Services **USA (724) 776-3770** Fax registration requires credit card payment.

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

TMS2001 130TH ANNUAL INTERNATIONAL MEETING & EXHIBITION

Advance Registration Deadline: January 22, 2001

PAYMENT MUST ACCOMPANY FORM.
Forms received past this date will be processed at the on-site fee.
Instructions: Check your selections and fill in the necessary information. Please print or type.

MEMBER OF: ☐ TMS ☐ ISS ☐ SME ☐ SPE Member Number: _____

THIS ADDRESS IS: ☐ Business ☐ Home Employer/Affiliation: _____

☐ Dr. ☐ Prof. ☐ Mr.

☐ Mrs. ☐ Ms. _____
LAST NAME FIRST NAME MIDDLE INITIAL

Address: _____

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

Telephone: _____ Fax: _____
COUNTRY AREA/CITY LOCAL NUMBER COUNTRY AREA/CITY LOCAL NUMBER

E-Mail Address: _____ Guest/Spouse Name: _____
GUESTS DO NOT RECEIVE ADMISSION TO TECHNICAL SESSIONS.

REGISTRATION FEES:

	ADVANCE FEES (until 1/22/01)	ON-SITE FEES (after 1/22/01)
☐ Member	$390 M	$490 ML
☐ Non-Member Author	$390 NMA	$490 NMAL
☐ Non-Member *	$520 NM	$600 NML
☐ Student Member ##	$0 STU	$0 STUL
☐ Student Non-Member ## *	$25 STUN	$25 STUNL
☐ TMS Retired Member	$200 RM	$200 RML
☐ Exhibit Booth Personnel	$0 E	$0 EL
☐ Exhibit Attendee	$35 EO	$35 EOL

* Includes TMS membership for 2001
Students must attach a copy of their school's student identification card.

PUBLICATION ORDERS:

ALL pre-ordered books not indicated for shipment MUST be picked up at the Publications Sales area in the convention center.

Please ship to the above address: No. of books _____
$15 per book $_____(SB)

☐ 4801 Light Metals 2001 (CD-ROM & Book Set) $164
☐ 478X Chemistry and Electrochemistry of Corrosion and Stress Corrosion Cracking $96
☐ 4798 Cyanide: Social, Industrial, and Economic Aspects $86
☐ 4895 Elevated Temperature Coatings CD-ROM $60
☐ 4887 EPD Congress 2001 ..... $125
☐ 4909 Innovations in Processing and Manufacturing of Sheet Materials $97
☐ 481X Magnesium Technology 2001 ..... $124
☐ 4879 Structural Biomaterials for the 21st Century $65

TUTORIAL LUNCHEON LECTURE TICKETS:

OPTIONAL BOX LUNCHES	FEE	NO.	TOTAL
Monday 2/12/01 (SPONSORED BY YOUNG LEADERS)			
☐ Young Leaders Extractive Metallurgy	$15	_____	$_____EM

SOCIAL FUNCTION TICKETS:

	FEE	NO.	TOTAL
Monday 2/12/01			
☐ Larry Kaufman Honorary Dinner	$55	_____	$_____KD
Tuesday 2/13/01			
☐ TMS Banquet	$60	_____	$_____AD
☐ Tables of 8	$480	_____	$_____AD8
Table Sign to Read: _____			
☐ Extraction & Processing Division Luncheon	$25	_____	$_____EP
☐ Tables of 8	$200	_____	$_____EP8
Table Sign to Read: _____			
Wednesday 2/14/01			
☐ Light Metals Division Luncheon	$25	_____	$_____C
☐ Tables of 8	$200	_____	$_____L8
Table Sign to Read: _____			
☐ Roger Staehle Honorary Dinner	$55	_____	$_____SD

PLANT TOUR:

	FEE	NO.	TOTAL
Thursday 2/15/01			
☐ Nasa Michoud Assembly Facility	$35	_____	$_____NT

2001 MEMBERSHIP DUES—FOR CURRENT TMS MEMBERS ONLY:

Advanced registrations received after December 31, 2000 must be accompanied by your 2001 dues payment to be processed at the member fee.

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

TOTAL FEES PAID: $ _____

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: _____

REFUND POLICY: Written requests must be mailed to TMS, post-marked no later than January 22, 2001. A $50 processing fee will be charged for all registration cancellations.

Housing Registration Form

FOR THE TMS ANNUAL MEETING AND EXHIBITION ■ FEBRUARY 11–15, 2001 ■ NEW ORLEANS, LOUISIANA

RESERVATIONS MUST BE RECEIVED AT THE HOUSING BUREAU BY JANUARY 4, 2001

RETURN HOUSING FORM: (choose only one option)
Hours of operation: 8:00 am–5:00 CST Monday–Friday
- VISIT www.tms.org
- CALL 847-940-2153 (International); 800-424-5250 (Domestic)
- FAX to 847-940-2386 (International); 800-521-6017 (Domestic)
- MAIL to TMS Housing Bureau, 108 Wilmot Road, Suite 400, Deerfield, IL 60015-0825

TMS 2001 130TH ANNUAL INTERNATIONAL MEETING & EXHIBITION

Arrival Date _____ Departure Date _____

Last Name _____ First Name _____ MI _____

Company _____

Street Address _____

City _____ State/Country _____ Zip/Postal Code _____

Daytime Phone _____ Fax _____

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

Accompanying Person _____

☐ Non-Smoking Room Requested ☐ Special Needs

INDICATE 1st, 2nd AND 3rd HOTEL CHOICE AND TYPE OF ACCOMMODATION

1. _____
2. _____
3. _____

If all three (3) requested hotels are unavailable, please process this reservation according to: (check one) ☐ Room Rate ☐ Location

Accommodations (check one)

☐ 1 person/1bed ☐ 2 people/1 bed
☐ 2 people/2 beds ☐ 3 people/2 beds
☐ 4 people/2 beds ☐ One bedroom suite
☐ Two bedroom suite

Hotels

Headquarters
Hilton Riverside
$188/Classic s/d
$208/Deluxe s/d
$243/Towers s/d

Doubletree Hotel
$169/single
$189/double

Embassy Suites
$179/single
$199/double

Hampton Inn & Suites
$164/single
$164/double

Hilton Garden Inn
$182/single
$202/double

Holiday Inn Select
$165/single
$165/double

Marriott Hotel
$199.00/single
$199.00/double

Wyndham Canal Place
$195/single
$195/double

Wyndham Riverfront Hotel
$179/single
$199/double

CONFIRMATIONS
Confirmation will be mailed, faxed or e-mailed to you from the TMS Housing Bureau 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 2 weeks, please call the Housing Bureau.

CHANGES/CANCELLATIONS
All changes and cancellations in hotel reservations must be made with the TMS Housing Bureau on or before January 4, 2001 to avoid a $16 processing fee. After January 4, 2001 and prior to 72 hours before arrival date, changes and cancellations must be made with your assigned hotel. Your deposit will be refunded less a $16 processing fee. Any cancellations made within 72 hours of the arrival date will result in forfeiture of the full deposit.

RESERVATIONS/DEPOSITS
All reservations are being coordinated by the TMS Housing Bureau. Arrangements for housing must be made through the TMS Housing Bureau and NOT with the hotel directly. All housing reservation forms must be received by Thursday, January 4, 2001. Deposits: A $150 per room deposit is required to make a reservation; a $300 deposit is required for a one-bedroom suite and a $450 deposit is required for a two-bedroom suite. The deposit amount is payable by credit card or check (mail only). The credit card will be charged immediately. If paying by check, mail your payment with this completed housing form. All checks must be made payable to the TMS Housing Bureau in US funds drawn on a US bank. No wire transfers will be accepted.

CREDIT CARD:

☐ Visa ☐ MasterCard ☐ Diners Club ☐ American Express ☐ Discover

Expiration Date: _____

Card No.: _____

Cardholder Name: _____

Authorized Signature: _____

Please read all hotel information prior to completing and submitting this form to the Housing Bureau. Keep a copy of this form. Use one form per room required. Make additional copies if needed.

Continuing Education Registration Form

FOR THE TMS ANNUAL MEETING AND EXHIBITION ■ FEBRUARY 11–15, 2001 ■ NEW ORLEANS, LOUISIANA

PLEASE CHOOSE ONLY ONE OPTION FOR SENDING FORM.

WEB	Take advantage of the convenience of on-line pre-registration via the TMS website: **http://www.tms.org** Web registration requires credit card payment.	**FAX**	Fax this form to TMS Cont. Education Dept. **USA 724-776-3770** Fax registration requires credit card payment.	**MAIL**	**Return this form with payment to** Cont. Education Dept. TMS 184 Thorn Hill Road Warrendale, PA 15086

TMS2001 130TH ANNUAL INTERNATIONAL MEETING & EXHIBITION

Advance Registration Deadline: January 22, 2001
PAYMENT MUST ACCOMPANY FORM.
Forms received past this date will be processed at the on-site fee structure.
Please print or type

Member of: ☐ TMS ☐ ISS **Member Number:** _____
☐ SME ☐ SPE

☐ Dr. ☐ Prof. ☐ Mr. ☐ Mrs. ☐ Ms. _____
LAST NAME FIRST NAME MIDDLE INITIAL

Employer/Affiliation: _____

Address: _____

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

Telephone: _____ Fax: _____

E-Mail Address: _____

CONTINUING EDUCATION SHORT COURSES	ADVANCE TO 1/22/00		ON-SITE AFTER 1/23/00	
Check your selections. *See brochure for cancellation and refund policies.*	**MEMBER**	**NON-MEMBER**	**MEMBER**	**NON-MEMBER**
Excellence in Professional Communications ☐ Sunday, 2/11/01	$260	$310	$260	$310
Molten Salt Chemistry and Process Design: from Smelter to Casthouse ☐ Saturday, 2/10/01 & Sunday, 2/11/01	$645	$735	$695	$785
Heat Treatment of Wrought and Cast Aluminum Alloys ☐ Saturday, 2/10/01 & Sunday, 2/11/01	$645	$735	$695	$785
Total	$ _____			

PAYMENT ENCLOSED:

☐ **Check, bank draft, or money order made payable to TMS**—Payment shall be made in US dollars drawn on a US bank.

☐ **Credit Card**—Card No.: _____ Expiration Date: _____

☐ Visa ☐ MasterCard ☐ Diners Club ☐ American Express

Cardholder Name: _____

Signature: _____

REFUND POLICY:
Written request must be mailed to TMS, post-marked no later than January 22, 2001. A $50 processing fee will be charged for all registration cancellations.

Accompanying Tour Registration Form

FOR THE TMS ANNUAL MEETING AND EXHIBITION ■ FEBRUARY 11–15, 2001 ■ NEW ORLEANS, LOUISIANA

DESTINATION MANAGEMENT, INC. NEW ORLEANS has arranged tours for members/guests of the TMS Annual Meeting & Exhibition, February 11–15, 2001.

Please make your reservation by noting choice of tour, day, and time. Pre-sold tickets will be held at the tour desk located in La Louisiane Ballroom A in the Ernest N. Morial Convention Center.

TMS2001
130TH ANNUAL INTERNATIONAL MEETING & EXHIBITION

DESCRIPTION	DATE/TIME	PRICE	NO.	AMT DUE
New Orleans City Tour	Monday, February 12, 2001 ■ 9:30 am–12:30 pm	$18	_____	$_____
Jean Lafitte Swamp Tour	Tuesday, February 13, 2001 ■ 9:30 am–12:30 pm	$35	_____	$_____
Mardi Gras World/ New Orleans Mint Museum	Wednesday, February 14, 2001 ■ 12:30 pm–4:00 pm	$28	_____	$_____
			Total:	**$**_____

Name: _____

Address: _____

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

Phone: _____ Fax: _____

PAYMENT OPTIONS

☐ Check Enclosed (Remit in U.S. Funds)

☐ Charge My Account: ☐ Visa ☐ MasterCard ☐ Discover ☐ American Express

Card No.: _____ Expiration Date: _____

Cardholder's Name: (please print) _____

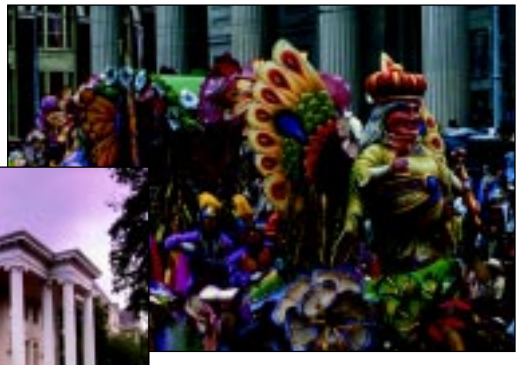
Signature: _____

WE CANNOT ACCEPT PHONE ORDERS

Please make checks payable to and mail to:

Destination Management, Inc. New Orleans
610 South Peters Street, Suite 200
New Orleans, Louisiana 70130
Fax: (504) 592-0529
Attn: Cheryl

- Please have your reservations in by January 29, 2001.
- Cancellations must be received in writing by February 5, 2001.
- You will receive a full refund for any cancellations received by this date.
- Credit card orders may be faxed to 504/592-0529.
- All tours, unless otherwise indicated, are based on 30 participants.
- DMI reserves the right to cancel any of these tours should minimum number not be met.



Q: How do I maximize my investment in the 2001 TMS Annual Meeting?

For years, TMS annual meeting proceedings volumes have acted as important reference sources for their fields. This year's proceedings selection includes new volumes in TMS's popular Light Metals and EPD Congress series:

- **Light Metals 2001** is the newest installment in the Light Metals series, which has become the definitive annual reference source in the field of aluminum production and related light metals technologies. The Light Metals 2001 package includes both the hardcover proceedings volume and CD-ROM.
- **EPD Congress 2001** is the newest edition in the Extraction and Processing Division Congress series, which has become the definitive annual forum for new technological developments in the process metallurgy community.

THIS YEAR, TMS ALSO OFFERS THE FOLLOWING SYMPOSIUM PROCEEDINGS VOLUMES:

- Chemistry and Electrochemistry of Corrosion and Stress Corrosion Cracking
- Cyanide: Social, Industrial, and Economic Aspects
- Elevated Temperature Coatings
- Innovations in Processing and Manufacturing of Sheet Materials
- Magnesium Technology 2001
- Properties of Nanocrystalline Materials
- Structural Biomaterials of the 21st Century

VISIT THE PUBLICATIONS SALES AREA TO PURCHASE ANNUAL MEETING PROCEEDINGS VOLUMES

YOU CAN ALSO RESERVE COPIES OF THE FOLLOWING PROCEEDINGS VOLUMES, WHICH WILL BE AVAILABLE SOON FROM THE 2001 TMS ANNUAL MEETING:

- Automotive and Joining Aluminum
- Lightweight Alloys for Aerospace Applications (Available in portable document format.)

. . . critical information for surviving the aggressive pace of 21st Century business.

A: *PURCHASE A TMS CONFERENCE PROCEEDINGS VOLUME AND KEEP VALUABLE TECHNICAL INFORMATION AT YOUR FINGERTIPS.*

2001 EXHIBITION
Exhibit Hours
9:30 AM - 3:00 PM
Ernest N. Morial Convention Center - Hall A

★★★

Ice Cream Treat
12:15 PM - 2:15 PM
Ernest N. Morial Convention Center - Hall A

★★★

INSTITUTE OF METALS LECTURE & ROBERT MEHL MEDALIST
"New Discoveries in Deformed Metals"
12:00 PM - 2:00 PM
Hilton Riverside Hotel - Grand Salon A, Sect. 1,2,4,5

★★★

TECHNICAL DIVISION LUNCHEON & LECTURE
Light Metals Division Luncheon
12:00 PM - 2:00 PM
Hilton Riverside Hotel - Grand Ballroom A

★★★

Roger Staehle Honorary Dinner
6:00 PM - 10:00 PM
Hilton Riverside Hotel - Marlborough A&B

Alumina & Bauxite: Bayer Process Development

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Gerald I.D. Roach, Alcoa World Alumina, Alcoa Technical Center; Jacques M. Mordini, Aluminium Pechiney, Gardanne 13541 France

Wednesday AM Room: 217
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Frank Kimmerle, Alcan International, Ltd., Arvida Res. and Dev. Centre, Jonquiere, Quebec, Canada

8:30 AM
Wet Oxidation in QAL High Temperature Digestion for Plant Productivity Improvement: *John Anderson*[1]; Lyndon Armstrong[1]; Daniel Thomas[1]; [1]Queensland Alumina, Ltd., R&D Grp. Tech. Svcs. Dept., P.O. Box 1, Gladstone, Queensland 4680 Australia

Oxygen injection into Bayer digestion of bauxite is known to enhance degradation of organics to improve liquor and hydrate colour. The benefit on precipitation operations including liquor productivity is not well documented in the public literature or established in plant practice. This paper will outline results for Weipa bauxite digestion at high temperature QAL conditions with various oxygen charges up to the solubility level, in single and multiple digestion-precipitation cycles. This research extends the Bayer published knowledge by quantifying the impact of Wet Oxidation on Weipa digested liquor in terms of hydrate yield, sizing, classification performance, bound soda incorporation, organic degradation pathways, and by-product oxalate and carbonate formation. This work advances industry evaluation of such a process for a high temperature plant where achievable A/C at the Press Floor determines L-P supersaturation.

9:00 AM
Alkali Cations-Role and Effect on Gibbsite Crystallisation: *Joanne S.C. Loh*[1]; Helen R. Watling[2]; Gordon M. Parkinson[1]; [1]A. J. Parker Cooperative Research Centre for Hydrometallurgy, Curtin University of Technology, Sch. of Appl. Chem., G. P.O. Box U1987, Perth, Western Australia 6845 Australia; [2]A. J. Parker Cooperative Research Centre for Hydrometallurgy, CSIRO Mins., P.O. Box 90, Bentley, Western Australia 6982 Australia

One of the most important quality control parameters in the alumina industry is the soda content in gibbsite, which is retained in the product after calcination to produce alumina. Sodium is incorporated in the gibbsite precipitation step of the Bayer process, implying that the incorporation of sodium into the crystal lattice is closely associated with the mechanism of gibbsite precipitation. As part of an ongoing fundamental study of the mechanisms of gibbsite crystallisation, we have studied the effects of substituting potassium and caesium for sodium in synthetic Bayer liquors. Bulk precipitation tests indicate that desupersaturation is faster from cation substituted synthetic Bayer liquors, with nucleation dominating the other growth processes of agglomeration and ordered growth. The effect of seed type has been investigated, with results suggesting that the seed morphology has a significant effect on the aluminium desupersaturation and gibbsite growth rates whilst the alkali cations may have an effect on the product morphology.

9:30 AM
Study on the Application and Mechanism of Cationic Surfactant on the Precipitation of Sodium Aluminate Liquor: *Yanli Xie*[1]; [1]Northeasten University, Mat. and Metall. Div., Shenyang, Liaoning 110006 PRC

More and more additives had been used in the process of seeded precipitation of sodium aluminate liquor, such as Nalco CGM, Alclar CM 5159, etc. They increased the particle size of gibbsite but couldn't enhance the precipitation of sodium aluminate liquor. In order to make up the loss some cationic surfactant was studied in this paper. As a result, we found that the application of cationic surfactant in precipitation of sodium aluminate liquor could not only increase the precipitation rate but improve the quality of hydroxide alumina.the optimum quantity of surfactant added was decided, the mechanism of enhancing seeded precipitation process by adding cationic surfactant was also discussed in detail.

10:00 AM Break

10:20 AM
Evaluation of Process Parameters that Allow Processing Pijiguaos Bauxite with Reactive Silica <1.1 %: *Anibal Martinez*[1]; *Jesus Noya*[1]; Maritza Faneitte[1]; [1]C.V.G Bauxilum, Proc. Quality Cont., Zona Indust. Matanzas, Pto. Ordaz, Bolivar 8011 Venezuela

C.V.G Bauxilum Processes Pijiguaos Bauxite whose main characteristic is low content (1-2%) reactive silica. The adaptation of the plant to process this bauxite included a predesilication step prior to digestion, based on a reactive silica content between 1.7-2.0%. This caused a strong variation in the bauxite chemical composition. To minimize this variation and to decrease the specific bauxite consumption, a reduction was required of the mineral reactive silica content, which implies processing bauxite with a value <1.1%. To process bauxite with this level of reactive silica,(to achieve a 2 MM TPY alumina production level) and to guarantee its control in the liquor, a residence time greater than 10 hours is required, however the design maximum time is 9 hours which does not guarantee control of this impurity in the process. The present work explores the possibility to adapt the plant to this new situation and to quantify the impact of this factor in the predeslicated liquor silica concentration. It also correlates laboratory and plant data and evaluate new process parameters that guarantee control of this impurity in the final product and reduces the potential negative impact in the rest of the process.

10:50 AM
The Influence of Lime Addition Amount on Scaling Rate in Preheating Process of Diasporic Bauxite Slurry: *Zhonglin Yin*[1]; *Songqing Gu*[1]; [1]Zhenzhou Light Metals Research Institute, Shangjie, Zhengzhou 450041 PRC

The relationship between the scaling rate and lime addition amount in Bayer preheating process of diasporic bauxite slurry has been

studied. The scale formed in preheating process of diasporic bauxite slurry mainly contains Si-containing, Ti-containing and Mg-containing minerals. Raising lime addition amount can decrease and restrain the formation of Si-containg scale and Mg(OH)2 scale. Lime addition amount does not have an obvious influence upon the scaling rate of Ti-containing minerals. Properly increasing the lime addition amount should decrease the general scale formation in preheating process of diasporic bauxite slurry and this opinion has been verified by industrial results.

11:10 AM

Improvements on Digestion in Bayer Process: *Guoyao Gan*[1]; *Longzhang Wang*[1]; [1]Pingguo Aluminum Company, Pingguo, Guangxi 531400 PRC

In order to increase the capacity of alumina production and decrease the energy consumption in Pingguo Alumina Plant, improvements on control techniques for digestion were researched and developed. They include improving the control of chemical composition of slurry to be digested and digestion operation status and energy consumption. Very good performances and technical and economical figures for digestion process with diaspore bauxite have been obtained by these improvements in Pingguo alumina refinery.It is proved by several years' practice that under the condition of keeping digestion yield more than 93%, alumina capacity is increased by 33%, Rp in digested slurry is increased from 1.11 to 1.18 and energy consumption in digestion process is cut down from 2.2 tons live steam per ton of alumina to 2.0 tons live steam per ton of alumina.

Aluminum Joining-Emphasizing Laser and Friction Stir Welding:
Session 2 - Joining Aluminum for Automotive and Structural Applications

Sponsored by: Light Metals Division, Aluminum Association
Program Organizers: John A.S. Green, The Aluminum Association, Washington, DC 20006-2168 USA; J. Gilbert Kaufman, Columbus, OH 43220-4821 USA

Wednesday AM Room: 214
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Peter Pollak, The Aluminum Association, Washington DC 20006 USA; J. Gilbert Kaufman, Columbus, OH 43220-4821 USA

8:30 AM

New Developments in Arc Welding for Automotive Applications: *Ian D. Harris*[1]; [1]Edison Welding Institute, 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

Arc welding is a suite of highly productive and flexible fabrication tools which is widely used across a broad spectrum of industries. The automotive industry is a significant user of arc welding at the component and subassembly stage of the manufacturing operation, particularly GAS Metal Arc Welding (GMAW). Autobody and vehicle assembly, on the other hand, currently uses very little arc welding. Trends towards spaceframe vehicle bodies to save weight and increase stiffness compare to traditional sheet metal body-in-white (BIW) offer increased opportunities for high-productivity arc welding in the body assembly plant, and/or Tier 1 suppliers. This paper discusses some of the current trends and future opportunities in the use of arc welding for fabrication of vehicle frames (traditional and spaceframe) and subassemblies, including suspensions, engine cradles, and similar parts in steel and aluminum alloys.

9:00 AM

Filler Alloy Selection for Welding Aluminum Automotive Components: *Tony Anderson*[1]; [1]AlcoTec Wire Corporation, 2750 Aero Park Dr., Traverse City, MI 49686-9154 USA

We shall examine the possibilities for improvements associated with both quality and productivity through selection of the most suitable aluminum filler alloy for specific applications. Typically there are a variety of filler alloys available which may be used to join any given base alloy. However there are a number of variables associated with the selection of the most suitable filler alloy to be used. The selection of the most suitable filler alloy should be based on the completed weld performance requirements. If a weld is supporting a major structural member which is subjected to high stress levels then strength may be of primary concern. In this situation the benefits relating to the significant differences in filler alloy shear strength should be understood. If a component is non-structural and required to be part of a sealed cooling or heating system the main consideration is to produce leak free welds. In this situation the advantages of a filler alloy to flow into and seal the joint is of pri me importance. Benefits in productivity through lowering leakage rates have been experienced through changing to filler alloys which provide improvement within this filler alloy characteristic. Some considerations for the selection of a filler alloy are typically. Ease of welding-this being the relative freedom from weld cracking. By use of hot cracking sensitivity curves for the various aluminum alloys we can establish the filler alloy/base alloy crack sensitivity. Strength of welded joint-Consideration of the tensile strength of groove welds and shear strength of fillet welds when welded with different filler alloys. Ductility. A consideration if forming operations are to be used during fabrication and may also be a design consideration for service. Corrosion Resistance–A consideration for some service conditions and are typically based on exposure to fresh and salt water. Sustained Temperature Service–The reaction of some filler alloys and base alloys at sustained elevated temperature may promote premature component failure due to stress corrosion cracking. Color Match–Base alloy and filler alloy color match after anodizing can be of major concern in some cosmetic applications. Post Weld Heat Treatment–The ability of the filler alloy to respond to post weld heat treatment associated with joint design. In summary, there are many considerations relating to the selection of the most suitable filler alloy for a specific base alloy and completed product application. The understanding of these variables is a significant aspect in selecting the filler alloy which will provide for improved characteristics for a specific welded component.

9:30 AM

Structural Crimping: Its Potential for High Performance, Low Cost Joining: *Glenn S. Daehn*[1]; Peihui Zhang[1]; [1]Ohio State University, Mats. Sci. and Eng. Dept., 2041 College Rd., Columbus, OH 43210 USA

One of the noteworthy features of high velocity metal forming is that under many circumstances one may compress a tube against a mandrel or expand it into an opening and it will lock into that opening effectively without springback. While there has been little academic study of this mode of joining it has been used commonly in industry, including for high performance applications such as joining ends on torque tubes in commercial aircraft. It seems that in general the use of high velocity forming as accomplished by electromagnetic forming can have a much more general range of applications than it presently does. One of the limiting factors is that the performance of such joints is not well understood. Here we will show that existing numerical models can shed much light on this process and the resulting joint performance.

10:00 AM Break

10:15 AM

Making Aluminum More Competitive in Infrastructure Applications: *J. Randolph Kissell*[1]; Brian J. Malloy[2]; [1]The TGB Partnership, 1325 Farmview Rd., Hillsborough, NC 27278 USA; [2]Reynolds Metals, 13203 N. Enon Church Rd., Chester, VA 23836 USA

Aluminum alloys have many and diverse infrastructure applications including window and skylight frames, culverts, light poles, curtainwalls, rain-carrying goods, metal roofing, space frames, and others. In fact, about 3 billion pounds of aluminum are used annually in American building and construction markets. Aluminum is usually used because it is easy to fabricate, corrosion resistant, has a high strength to weight ratio, and is easy to join by a variety of commercial processes. Yet to keep up with competing materials, aluminum alloys and joining techniques need to improve. Recent

developments that may make aluminum more competitive in this market are discussed in this paper.

10:45 AM

The Effect of Postweld Heat Treatment on the Mechanical Properties of Aluminum Alloy 6082 T6 Alloy: *Frank Feng*[1]; [1]Alcan International, Ltd., Kingston Res. and Dev. Ctr., P.O. Box 8400, 945 Princess St., Kingston, Ontario K7L 5L9 Canada

Aluminum alloy 6082 T6 plates were welded by GMAW process in this study. The HAZ in the weld exhibited significant softening due to the welding thermal cycle. Accordingly, the joint efficiency of the weld is only about 0.65 in welding 12 mm thick plate. To improve the welding joint strength, different post weld treatment were conducted. Natural aging showed no noticeable effect. However, postweld heat treatment (artificial aging) at 175°C for 8 hours in oil bath is effective in recovering the weld strength. The ultimate tensile strength and the yield strength were increased by 25 MPa and 75 MPa respectively. Through microstructual investigation with SEM and TEM, it was found that this substantial strength improvement is the result of fine particle precipitation in the HAZ during the artificial aging process.

11:15 AM

An Investigation of the Bearing Strength of Some Aluminum Alloys: *Craig Menzemer*[1]; [1]University of Akron, Dept. of Civil Eng., Akron, OH 44325-3905 USA

Bolted and riveted joints have been widely used in aluminum structures. Some examples include building structures and cladding, roofing systems, railcars, intermodal shipping containers and trailer bodies. Proper joint design requires due consideration of all appropriate limit states. In this study, both bearing capacity and hole deformation of AA5052, AA5454 and AA3003 alloys were evaluated experimentally for confined and unconfined joints. Failure modes included excessive deformation, net section tensile rupture and tear out of the fastener. A recommendation for an allowable bearing stress equal to twice the ultimate tensile strength of the connected material is provided. In addition, elimination of the bearing yield limit state for bolted connections is suggested.

Aluminum Reduction Technology - Stream I: Modeling and Magnetics

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: John Chen, University of Auckland, Department of Chemical & Materilas Engineering, Auckland, New Zealand; Eric Jay Dolin, USEPA, MC 6202J, Washington, DC 20460 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday AM — Room: 206-207
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Torstein Utigard, University of Toronto, Dept. of Metall. & Mats. Sci., 184 College St., Toronto, Ontario M5S 3E4 Canada; Vinko Potocnik, Alcan Internatinal, Ltd., Arvida Res. and Dev. Ctr., 1955 Boul. Mellon, Jonquière, Québec G7S 4K8 Canada

8:30 AM

Multimedia for Training Cell Operators: Bernard Bouchard[2]; Claude Fradet[1]; *Jonathan Lapointe*[3]; Gerald Rivard[3]; [1]Alcan International Ltd., Arvida R&D Ctr., 1955 Mellon Blvd., P.O. Box 1250, Jonquiere, Quebec G7S 4K8 Canada; [2]Alcan Primary Metal, Arvida Work, Jonquiere, Quebec, Canada; [3]Groupe Vision Interactif, 72 West Jacques Cartier St., Chicoutimi, Quebec G7J 1G1 Canada

A software was developped to assist the training of the cell operators. Since it is dificult to visualize what is happening in a Hall Heroult cell, it takes months of training and years of experience to develop good cell operators. The use of Multimedia and 3D simulations are helpful to understand the links between the problems we observe on cells and their causes and corrective actions. Multimedia increases knowledge retention and reduces training duration. It helps to get more efficient operators in a shorter time.

9:00 AM

Planning Smelter Logistics: A Process Modeling Approach: *Ingo L. Eick*[1]; Detlef Vogelsang[1]; Andrae Behrens[2]; [1]VAW Aluminium-Technologie GmbH, Process Model., Georg von Boeselagerstr. 25, Bonn 53117 Germany; [2]Gesellschaft fuer Prozessautomation & Consulting GmbH, Altchemnitzer Strasse 52/54, Chemnitz 09120 Germany

A dynamic logistic model, based on High Level Petri Nets, was generated to aid the planning of potroom activities, potroom traffic and logistic equipment needed for a smelter expansion project. This model includes all relevant pot-tending operations, such as anode changes with cavity cleaning and covering, metal tapping, alumina feeding, beam raising, bath tapping, pot stoppage and start-up, gantry transfer and crane maintenance exchange. The workflow and traffic patterns in the smelter are simulated to analyze equipment utilization and bottlenecks. The rule-driven model incorporates such features as operations scheduling, collision detection as well as the entire material handling process. Pointers for the optimization of the potroom layout, e.g. the consequences of an additional passageway, can be deduced. This discrete event simulation predicts the capacity utilization of logistic equipment, like cranes and service vehicles. A visualization surface provides a dynamic follow-up to all simulated procedures and traffic activities of the model.

9:25 AM

Influence of Different Energy Models on Overall Balancing of Primary Aluminum Smelting: *Matthias Dienhart*[1]; Zeynel Alkan[1]; Sebastian Briem[1]; Olaf Kugeler[1]; Rainer Quinkertz[1]; Kurt Kugeler[1]; [1]RWTH Aachen, Instit. for React. Safe. and React. Tech., Eilfschornsteinstr. 18, Aachen 52062 Germany

Assessing an overall balance of primary aluminum production shows the substantial influence of the energy supply. Especially the kind of electricity supply of primary aluminum smelters can cause significant environmental effects. Due to this fact the electricity supply systems of aluminum smelters have to be indicated and described by appropriate energy models. Up to now different methods are used to model the energy supply of primary aluminum smelters. Beside the most commonly used "national electricity mix" model the authors will introduce the "national or regional base-load mix" and the "contract mix" model. This paper will point out the advantages and disadvantages of these three energy models. After this the influence of the chosen model on the primary energy demand and carbon dioxide equivalent emissions will be shown on the basis of chosen aluminum producing countries in 1997. Concluding the authors recommend the best suited power supply model for overall balancing primary aluminum smelters.

9:50 AM

Development the System of Forecast the World Prices of Aluminium and Its Application for Commercial Activity: *Boris Arlyuk*[1]; Michael Fiterman[1]; [1]Alumconsult, Ltd., St. Petersburg, Russia

The developed strategy of trader, manufacturer and consumer in operations of sale and purchase of aluminium at LME is based on two components. First is a qualitative and quantitative forecasting LME prices of primary aluminium for the future periods. Second-definition of the optimum moment and volume of purchase or sale the metal. For the forecast of the future prices is developed the system of forecasting the world prices of aluminium in medium term (about 3 months forward) period which is successfully applied since September 1999. By developed system beside the quantitative forecasts of the prices, the probability is estimated that the maximum future prices will be above current actual price and the similar probability for the minimal future prices. For acceptance the optimum decisions on purchase and sale of metal the economic model of behaviour of trader is developed. This model analytically connects profit at operations purchase-sale to strategy of these operations, accuracy of the forecasts, both material and financial restrictions in activity of the company. The optimum strategy of trader is expressed as the law of acceptance the decisions on the moment and volume of purchase or sale depending on results of the

current forecast and stock of metal, and also volume of working capital. The strategy of trader is optimised using the actual LME data for the period from 1989 till 2000. Thus has appeared that the possible profit makes from $136 up to $323 per 1 t sold metal (profit is given excluding the interest for bank credit 8% annually, depending on volume of warehouse and financial resources of the company). At usage the model for sale the metal from producer to trader with subsequent additional pricing is providing increase of the income in the average by 20 $/t. Similar system is developed for consumers.

10:15 AM Break

10:25 AM

Albras Magnetic Compensation: Jose Gilvando Andrade[1]; Jose Juarez Borges[1]; Neil Baker[1]; Nelcindo Gonzales[1]; Ricardo Lara[1]; *Guilherme Epifanio*[1]; [1]Albras Aluminio Brasileiro SA, Redução-Fundição, Estrada PA 483 Km 21, Vila Murucupi, Barcarena, Para 68447 000 Brasil

The busbar configuration has a very significant effect on cell stability. It can be changed to improve stability and current can be safely increased. Due to good results obtained from 13 test cells, that had been operated for more than 12 months, Albras decided to move all pots to that new busbar configuration. So, from November 1997 to February 1999, 864 AP13 Albras pots were magnetically compensated by re-arranging the busbars. In order to accomplish this task in such a short period, a procedure for "hot conversion" was developed, i.e., most of the pots were converted without being shutdown. As a result, it was possible to convert up to 4 pots/day. No operational disturbances were reported. The use of magnetic shields allowed busbar welding "on line" in the potroom basements. Five welding crews worked simultaneously and safety measures received special attention mainly regarding the grounding system, electrical shock prevention and heat stress. No accidents have been reported. Since the conversion, Alb ras has continuously improved its potline operating results such as current efficiency and energy consumption. In addition, as the pots became more stable, it was possible to start a current increasing program, now in progress. At the bottom line the Albras magnetic conversion has proven to be a technically and economically sound project. This paper presents the project development and methodology, showing the updated achieved results and discussing possible future developments.

10:50 AM

Comparison of Measured and Calculated Metal Pad Velocities for Diffrerent Prebake Cell Designs: *Vinko Potocnik*[1]; Frédéric Laroche[1]; [1]Alcan Internatinal, Ltd., Arvida Res. and Dev. Ctr., 1955 Boul. Mellon, P.O. Box 1250, Jonquière, Québec G7S 4K8 Canada

Metal velocities were measured in different cell designs using iron rods and, in some cells and some locations, also Alcan portable vane flow meter. The measurements were compared with velocities calculated by ESTER/PHOENICS with diffrent turbulent viscosity parameters. The agreement between calculated and measured velocities is good for a specific set of model parameters. The importance of well defined boundary conditions used in the electric current distribution model is discussed.

11:15 AM

Stability of Hall-Heroult Cells: *Kjell Kalgraf*; [1]Elkem Research, P.O. Box 8040, Vaagsbygd, Kristiansand N-4602 Norway

Stability of Hall-Heroult cells is determined by Navier-Stoke's equation. The equation may be divided into two independent equations by taking the divergence and curl of Navier-Stoke's equation. The divergence part determines the motion and stability of magneto-gravitational waves. When taking the curl, all fields determined by gradients disappear, i.e. gravitation and pressure disappears, and we are left with an equation that contains only magnetic field and velocity. This equation states that the magnetic field is driving a circulation pattern in the cell, and the equation has stability conditions of its own, related only to magnetic fields and geometry. The associated waves are a slow type of wave called Alfven waves, and have oscillation periods that compares well with periods observed in cells. Alfven waves are strongly damped in the electrolyte, but can propagate in the metal which has a high electrical conductivity, and are seen as different types of metal instabilities. We develop stability criteria for both types of equations and supply experimental data.

11:40 PM

Effect of Cell Current Distribution on the Magnetic Field Inside a Cell: *Sun Yang*[1]; Feng Nai-xiang[1]; Leng Zhengxu[2]; Shi Chongguang[2]; Xie Qingsong[2]; Wang Youlai[2]; [1]Northeast University, Shenyang, China; [2]Guizhou Aluminum Smelter, China

The surface line-charge method is described to calculate the magnetic field inside a 160kA prebaked cell. The effect of cell current distribution on the magnetic field inside the cell was calculated. It is shown that the magnetic field inside the cell are substantially affected by the cell current distribution.

Aluminum Reduction Technology - Stream II: Sampling & Sensors

Sponsored by: Light Metals Division, Extraction & Processing Division, Materials Processing and Manufacturing Division, Aluminum Committee, Pyrometallurgy Committee, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: Adrian Deneys, Praxair, Inc., Tarrytown, NY 10591 USA; Derek Fray, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3Q2 UK; Matt Krane, Purdue University, Department of Materials Engineering, West Lafayette, IN 47907; Markus Reuter, Delft University of Technology, Applied Earth Science, Delft 2628 RX The Netherlands; Fiona Stevens McFadden, University of Auckland, Chemistry and Materials Engineering, Auckland, New Zealand

Wednesday AM — Room: 230
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Fiona Stevens McFadden, University of Auckland, Chem. and Mats. Eng., Auckland, New Zealand; Adrian C. Deneys, Praxair, Applications Res. and Dev., Tarrytown, NY 10591 USA

8:30 AM

Control of Temperature in Aluminium Reduction Cells-Challenges in Measurements and Variability: *Fiona Jean Stevens McFadden*[1]; Daniel Whitfield[1]; Barry J. Welch[1]; [1]University of Auckland, Chem. and Mats. Eng. Dept., Auckland 92019 New Zealand

The temperature of the electrolyte is one of the key process variables in a reduction cell, as it has a strong influence on cell current efficiency. The variance in temperature, a measure of control performance, is typically higher than desired and smelter operators would generally speaking like to see an improvement. Measurement of temperature is made difficult by a number of factors such as the corrosive nature of electrolyte and the spatial and temporal variation resulting from the semi-batch, semi-continuous nature of the process. Difficulties also arise in the interpretation of the temperature measurement and feedback to control action, as temperature change can result from a change in excess aluminium fluoride, alumina concentration and/or superheat. Depending upon the cause of temperature variation the appropriate control action may vary.

9:00 AM

Development and Application of a Novel Sensor for Combined Bath Temperature and Cathode Voltage Drop Measurements in Aluminium Reduction Cells: Bernd Rollofs[2]; *Peter White*[1]; Rik Kelchtermans[1]; Neal Wai-Poi[2]; Paul Verstreken[1]; [1]Heraeus Electro-Nite International N.V., Prod. Mgmt., Centrum Zuid 1105, B-3530, Houthalen B-3530 Belgium; [2]Corus Voerde, Reduction, Huttenwerk Voerde, Scheleusenstrase D-46562, Voerde D-46549 Germany

Cathode voltage drop measurements are conducted by most smelters on a periodic basis. Whilst the measurement is necessary to

optimise target voltage, most smelters consider the present methods to be cumbersome, labour intensive and the accuracy can be questionable due to a heavy reliance upon human interpretation. A new technique of combining cryolite bath temperature measurement with CVD measurement has been developed which provides a novel method of measurement that significantly reduces, and in some cases, eliminates the labour cost associated with conventional CVD methods. This paper describes the development of the sensor, presents field tests to assess the sensor reproducibility and illustrates the recent application of the sensor into routine use at the Corus Voerde aluminium smelter in Germany. The benefits of this new technique, as perceived by the smelter are identified. Future possibilities for the sensor including monitoring of sludge and ridge build up are also discussed.

9:25 AM

Fuzzy Pattern Recognition of Temperature for Aluminum Electrolyte: *Zeng Shuiping*[1]; [1]North China University of Technology, Instit. of Auto., Shijinshan District, Beijing City, Beijing 100041 China

This paper develops a Fuzzy Pattern Recognition model for aluminum bath temperature based on a lot of data of temperature measurement in alumina reduction process. According to the these data the paper concludes the some fuzzy rules and makes inferences by max-min methods. In the end, the output, i.e., temperature of the system is defined by centroid defuzzification. Two methods are adopted in this pattern recognition. One is by fuzzy relation matrix; and the other is by Matlab Fuzzy Logic Toolbox. Verification results show the average relative error is less than 0.4%, which indicates the model can recognize the temperature of Al-electrolyte well. Therefore, if the method is applied in aluminum production, it can reduce the cost and the labor intensity of temperature measurement, and much benifits to the cell-control system.

9:50 AM

Real Time Alumina Distribution Measurement in Industrial Cells: *Richard G. Haverkamp*[1]; [1]Massey University, Inst. of Techn. and Eng., Private Bag 11222, Palmerston North 1015 New Zealand

The distribution of alumina in a smelter cell has been measured in real time. Measurements are performed with a hand-held or stand-mounted probe connected to a self-contained power supply and laptop computer adapted without shielding to work in a high magnetic field environment. Either individual measurement of alumina concentration or continuous alumina concentration measurements can be made. This gives another tool for monitoring cell electrolyte flows and alumina concentration gradients. These measurements can be used, for example, in conjunction with simultaneous temperature measurements to show the transport and dissolution of the alumina within the cell after feeding and to identify regions that are poorly fed by alumina. The system has been adapted to work in a range of electrolyte compositions with each calibration applicable within a limited range of cryolite ratio.

10:15 AM Break

10:25 AM

Development of Techniques for Measuring the Composition of Low Temperature Electrolytes: *Olivier Crottaz*[1]; Jennifer Purdie[1]; Vittorio de Nora[1]; [1]Moltech S.A., 9 Rte. de Troinex, Carouge, Ge 1227 Switzerland

Low temperature, cryolite-based electrolytes for the Hall-Héroult process continue to be of interest to the Aluminium industry. In a conventional Hall-Héroult cell, lower temperature operation could increase energy efficiency and improve materials performance. Low temperature electrolytes may also facilitate the operation of an inert anode, since the solubility of many metal oxides decreases with temperature. The ability to measure bath composition (AlF3, Al2O3 and other components) quickly and accurately will be critical to successful operation of low temperature electrolytes. Liquidus temperature becomes more sensitive to small changes in concentration as AlF3 concentration increases. Measurement and control of Al2O3 concentration would be particularly important to operation with inert anodes. In this paper the development of techniques for analysis of high AlF3 electrolytes for low temperature operation is discussed. The techniques include both laboratory analysis methods, and the use of available sensors for instantaneous measurement of superheat and alumina concentration, which together might allow a more rapid estimation of bath composition.

10:50 AM

Development of a Sensor to Measure Velocity in High Temperature Liquid Metals: *Stavros A. Argyropoulos*[1]; [1]University of Toronto, Dept. of Metall. and Mats. Sci., 184 College St., Toronto, Ontario M5S 3E4 Canada

This paper will describe the development of a sensor to measure localized velocity in high temperature liquid metals. This sensor utilizes a sphere which is immersed in a moving liquid metal. By measuring the melting time of sphere, the liquid metal velocity can be inferred. The elementals for the development of this sensor were carried out in a commercial purity aluminum bath and in liquid low carbon steel. In developing the sensor, we investigated a range of bath temperatures, as well as the impact of different sphere diameters. Results showed that the sphere melting time was related linearly to the magnitude of flow velocity for the range of velocities of 0-40 cm/sec and for bath superheats up to 100°C. How this sensor can be adapted to be used in other high temperature reactive liquid metals and liquid slags will be shown. In addition, a modification of this technique to detect direction of velocity in these high temperature hostile fluids will be described.

11:15 AM

Novel Solid State Sensor for Mg in Molten Al: *Girish Madhav Kale*[1]; [1]University of Leeds, Min. and Miner. Eng., Sch. of P, E & M Eng., Clarendon Rd., Leeds, West Yorkshire LS2 9JT UK

A novel and completely solid state sensor for measuring dissolved magnesium in molten aluminium for demagging and alloying operation has been developed employing two different novel solid electrolytes. Novel bi-phasic reference electrode materials have been used in designing the Mg-sensor. The solid state sensor has been tested between 963 to 1003K in molten Al-Mg alloys. The sensor was found to respond rapidly to change in concentration of Mg in molten alloy between 0.0003 to 0.03 weight fraction of Mg in Al. The present paper will discuss the preparation of solid electrolyte materials, electrical characterisation of the solid electrolyte materials by ac-impedance spectroscopy, preparation of the reference electrode materials, fabrication of sensor and testing of sensor in the laboratory.

11:40 AM

Sensitivity Analysis of the Thermal Detection of the Freeze Profile in an Aluminium Reduction Cell: Laszlo Istvan Kiss[1]; Rung Tien Bui[1]; Paul Desclaux[2]; *Pascal Boily*[1]; [1]University of Quebec at Chicoutimi, DSA, 555 boul de l'Universite, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International, CRDA STE, 1955 Boul Mellon, Jonquiere, Quebec G7S 3H5 Canada

The potentials of the thermal detection of the freeze profile were studied by laboratory experiments and by sensitivity analysis. In the experimental set-up, the freeze was represented by a sand layer placed on top of a carbon slab. Temperatures were measured in different points inside the carbon slab. The shape of the isothermal surface of the sand layer was identified by a numerical procedure based on the solution of the inverse heat conduction problem. The material properties, geometrical parameters, boundary conditions that are used in the inverse solution, influence the performance of the identification procedure. The sensitivity values of the freeze detection were determined for a wide range of the influencing parameters like the temperature of the interface, thermal conductivity of the freeze and cathode side-block (aging) as well as for non-zero parietal heat fluxes.

Aluminium Reduction Technology/Carbon Technology: Joint Session: "Responding to Inert Anodes and Other Technology Changes in The Aluminum Industry-The Benefits, Challenges and Impact on Present Technology"

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Les Edwards, CII Carbon, Chalmette, LA 70004 USA; Halvor Kvande, Hytdro Aluminium, Oslo, N-0240, Norway

Wednesday AM — Room: 215-216
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Nolan E. Richards, 117 Kingswood Dr., Florence, AL 35630 USA

Papers presented in this session along with a summary of the Panel Discussion will be published in the May 2001 volume of Journal of Metals

8:30 AM Introductory Comments by Program Organizers

8:40 AM
Cell and Smelter Energy Balances and Environmental Benefits: *Halvor Kvande[1];* [1]Hydro Aluminium, Oslo N-0240 Norway

Decomposition voltages, anode over-voltages and voltage equivalents of energy to make aluminum, total cell voltage, energy consumption and cell heat balance considerations for inert anode cells, global environmental benefits and CO_2 emissions under different energy consumption scenarios.

9:05 AM
Inert Anode Materials Challenges: *Don Sadoway[1]*; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Cambridge, MA 02139-4307 USA

Summary of different materials approaches (metals, cermets etc), fundamental advantages/disadvantages of each (corrosion rates, electrical conductivity etc), passivation issues, fabrication technology (lab vs commercial scale), material and fabrication cost considerations, mechanical handling.

9:30 AM
Cell Operation(s) and Metal Purity Challenges: *Jomar Thonstad[1]*; [1]Norwegian University of Science and Technology, Dept. of Matls. Techn. & Electroch., Trondheim, N-7491, Norway

Alumina concentration & solubility issues, electrolyte composition, cell control at lower inter-polar distances, current density and bubble release issues, handling of O_2 evolution, metal contamination and purity issues.

9:55 AM
Next Generation Vertical Electrode Cells: *Craig Brown[1]*; [1]Northwest Aluminum Technology, 13512 Wallingford Ave N, Seattle, WA 98133 USA

Vertical electrode cell concept and advantages (footprint, capital productivity, comparison to other electrolytic processes), additional challenges over horizontal or drained cathode/inert anode cells, slurry cell concept and update on progress.

10:20 AM Break

10:30 AM
Economics of Inert Anode and Wettable Cathode Retrofit Options: *Jeff Keniry[1];* [1]Alumination Consulting, 2 Governors Dr., Mt Macedon, Victoria 3441, Australia

Cost estimates for inert anode manufacture (materials & energy), carbon production cost estimates (material & energy), smelter energy requirements with different inert anode configurations (straight retrofit, combined with drained wettable cathode cell, vertical electrode cell), cell retrofit/construction costs under each scenario.

10:55 AM
Panel Discussion with Questions from Audience

Cast Shop Technology: Inclusions & Alloying

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: John F. Grandfield; CSIRO Australia, Preston, Victoria 3072 Australia; Paul Crepeau, General Motors Corporation, 895 Joslyn Road, Pontiac, MI 48340-2920 USA

Wednesday AM Room: 208-210
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: C. Edward Eckert, Apogee Technologies, New Kensington, PA USA; David DeYoung, Alcoa, Alcoa Center, PA USA

8:30 AM

Error Analysis of LiMCA II Data: *Martin Syvertsen*[1]; Thorvald Abel Engh[2]; [1]SINTEF Materials Technology, Process Metall. and Ceramics, Alfred Getz vei 2b, Trondheim 7491 Norway; [2]Norwegian University of Science and Technology, Dept. of Mats. Techn. and Electroch., Alfred Getz vei 2b, Trondheim 7491 Norway

LiMCA II is widely used for monitoring inclusion content in aluminium melts during melt treatment and casting. The advantages of LiMCA II compared to other inclusion measurements techniques as PODFA, LIAS, Alcoa samples, UC samples, etc. is that LiMCA II measures the inclusion number (or fraction) size distribution semi-continuously during a the cast. The time dependency of the filtration efficiencies may be determined as soon as the data can be analyzed. However, LiMCA II measurements and filtration efficiencies are often presented without any error estimates. This is because the present LiMCA II does not automatically calculate them.
This paper shows how the choice of size classes (in size distributions), and voltage and current noise introduce uncertainties. Also, because the inclusion counting process is Poisson-distributed, the counted numbers themselves introduce uncertainties in the measurements. This applies both for the number size distributions, the time variation of the volume fraction, and the removal efficiencies.

8:55 AM

Effect of In-line Processes on LiMCA Measurement of Inclusions: *Mark Cooksey*[1]; Tiffany Ware[1]; Malcolm Couper[1]; [1]Comalco Research & Technical Support, 15 Edgars Rd., Thomastown, Victoria 3074 Australia

Over recent years, the Liquid Metal Cleanliness Analyser (LiMCA) has become a commonly used tool for measuring the number and size of inclusions in molten aluminium. LiMCA generally provides a good measure of inclusions in the melt, but in-line processes can influence the results obtained. For example, measurements cannot distinguish between inclusions and micro-bubbles produced by a degasser, so that the performance of the degasser with respect to inclusions is difficult to assess. One method of reducing the impact of bubbles is to use a probe that is designed to prevent bubbles from passing through the measurement orifice. It has also been suggested that measuring during the pressure cycle (rather than vacuum cycle) allows bubbles to float out and hence not cause interference. However, these methods may effect the measurement of inclusions. Controlled production conditions have been used to better understand the impact of these methods on LiMCA inclusions measurement.

9:20 AM

The Influence of Grain Refiners on the Efficiency of Ceramic Foam Filters: *Nicholas Grant Towsey*[1]; Wolfgang Schneider[1]; Hans-Peter Krug[1]; Angela Hardman[2]; Neil Keegan[3]; [1]VAW Aluminium, Casting Technology, R&D, Georg-von-Boeselager Str. 25, Bonn 53125 Germany; [2]London & Scandinavian Metallurgical Company, Ltd., Rotherham, South Yorkshire UK; [3]Pyrotek Engineering Materials, Ltd., Netherton, West Midlands UK

An extensive program of work has been carried out to evaluate the efficiency of ceramic foam filters under carefully controlled conditions. This work, reported at previous TMS meetings, showed that in the absence of grain refiners, ceramic foam filters have the capacity for high filtration efficiency and consistent, reliable performance. The current phase of the investigation focuses on the impact grain refiner additions have on filter performance. The high filtration efficiencies obtained using 50 or 80ppi CFF's in the absence of grain refiners, diminish when Al-3%Ti-1%B grain refiners are added. This, together with the impact of incoming inclusion loading on filter performance and the level of grain refiner addition are considered in detail. The new generation Al-3%Ti-0. 15%C grain refiner has also been included. At typical addition levels (1kg/tonne) the effect on filter efficiency is similar to that for titanium boride based grain refiners. The work was again conducted on a production scale using AA1050 alloy. Metal quality was determined using LiMCA and PoDFA. Spent filters were also analysed.

9:45 AM

Advanced Addition Practices with AlSr Rod for Accurate and Cost Effective Modification in the Casthouse and Aluminium Foundry: *Piet C. van Wiggen*[1]; Jaco K. Belgraver[2]; [1]KBM Affilips B. V., Res. & Dev., Waalkade 2, OSS, Noord Brabant 5340 AT, The Netherlands; [2]KBM Master Alloys B. V., Res. & Dev., Kloosterlaan 2, Delfzijl, Groningen 9930 AD, The Netherlands

For optimum mechanical properties, AlSi casting alloys often require microstructural modification by AlSr master alloys. These additions are usually carried out in the transport ladles or the furnace long before casting. Consequently, such procedures are relatively expensive due to the high strontium losses which occur throughout the process. Moreover, the consistency in the recovery of the Sr level in the as-cast products is limited. In this respect, the use of the fast dissolving AlSr rod in combination with the in-line and last-minute addition concepts, offers both an economical and technical perfect solution. The paper will describe casting tests which are illustrative for the fast dissolution and quick modification performance of the AlSr rod product. A number of addition methods will be outlined to explain how both concepts are currently applied in the casthouse and aluminium foundry.

10:10 AM Break

10:20 AM

Alloying Kinetics of Mn in Aluminum Melt: *Young E. Lee*[1]; Dana L. LeMasters[1]; [1]Eramet Marietta, Inc., Rt 7 South, Riverview Dr., Marietta, OH 45750 USA

The recovery performance of Mn and Al compacts is observed to be erratic when the Al content in the compacts is decreased. This report examines the causes for the observed erratic performance. When added cold in aluminum melt, the alloying process of Mn-Al compacts takes place by the sequence of incubation, dispersion, and homogenization. The incubation delays the onset of the recovery reaction. It is controlled initially by the convective heat transfer and later by the exothermic heat of reactions. After the incubation period, the Mn-Al compacts break down into small discrete units and are dispersed in aluminum bath. Once this happens, the kinetics of the alloying reactions is controlled by the heat and mass transfers. When it contains aluminum, the thermal conductivity of Mn-Al compact is high, and the recovery process commences quick. The effect of compact size is minimum. When it does not contain aluminum, the onset of the recovery reaction is delayed, and the time for full recovery becomes excessively long and is affected significantly by the compact size. One effective way to improve the dissolution rate of the Mn compacts without aluminum is to make the compact size as small as possible.

10:45 AM

Inclusion Removal Kinetics During Chlorine Fluxing of Molent Aluminium: *Torstein A. Utigard*[1]; Raja R. Roy[1]; Claude Dupuis[2]; [1]University of Toronto, Dept. of Metall. and Mats. Sci., 184 College St., Toronto, Ontario M5S 3E4 Canada; [2]Alcan International Limited, Arvida Res. and Dev. Centre, 1955 Mellon Blvd., P.O. Box 1250, Jonquière, Québec G7S 4K8 Canada

Understanding of inclusion removal kinetics is essential to achieve further reduction of chlorine utilization during fluxing of molten aluminum alloys in furnace. This paper presents experimental works carried out to study the mechanism of non-metallic inclusion as a

function of key parameters (fluxing gas flow rates, chlorine concentration and stirring energy) affecting inclusion separation. Inclusion removal takes place in two steps; initially there is an incubation period during which the inclusions are not removed followed by an exponential decay of inlcusion concentration with time. Based on thses observations, a regulated chlorine fluxing strategy that can lead to significant reduction of chlorine utilization while meeting the required metal cleanliness level is proposed.

11:10 AM

Flow Structure and Stability of the Deposition Layer in Deep-Bed Filters: *Laszlo Istvan Kiss*[1]; Rung Tien Bui[1]; Duygu Kocaefe[1]; Yannick Fortin[1]; Peter Waite[2]; [1]University of Quebec at Chicoutimi, Dept. of Appl. Sci., 555 boul. de l"Universite, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International, CRDA TMCR, 1955 Boul Mellon, Jonquiere, Quebec G7S 3H5 Canada

The flow structure and the formation of deposition layers during the deep-bed filtration of aluminium was studied experimentally in water models. Various two- and three-dimensional collector elements were arranged in the test section. The flow structure was analysed by visualisation techniques and also by quantitative methods like LDV and PIV. As self-generated oscillations in the flow can wash away already deposited particles, the limits of the transition flow regime were determined in function of the flow velocity and bed geometry. the evolution of the shape and thickness of the deposition layer was followed and recorded. The release and re-entrainment of the inclusions was observed during the starting and stopping of the flow, as well as under the effect of external mechanical vibrations. The role of the adhesion force between the inclusions and collector elements in comparison to the cohesion between the particles inside the deposition layer was also analysed.

Chemistry and Electrochemistry of Corrosion and Stress Corrosion: A Symposium Honoring the Contributions of R.W. Staehle: Pitting, Crevice Corrosion and Crack Initiation - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee, Jt. Nuclear Materials Committee

Program Organizer: Russell H. Jones, Battelle Pacific Northwest National Laboratory, Richland, WA 99352 USA

Wednesday AM Room: 222
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Peter M. Scott, Framatome, Paris 92084 France; Dan van Rooyen, Wading River, NY 11792 USA

8:30 AM

The Critical Size of Strain Induced Pits in the Initiation of Stress Corrosion Cracking of Low Alloy Steels: *Tomomi Murata*[1]; [1]Nippon Steel Corporation, 1-7-14 Katsuradai-Minami, Sakae-ku, Yokohama 247-0033 Japan

The case study of practical failures of low alloys steels primarily due stress corrosion cracking clearly indicates the presence of precursor for cracks to initiate. Statistical analysis of the size and shape of strain induced pits suggests that there is a critical size and shape of a pit above which it would provide the local condition for cracks to initiate. This paper deals with the assessment of a pit of critical size and shape distributed among numerous others. Accelerated tests for stress corrosion cracking will be reexamined.

9:00 AM

The Effects of Solution Chemistry on Passive Film Fracture and Stress Corrosion: *David F. Bahr*[1]; M. Pang[1]; D. Rodriguez-Marek[1]; C. Johnson[2]; [1]Washington State University, Mech. & Mats. Eng., P.O. Box 642920, Pullman, WA 99164-2920 USA; [2]Central Washington University, Mech. Eng. Techn., Ellensburg, WA 98926

Solution chemistry can be associated with some forms of stress corrosion cracking in alloys with passivating films, like stainless steel and titanium. However, there is little direct evidence regarding the role solution chemistry plays on the strength the passivating film. Recent developments in nanomechanical testing using indentation techniques and scanning probe microscopy allow the fracture of passivating films to be measured under electrochemical control. The strength of these films has been measured using nanoindentation in 304 stainless steel under electrochemical polarization in aqueous solutions containing various halide concentrations. These experiments have been correlated to bulk slow strain rate and fracture toughness testing in similar environments while monitoring current transients from film fracture. The relationship between the chemistry of the environment and the mechanical properties on both the nano and bulk length scales will be discussed.

9:30 AM

Contribution of the Initiation Process to Stress Corrosion Cracking of Stainless Steel: *Toshio Shibata*[1]; Takumi Haruna[1]; Kozo Ohnishi[1]; [1]Osaka University, Dept.Mats. Sci. and Proc., Grad. Sch. of Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

The initiation of cracks in the stress corrosion cracking(SCC) failure process has to be evaluated quantitatively for making reliable assessment of the SCC susceptibility and analyzing mechanstic aspects of the SCC process. We have been concerned for years with characterizing the initiation process of SCC cracks which are successfully separated from the propagation by using an in-situ surface observation system consisting of a CCD camera and video recording apparatus. Environmentally sensitive and stochastic nature are very specific features of the crack initiation which will be discussed with examples for stainless steels in various environments.

10:00 AM

The Effect of Chloride and Thiosulfate on the Transient Instability of Alloys 600, 690 and 800: *Zhi Fang*[1]; Roger W. Staehle[2]; [1]Medtronic, Inc., Des. Reliability and Prod. Test., Medtronic Energy and Comp. Ctr., 6700 Shingle Creek Pkwy., Brooklyn Center, MN 55430 USA; [2]University of Minnesota, Dept. of Chem. Eng. and Mats. Sci., 22 Red Fox Rd., North Oaks, MN 55127 USA

The effect of chloride and thiosulfate on pitting and the transient instability of passive film of Alloys 600, 690 and 800 was studied at 95°C using the potentiodynamic polarization method. The pitting breakdown potentials of Alloys 600, 690 and 800 decrease linearly with logarithm of chloride concentration. The three alloys exhibit the highest breakdown potentials in chloride solutions at pH6 in the pH range of 3.5 through 8. The transient instability of protective films was assessed using stress corrosion cracking (SCC) parameter, that incorporates the ratio of current densities obtained in fast and slow potentiodynamic scans. The potentials at which SCC most likely occurred are strongly dependent on chloride concentration, very less dependent on pH of chloride solutions and are generally correlated with the breakdown potentials for Alloy 800, in the low anodic potential ranges for Alloys 600 and 690 in chloride solutions. Interaction of chloride and thiosulfate at total concentration of 10-2M significa ntly reduces breakdown potentials especially for Alloys 600 and 800 whereas at total concentration of 1.0M less effect on pitting resistance and a relative higher inhibitory effect on SCC susceptibility than at 0.01M concentration. Alloy 690 exhibits generally the highest pitting resistance and lowest SCC susceptibility in chloride and thiosulfate solutions in neutral pH region.

10:30 AM

Influence of Applied Potential on the Environmentally Assisted Cracking of Nanofilms of 304 Stainless Steel: R. A. Etien[1]; V. Radmilovic[1]; B. Tang[1]; T. M. Devine[1]; [1]University of California, Dept. of Matls., Berkeley, CA USA

Thin films of 304 stainless steel were prepared by pulse laser deposition (PLD) onto silicon substrates. Square and rectangular shaped holes were etched through the silicon, creating thin film membranes of stainless steel. Residual tensile stresses were intro-

duced into the stainless steel films by suitable heat treatments. The films exhibited body-centered cubic crystal structure and a grain size of 40 nm. The thin film samples with residual tensile stresses were immersed in 0.75M HCl and their times to failure at the corrosion potential were measured. Failures occurred in a matter of seconds. In contrast, no failures occurred when samples were cathodically polarized in the same solution. Such samples did fail very quickly when cathodic polarization was shut-off. Bulk samples of ferritic stainless steels are known to be susceptible to hydrogen embrittlement. The present results suggest that nanostructured films are more resistant to hydrogen assisted cracking than macroscopic sized samples.

11:00 AM

Predicting and Improving the Reliability of Electronic Equipment in Harsh Environments: R. B. Comizzoli[1]; C. A. Jankoski[1]; G. A. Peins[1]; L. A. Psota-Kelty[1]; D. J. Siconolfi[1]; *J. D. Sinclair*[4]; [1]Lucent Technologies, Bell Laboratories, Rm 1D-259, 600 Mountain Ave., Murray Hill, NJ 07974 USA

Environmental effects on electronic equipment are frequently quite difficult to predict and prevent. Environmental degradation is dominated by fine particles (0.1 to 2.5 mm) produced by fossil fuel combustion. They are rich in ammonium, sulfate, and nitrate. Because of their hygroscopicity, these particles can have dramatic effects on performance. The concentrations of substances in the environment that are hazardous to electronics vary dramatically around the planet. The total mass of suspended particulate and the concentration of sulfate found in some parts of Asia are more than ten times the concentrations found in some other regions. These wide variations challenge the ability of designers to meet reliability expectations. Research-based design strategies for predicting/mitigating the effects of pollutants on corrosion and other degradation processes are still in the early stages of development. Methods used by Bell Laboratories will be discussed.

11:30 AM

A Study on Corrosion Characteristics of Thermally Insulated Pipeline with Insulation Defects: *Jung-Gu Kim*[1]; *Yong-Wook Kim*[1]; [1]Sungkyunkwan University, Metall. Eng. Dept., 300, Chunchun-Dong, Jangan-Gu, Suwon, Kyunggi-Do 82 South Korea

The thermally insulated underground pipelines have been used for district heating systems. The insulated pipelines can be exposed to the danger of corrosion if the insulation becomes wet. The corrosion properties of thermally insulated pipeline were investigated in synthetic ground water by polarization test, galvanic corrosion test and SEM analysis. In polarization tests, the corrosion of thermally insulated pipeline was active and uninsulated surfaces were more active than insulated surfaces in tested temperatures. Insulation is a barrier to prevent dissolution of corrosion product. XRD analysis (Cu K¥á radiation) revealed that corrosion products were ¥á-FeOOH, ¥ã-FeOOH, Fe3O4, CaCO3. When the pipeline was exposed to corrosive environments for a long time, the corrosion rate gradually increased due to increasing moisture content and concentration. Corrosion was accelerated with insulation defects; holiday and disbonding.

Computational Thermodynamics and Materials Design: Phase Equilibria and Phase Transformation II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Jt. Computational Materials Science & Engineering, Thermodynamics & Phase Equilibria Committee

Program Organizers: Zi-Kui Liu, Penn State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; Ibrahim Ansara, LTPCM-Enseeg, Grenoble, France; Alan Dinsdale, National Physical Laboratory, UK; Mats Hillert, Royal Institute of Technology, Materials Science & Engineering, Stockholm 10044 Sweden; Gerhard Inden, Max-Planck Institute-Duesseldorf, Düsseldorf D-40074 Germany; Taiji Nishizawa, Tohoku University, Japan; Greg Olson, Northwestern University, Department MSE, 2225 N. Campus Drive, Evanston, IL 60208 USA; Gary Shiflet, University of Virginia, Department of Materials Science & Engineering, Charlottesville, VA 22903 USA; John Vitek, Oak Ridge National Laboratory, Oak Ridge, TN USA

Wednesday AM Room: 201
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Ibrahim Ansara, Institute National Polytechnique de Grenoble, Grenoble, France

8:30 AM

Ostwald's Rule and the Development of a Dynamical Metastable Relation in Gold-75 Nickel: *J. S. Kirkaldy*[1]; G. S. Weatherly[1]; K. Janghorbian[1]; [1]McMaster University, Brockhouse Inst. for Mals. Res., Hamilton L8S-4M1 Canada

The Au-Ni alloy possesses a high Tc miscibility gap which is associated with the Au Goldschmidt radius of 1.15 that for Ni. This has the further consequence that coherency strain enters strongly into the free energy sequence of the associated transformations. Ostwald's Rule, which is a corollary of the Onsager-Prigogine principle of minimum dissipation, states that where distinct patterning sequences are feasible nature chooses a sequence passing through one or more high free energy states rather than a direct decay to the ground state. Where the isothermal decay of the Helmholz F is continuous this imperative is expressible as solutions of Ginzburg-Landau differential equations. We have recorded such a sequence in the pre-quenched 75 at% Ni alloy on the TEM hot stage at 500ºC starting with one, two and three-dimensional ~10nm modulations with strain-relaxing L1o ordering at the nodes. This structure acts as the nucleus for near single crystal coarsening to a lamellar product (~100nm) which consists of alloy partitioning to 50:50 L1o and nearly pure nickel. We conclude that this state, like solid state amorphization in Co-Nb, is dynamical rather than a metastable coherent equilibrium as argued by Allen and Cahn for tricritical reactions in 23 Fe-Al.

8:55 AM

Component Activity Measurements in the Ti-Al-O System by Knudsen Cell Mass Spectrometry: *Evan Copland*[1]; Nathan Jacobson[1]; [1]NASA Glenn Research Center, 21000 Brookpark Rd., Mail Stop 106-1, Cleveland, OH 44135 USA

Currently there is great interest in gamma-TiAl based titanium aluminide materials (containing alpha subscript 2 Ti_3 Al and gamma-TiAl) for structural applications at intermediate to high temperatures. However above ~850ºC they suffer from poor oxidation resistance, characterized by the formation of a non-protective TiO_2+Al_2O_3 scale. Their inability to form a protective Al_2O_3 rich scale is interesting as experimentally determined isothermal sections of the Ti-Al-O system show gamma-TiAl+alpha subscript 2-Ti_3 Al structures are only in equilibrium with Al_2O_3. To better understand this non-protective scaling behavior, experimental measurements of thermodynamic activities and interactions between aluminum, oxygen and titanium are needed in the alpha subscript 2-Ti_3 Al phase. The

progress in measuring component activities as a function of composition by the Knudsen cell mass spectrometric method is reported here. The major experimental issue with Knudsen cell mass spectrometry is the conversion of measured ion intensities to vapor pressures and/or activities. An elegant solution to this problem is the use of an internal standard with a multiple Knudsen cell configuration (1,2). It would be ideal to use the pure component as the standard, however this is not always possible due to mixing of the molecular beams emerging from the two cells. Therefore copper–in a ZrO_2 cell–was used as an in situ standard for aluminum and oxygen, while nickel–in a ZrO_2 cell–was used for the titanium activity measurement. The measured in situ standard intensities were related to the pure component ion intensity by measuring the ionization cross-section ratios ($sigma_{Cu}/sigma_{Al}$, $sigma_{Ni}/sigma_{Ti}$, and sigma subscript Cu/$sigma_{Al2O}$) in separate calibration runs. This method removes the need to determine absolut e values of the ionization cross sections (2). The ionization cross-section ratios were determined by comparing the measured vapor pressures to the tabulated vapor pressures (3) of pure copper, nickel, luminum, titanium and Al_2 O(g). The component activities for aluminum and titanium were calculated from the ratio of the measured component intensity over the alloy and the in situ standard in addition to the measured ionization cross-section ratios. The $P(O_2)$ was determined from the measured intensities of Al(g) and Al_2 O(g) by a third law calculation with the tabulated K values for the 2Al+_O_2 = Al_2 O(g) (3,4). Values of titanium, aluminum, and oxygen activities are reported for selected alloys in and around the alpha subscript 2-Ti_3 Al phase. These data are compared to the recent assessment of the Ti-Al-O system by Lee and Saunders (5).

9:20 AM

Direct Evidence of the Magnetically Induced Phase Separation in the FCC Phase and Thermodynamic Calculations of Phase Equilibria of the Co-Cr System: *Katsunari Oikawa*[1]; Gao-Wu Qin[1]; Tamio Ikeshoji[1]; Ryusuke Kainuma[2]; Kiyohito Ishida[2]; [1]Tohoku National Industrial Research Institute, Mats. Eng., 4-2-1 Nigatake, Miyagino-ku, Sendai, Miyagi 983-8551 Japan; [2]Tohoku University, Mats. Sci., Aramaki Aobayama 02, Aoba-ku, Sendai, Miyagi 980-8759 Japan

Two-phase equilibria between the ferromagnetic fcc and the paramagnetic fcc phase from 800°C to 900°C in the Co-rich region was detected by the diffusion couple technique. Two phase separation region of the fcc was confirmed along the Curie temperature. The phase equilibria including the present results and the thermodynamic data of the Co-Cr system reported in the literature were analyzed on the basis of the thermodynamic evaluation. A set of thermodynamic values for the liquid, fcc, hcp, bcc and sigma phases was obtained. The calculated phase equilibria was in good agreement with most of the experimental data.

9:45 AM

Precipitation Kinetics of Niobium Carbide and Copper in a Low Carbon, Chromium Free Steel: *Michael S. Gagliano*[1]; Morris E. Fine[1]; [1]Northwestern University, Mats. Sci. and Eng. Dept., 2225 N. Campus Dr., Evanston, IL 60208 USA

Co-precipitation of niobium carbide and bcc copper is being investigated as a basis for making a high strength, easily weldable, low carbon, chromium-free structural steel as an alternative to tempered martensite. For a steel containing 0.0589 C, 1.37 Cu, 0.82 Ni, 0.49 Mn, 0.491 Si, 0.079 Nb, 0.034 Al (wt.%) theoretical curves were calculated for the nucleation and growth kinetics of NbC and Cu precipitates in ferrite as a function of time and temperature for a reaustenization temperature of 1000°C. All curves showed the typical inverse "C" type behavior with the maximum nucleation rates for both NbC and Cu occurring at about 600°C, however Cu nucleation was orders of magnitude faster than NbC. The maximum growth rate for the bcc Cu clusters was found to occur at 690°C while the growth rate for NbC in ferrite was a maximum at the transformation temperature from austenite to ferrite. Aging treatments selected on the basis of the theoretical curves and are in approximate agreement with them.

10:10 AM Break

10:20 AM

A Study of Phase Diagram in the Na2O2-NaOH System: *Junichi Saito*[1]; Kazumi Aoto[1]; [1]Japan Nuclear Cycle Development Institute (JNC), Struct. Safety Eng Grp., 4002 Narita, Oarai-machi, Higashi-Ibaraki, Ibaraki 311-1393 Japan

Sodium compounds, Na2O, Na2O2 and NaOH are formed by combustion of sodium in air. These compounds have strong reaction with structural materials. In particular, it is found that liquid Na2O2 corrodes significantly them. Corrosion rate varied largely depending on its temperature, component of mixture and present state (solid and/or liquid). Therefore it is very important to be clear the present state of mixture at certain temperature. Phase diagrams are utilized generally for understanding present state of material. However, there is no phase diagram in the Na2O2-NaOH system. In this study, thermodynamic measurements were carried out using various mixture of compounds in order to obtain the basic data for phase diagram. Based on these experimental datum, phase diagram in the Na2O2-NaOH system was calculated using Thermo-Calc which was the one of calculation program. Consequently, phase diagram in the Na2O2-NaOH system was constructed using experimental datum and calculation.

10:45 AM

Analysis of Microsegregation and Thermo-Mechanical Properties of Carbon Steels during Continuous Casting: *Dong Jin Seol*[1]; Young Mok Won[2]; Kyu Hwan Oh[1]; [1]Seoul National University, Sch. of Mats. Sci. and Eng., San 56-1 Shinrim-dong, Kwanak-ku, Seoul 151-742 Korea; [2]University of Illinois at Urbana-Champaign, Dept. of Mech. and Indust. Eng., Urbana, IL 61801 USA

Reduction of cracking during continuous casting of carbon steels has been focused again according as the recent casting conditions have trended toward higher casting speed and thinner strand thickness. The internal and surface cracking of strand has been analyzed with the thermo-mechanical model in mushy zone and numerical microsegregation analysis taking the d/g transformation into account. In the microsegregation analysis, local equilibrium was assumed at the interface, and solute back diffusion was considered in solid phase whereas perfect mixing was assumed in liquid phase. The partition coefficient, which determines solute redistribution at the interface of two phases, was evaluated with TQ, an application program interface of the Thermo-Calc. Cracking frequency was compared with the accumulated thermal strain and the d/g transformation-induced strain in the brittle temperature range which was evaluated by the microsegregation analysis. The effect of alloy composition such as sulfur content on cracking was also studied.

11:10 AM

Comparative Thermodynamic Study of the Zn-Cd-Sb System: *D. Zivkovica*[1]; Z. Zivkovica[1]; A. Kostov[2]; B. Vucinica[1]; [1]University of Belgrade, Techn. Fac., VJ 12, 19210 Bor Yugoslavia; [2]Copper Institute, Zeleni Bulevar 35, 19210 Bor Yugoslavia

The results of comparative thermodynamic analysis in the investigated sections in Zn-Cd-Sb system, done by quantitative DTA, calorimetric measurements and thermodynamic predicting methods are presented in this paper. Partial and integral molar quantities were determined in the temperature interval 800-1000K. Also, phase diagrams of the chosen sections were constructed based on the results of DTA, X-ray analysis and optical microscopy for the investigated alloys.

11:35 AM

Critical Evaluation and Comparison of Thermodynamic Models for the Liquids Phases: *Florian Kongoli*[1]; Ian McBow[1]; [1]Flogen Technologies, Metall., P.O. Box. 49529, C.P. du Musee, Montreal, Quebec H3T 2A5 Canada

Several thermodynamic models have been used for modeling of various liquid phases in chemical or metallurgical systems. Each model is based on a particular formalism such as the sub-lattice, quasichemical and association formalisms or on simple polynomial expansions of the excess Gibbs energy. Due to the physical assumptions they employ as well as the mathematical formulations they

use, they all have several strong and week points. In this work these models are critically evaluated and compared in terms of their physical and chemical significance, their generality and simplicity and the number of mathematical parameters used.

Cyanide: Social, Industrial, and Economic Aspects: Cyanide Management II

Sponsored by: Extraction & Processing Division, Waste Treatment & Minimization Committee, Precious Metals Committee, International Precious Metals Institute, Society of Mining, Metallurgy and Exploration, Inc., Northwest Mining Association
Program Organizers: Courtney Young, Montana Tech, Metallurgical and Materials Engineering, Butte, MT 59701 USA; Corby Anderson, Montana Tech., CAMP and Metallurgical and Materials Engineering, Butte, MT 59701 USA; Larry Twidwell, Montana Tech, Metallurgical and Materials Engineering, Butte, MT 59701 USA

Wednesday AM Room: 225
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Dave Dreisinger, The University of British Columbia, Dept. of Mets. and Mats. Eng., Vancouver, B.C. V6T 1Z4 Canada; Tam Tran, The University of New South Wales, Sch. of Chem. Eng. and Indust. Chem., Sydney, Australia

8:30 AM Invited
Cyanide and the Environment: Barrick Gold Corporation's Perspective: *John Richard Goode*[1]; Jacques McMullen[1]; John Adrian Wells[1]; Kenneth Glynder Thomas[1]; [1]Barrick Gold Corporation, Royal Bank Plaza, South Tower, Suite 2700, 200 Bay St., Toronto, Ontario M5J 2J3 Canada

Barrick Gold Corporation operates gold mines in the United States, Chile, Peru, Canada, and in the near future, in Tanzania. Barrick meets environmental requirements and efficiently manages the cyanide constituents of its effluents using various cyanide destruction techniques. Company operations use, or have used, natural degradation, ferrous sulphate, Caro's acid, SO2/Air and SO2/O2 processes for cyanide destruction. This paper presents a general discussion of the options for controlling cyanide in plant effluent and provides details of Barrick's operating experience including general notes on costs.

8:55 AM Invited
The Case for Cyanide Recovery From Gold Plant Tailings-Positive Economics Plus Environmental Stewardship: *C. A. Fleming*[1]; [1]Lakefield Research Limited, Techn. & Bus. Dev., 185 Concession St., Lakefield, Ontario K0L 2H0 Canada

There are now several processes available for the recovery of free and WAD cyanide from gold plant solutions and pulps. In the past, the incentive for evaluating cyanide recovery in gold plant flowsheets has been economic. In many cases, it has been possible to demonstrate (at least in the laboratory and on paper), an improvement in process economics by: (1) reducing the costs of tailings detoxification, (2) recycling cyanide at lower cost than the purchase/delivery price of new cyanide, and (3) generating additional revenue via by-product sales. Despite these convincing arguments, few cyanide recovery plants have been built on operating gold mines. There is now arguably an even more compelling reason for mining companies to consider cyanide recycling. With the growing storm of negative public opinion that the use of cyanide in the mining industry is attracting–following several highly publicized spills over the last couple of years–the time has come for the gold industry to demonstrate environmental diligence and stewardship in the use of this commodity that is so vital for their industry. There is no doubt that the widespread implementation of cyanide recycling will reduce the impact of the cyanidation process on the environment, both by reducing the risk of spills (with less cyanide being transported from manufacturing plants to gold mines), and by reducing the loading of toxic and non-toxic metals and ions in the tailings. This paper discusses the various processes that are available for cyanidation, and presents the process chemistry and results of several laboratory and pilot plant trials that have been conducted over the last few years.

9:20 AM Invited
Use of Ion Exchange Resin for Cyanide Management during the Processing of Gold Ores: *Tam Tran*[1]; Ken Lee[1]; Kapila Fernando[1]; [1]The University of New South Wales, Sch. of Chem. Eng. and Indust. Chem., Sydney, NSW 2052 Australia

Most new gold mines in Australia now have to treat cyanide wastes before the tailings are discharged to storage ponds, following strict guidelines from the EPA. In New South Wales, the level of weak-acid dissociable cyanide allowed in tailing dams is now limited to 20 ppm. The paper deals with aspects of cyanide management during the processing of gold ores, particularly those containing high levels of cyanide-soluble minerals. Of interest to mining operations in Australia is the processing of copper-gold ores. Copper-gold ores have been technically difficult and uneconomical to process due to high consumptions of cyanide. Many copper oxide and sulphide minerals react with cyanide to form copper cyanide complexes, partly contributing to the high level of weak-acid dissociable cyanide in the tailings which is normally experienced during the processing of copper-gold ores. The cost for detoxification, if required for treating these tailings, would further inhibit the development of these copper-gold resource s unless the cyanide used for leaching was recovered. The waste processing operation is further complicated by the presence of thiocyanate (SCN) formed by the reaction of sulphide and cyanide. Although thiocyanate is not subjected to EPA's discharge regulations, its presence in cyanide wastes and tailings causes high consumption of detoxification reagents used for cyanide destruction. Several technologies have been proposed to process copper-gold ores, aiming to recover copper as a co-product and cyanide for recycling in conjunction with the leaching process. A critical review of these processes reveals that a pre-concentration stage (solvent extraction or resin ion exchange) is required. Cyanide is recovered by the conventional Acidification-Volatilisation-Regeneration technique in most cases. A critical evaluation is provided on technical aspects related to the use of ion exchange resin, particularly for cyanide management and for processing copper-gold ores. The experience of such an application at May Day Mine, NSW, Australia is to be discussed in detail.

9:50 AM Invited
The Role of Elution on the Recycling of Cyanide Using Ion Exchange Resins: Versiane Albis Leão[1]; *Virginia S.T. Ciminelli*[1]; [1]Universidade Federal de Minas Gerais, Dept. of Metall. and Matls. Eng., Rua Espírito Santo 35, Belo Horizonte, MG 30160.030 Brazil

This work is aimed at the selection of an appropriate eluant for cyanide recycling using ion exchange resins. Batch experiments were carried out with synthetic solutions containing high levels of copper in the presence of minor amounts of iron and nickel. The elution of the resins, Imac HP 555s, Amberlite IRA 900 (Rohm&Hass) and Purolite A860S (Purolite Co) was investigated using NaCl, zinc and nickel cyanides, as well as acid thiourea solutions. The results indicate that saline solutions are able to elute only the cyanocomplexes loaded on polyacrilic resins (Purolite A860S), failing to desorb the metals loaded on polystyrene based resins, especially the nickel cyanocomplex. Cyanide solutions of both zinc and nickel are able to elute over 90% of loaded metals. The same figures are observed with thiourea solutions. The resin Imac HP555s is recommend for base metals uptake while thiourea is suggested for the elution of the former resin. Using this system it is possible to release the overall copper and nickel b ounded cyanide since the chosen resin does not load significant amounts of iron in this particular system. The technological implications of the studied elution procedures are discussed.

10:15 AM Break

10:30 AM Invited
Copper-Gold Cyanide Recovery System: *William H. Jay*[1]; [1]Ortek Ltd., P.O. Box 16, South Perth, Western Australia 6151, Australia

Environmental and financial benefits for mines treating copper-gold ores is enhanced by recent laboratory and pilot plant studies.

The coordination of copper to a water-soluble and/or an organic soluble polychelating agent under alkaline conditions results in the displacement of the cyanide ion from the copper atom. The membrane separation of the polychelated copper enables the cyanide ion to be recycled and the copper is then directly electrowon from the polymer. The polychelating polymer is recycled. The entire process is conducted without the need for pH adjustment to the leach slurry.

11:00 AM Invited

HW Process Technologies, Incorporated's Engineered Membrane Separation (EMS™) of Copper and Gold in Cyanide Solutions: *John A. Lombardi*[1]; Ron Bernard[1]; [1]HW Process Technologies, Inc., 1208 Quail St., Lakewood, CO 80215 USA

HW Process Technologies, Inc. (HWPT) has developed a membrane-based ion fractionation system which specifically separates copper from gold and silver species in a cyanide matrix. This Engineered Membrane Separation (EMS™) system was pilot demonstrated on the 82,000 liters of pregnant leach solution (PLS) generated from thin layer leaching of tailings at a mine site in Durango State, Mexico. The EMS™ recovered ~90% of the gold-silver cyano-complex to a 90% volume permeate (based upon an EMS™ feed stream of 100% volume). The EMS™ rejected ~90% of the copper cyano-complex to a 10% (by volume) concentrate.

11:25 AM Invited

Solvent Extraction Recovery of Copper Cyanide from Spent Gold Mill Effluents: *David Dreisinger*[1]; Berend Wassink[1]; [1]The University of British Columbia, Dept. of Mets. and Mats. Eng., 309-6350 Stores Rd., Vancouver, B.C. V6T 1Z4 Canada

The cyanidation of copper containing gold ores results in the co-dissolution of copper along with gold. Copper dissolution results in elevated cyanide consumption that may make the treatment of the gold ore uneconomic. Copper-cyanide species in gold mill effluents may increase the environmental toxicity of the gold mill solutions. The objective of this study was to examine the use of solvent extraction to recover the copper cyanide species from gold mill effluents. Solvent extraction with Henkel (Cognis) reagents LIX 79, LIX 7950 and XI 78 was studied. Excellent extraction of copper and cyanide was achieved using pH control in extraction. Copper and cyanide may be stripped from the reagents using basic eluants. Various routes for copper and cyanide recovery from the strip solution were studied. Excellent results were obtained using electrowinning in both divided (Nafion membrane) and undivided cells. Cyanide recovery was obtained by AVR treatment of a bleed stream from electrowinning. Full results of solvent extraction, copper and cyanide recovery are reported.

Defect Properties and Mechanical Behavior of HCP Metals and Alloys: Phase Transformation and Microstructure

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Chemistry & Physics of Materials Committee, Jt. Nuclear Materials Committee, Titanium Committee

Program Organizers: Man H. Yoo, Oak Ridge National Laboratory, Metals & Ceramic Division, Oak Ridge, TN 37831-6115 USA; James R. Morris, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Carlos N. Tome, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Wednesday AM Room: 211
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Michael J. Mills, Ohio State University, Mats. Sci. and Eng., Columbus, OH 43210-1178 USA; David J. Bacon, University of Liverpool, Dept. of Eng., Liverpool L69 3GH UK

8:30 AM Invited

Deformation Behavior of HCP Alpha Ti and Ti-Al Alloys: *Jim Williams*[1]; Roy Baggerly[2]; Neil Paton[3]; [1]The Ohio State University, Dept. of Mats. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Metallurgical Consultant, Anacortes, WA USA; [3]Howmet Research Corporation, Whitehall, MI USA

Alpha Ti has a c/a ratio <1.633 and deforms by both slip and twinning. Unlike Zn and Cd, prism and pyramidal slip with an a vector are commonly observed in addition to basal slip. When c-axis strains are imposed, twinning and/or c+a slip are observed. Al is soluble in alpha Ti as a substitutional solute and solid solution strengthens it. Al additions also alter the deformation behavior and, at concentrations 6.6 wt. %, change the a slip character from wavy to planar while also increasing the relative amount of c+a slip and decreasing the contribution of twinning to the overall deformation. Oxygen also is ubiquitous in Ti as an interstitial impurity. There is an interaction between oxygen and Al which influences the tendency for planar slip. We have studied both polycrystals and single crystals of alpha Ti and Ti-Al alloys containing up to 6.6 wt. % Al. This talk will describe the effects of orientation, deformation temperature and Al content on the resolved shear stress and the deformation modes and slip character of Ti and Ti-Al alloys. We also will attempt to relate the observations on single and poly crystals. This work was originally supported by AFOSR.

9:00 AM

Advances in Characterizing Twin Morphology using Automated Electron Backscatter Pattern Analysis: *George C. Kaschner*[1]; John F. Bingert[2]; Thomas A. Mason[1]; [1]Los Alamos National Laboratory, Mats, Sci, Techn., MST-8 M/S G755, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Mats. Sci. Techn., MST-6, M/S G770, Los Alamos, NM 87545 USA

It has been shown that the deformation mechanisms in high-purity zirconium are extremely temperature and strain-rate dependent. In an effort to understand the amount of deformation accommodated through twinning current research has focused on characterizing the twin volume in zirconium polycrystals strained under a variety of test conditions. The quantification of the thickness and spacing of twins within the grains in a microstructure typically involves repetitively grinding, polishing, and photographing a sequence of layers in a single sample. The features in these layers must then be analyzed either by hand (eye) or via some automated image analysis which depends solely on morphological measures of the microstructure. Additionally, the standard assumptions employed in the stereological analysis of each section, such as random feature orientation, are most likely violated when highly-textured hexagonal materials are examined. We hope to overcome some of these difficulties by using the information present within a limited number of OIM scans of a polycrystalline section to determine twin thickness and spacing (and hence, twin volume). This analysis is based on the mathematical description of the lattice orientation at each raster point of an automated electron diffraction data set. This type of information will help determine a statistically relevant relationship between twin morphology, crystallographic texture and test parameters. We also hope to reveal the causality of twins in neighboring grains which appear to percolate through the microstructures of high purity zirconium specimens when they loaded under appropriate conditions. Estimates of the errors involved in applying standard stereological analysis in this case will also be presented.

9:20 AM

Insights into Zirconium Deformation Behavior from EBSD Measurements: *John F. Bingert*[1]; Thomas A. Mason[2]; George C. Kaschner[2]; Paul J. Maudlin[3]; George T. Gray[2]; [1]Los Alamos National Laboratory, MST-6, MS G770, Los Alamos, NM 87025 USA; [2]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, T-3, MS B216, Los Alamos, NM 87545 USA

The deformation of crystal-bar zirconium was investigated as a function of strain and strain rate through electron back-scattered diffraction (EBSD) characterization. The resultant data provided spatially resolved information on microstructure and texture evolution, individual twin system activity, and subsequent strain partitioning between twinned volume and parent grains. A range of deformation conditions was represented through quasi-static com-

pression, 3-point beam bend tests at room and cryogenic temperature, and Taylor cylinder impact experiments. Effects from the interplay between slip and twinning deformation modes on anisotropic plasticity are considered in order to address the apparent trend toward isotropy at high rates. The role of various length scales on deformation behavior will be considered, along with the implications of these length scales on the assumptions typically invoked for plasticity modeling.

9:40 AM

Hydride Formation in the Alpha Phase of Zr Rich Alloys: *G. K. Dey*[1]; R. N. Singh[1]; D. Srivastava[1]; S. Banerjee[1]; [1]Bhabha Atomic Research Center, Matls. Sci. Div., Trombay, Mumbai 400 085 India

The formation of the γ and δ hydride phases has been studied in pure zirconium as well as in dilute zirconium niobium alloys. These studies have involved the examination of the hydride formation in hydrogen charged specimens including those subjected to hydride formation under thermal gradients and stress gradients. The morphology and the crystallography of the hydride phases have been studied and the observations rationalized in terms of phenomenological theory of martensite crystallography. The mechanism of clustering of hydride plates in cold spots created by sharp thermal gradients has been examined. Reorientation of hydride plates has been reported earlier under stress gradients. TEM investigations of the reorientation process have shown how preferential dissolution of hydride plates, unfavorably oriented with respect to the tensile axis followed by their reprecipitation in a different cluster configuration can cause this phenomenon.

10:00 AM Break

10:20 AM Invited

The Beta to Alpha Phase Transformation and Microstructure Evolution in Ti-(25,35)Al Alloys: Uwe Pilchowski[2]; Veer Dhandapani[3]; Keith J. Leonard[1]; *Vijay K. Vasudevan*[1]; [1]University of Cincinnati, Mats. Sci. and Eng., 501D ERC, P.O. Box 210012, Cincinnati, OH 45221-0012 USA; [2]Daimler-Chrysler, Airbus, Bremen, Germany; [3]VLSI Technology, 9651 Westover Hills Blvd., San Antonio, TX 78251 USA

The kinetics of transformation of the high-temperature b.c.c. beta phase to the low-temperature h.c.p. alpha phase and microstructure evolution during continuous cooling in Ti-25Al and Ti-35Al alloys was studied using a computer-controlled temperature and electrical resistivity measurement system. Samples of the alloys were heated to the beta phase region in the device, as well as separately in a furnace, and cooled at various rates either by controlling the flow of a helium jet quench in the former or the quenching media (furnace, air, oil, water, etc.) in the latter. Using the in situ resistivity and thermal arrest data, the start and finish temperatures of the transformation were determined as a function of cooling rate and correlated with post-mortem light and transmission electron microscopy observations of microstructure to establish CCT diagrams. By coupling the data with physical models, the enthalpies/driving forces associated with the transformation were determined. A transition in reaction mode fr om diffusional to diffusionless was observed with an increase in cooling rate, this change being accompanied by a change in the alpha (alpha2) morphology from Widman-statten to massive-like to martensitic structures at low, intermediate and high cooling rates. TEM analysis revealed that the martensite substructure was composed of twinned primary plates with microtwins within them. The twin system in both was determined to be (-2201)[1-104] relative to the alpha2 phase or (-1101)[1-102] relative to the alpha phase, which is the same as that observed in Titanium martensites. The same transformation was also studied in a Nb-36Ti-40Al alloy, in which it was possible to retain the high-temperature beta phase and hence determine orientation relations and habit planes of the martensite plates with respect to the parent phase. These results are presented and discussed.

10:50 AM

Formation of Face-Centered-Cubic Phase in Bulk Nanostructured Ttitanium Prepared by Severe Plastic Deformation: *Jianyu Huang*[1]; Honggang Jiang[1]; Yuntian T. Zhu[1]; Terry C. Lowe[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech. Div., MS G755, Los Alamos, NM 87545 USA

The equilibrium phases of titanium are hexagonal-closed-packed (hcp) under 1155K and body-center-cubic (bcc) above this temperature. However, face-centered-cubic (fcc) Ti has been reported in mutilayed thin film when the bilayer thickness decreased to a critical value. Others claimed that the fcc phase is not present in the original as-grown thin film but appears only after ion beam milling involved in the transmission electron microscopy (TEM) sample preparation process. In this paper, we report for the first time the formation of a similar fcc phase in bulk nanostructured titanium prepared by severe plastic deformation (SPD). The existence of the fcc phase was confirmed by both large-angle tilting selected area electron diffraction (SAED) and high resolution transmission electron microscopy (HRTEM). The lattice parameter of this fcc phase is about 4.44 to 4.54 Å, as estimated from the SAED patterns. The defect structures observed in fcc Ti, such as (111) twins, 60° and 90° dislocations, are typical of those in fcc materials. HRTEM revealed definite orientation relationships (ORs) between the fcc and hcp phases, which in most cases are $[110]_F//[2\ 110]_H$ and in rare case are $[110]_F//[2\ 110]_H$ and $(002)_F//(0002)H$, where F and H represent fcc and hcp phases, respectively. The interface between the fcc and hcp phases is sharp and the habit plane is inclined to the basal plane of the hcp phase. The definite ORs infers that the phase transformation from hcp to fcc is martensitic in nature. The finding of fcc Ti in the bulk materials is scientifically significant, since it show the prospects of producing single fcc phase in bulk form that is not predicted in the equilibrium phase diagram. Also fcc materials are generally more ductile than hcp, and the existence of a fcc phase may thus alter the mechanical properties of Ti related materials.

11:10 AM

Fcc->HCP Phase Transformation In Co-base Alloys: *Armando J. Saldivar*[1]; Hugo F. Lopez[1]; [1]University of Wisconsin-Milwaukee, Dept. of Mats., 3200 N. Cramer St., EMS Bldg., College of Eng., Milwaukee, WI 53201 USA

In biomedical applications, the tribological behavior of Co-alloys is strongly dependant upon the microstructural features of these materials. The Co-Cr-Mo-C alloys exhibit two allotropic forms, the HCP phase, which is thermodynamically stable at room temperature, and the FCC phase, which becomes stable at elevated temperatures. Based on the fact that the HCP crystal structure gives rise to relatively low friction coefficients and reduced wear rates compared with the FCC structure, the isothermal FCC-HCP martensitic transformation in cast and wrought Co-base alloys was investigated using optical and electron microscopy. The microstructural evolution during the isothermal treatment and the most remarkable kinetic and physical characteristics of this transformation are explained and discussed in detail.

11:30 AM

Monte-Carlo Simulation of Grain Boundary Character Distribution of HCP Materials: *Sun-Keun Hwang*[1]; Hoi-Soo Ryoo[1]; [1]Inha University, Dept. of Metall. Eng., #253, Younghyun-Dong, Nam-Gu, Incheon 402-751 South Korea

A Monte-Carlo simulation method was developed to study the impact of Grain Boundary Character Distribution (GBCD) on grain growth and texture evolution in HCP materials. Simulated microstructures were constructed in which the texture of real material was closely re-generated. The method of microstructure generation consisted of extracting the volume fraction and the Gaussian half-width from the experimental ODF and discretizing them into the simulated microstructure. For the range of c/a=1.55~1.633, GBCD of the CSL boundaries of up to Σwas computed from randomly oriented microstructure. Distribution of the misorientation angles and the CSL boundaries agreed well with theoretical predictions in the literature. The result was checked with the experimental ODF of pure Ti, from which a conclusion was drawn in that the distribution frequency of Σ1 boundaries was high in the microstructure containing coarse grains of main texture components due to clustering. The orientation discretizing method was also applied to the case of Zr that has undergone equal-channel angular pressing (ECAP). The result of the analysis was consistent with the case of the real material, thus

implying the potential effectiveness of the present methodology in other systems.

11:50 AM

Distribution of c- and a-Dislocations in Tubes of Zr-Alloys: *Margarita Isaenkova*[1]; Yuriy Perlovich[1]; [1]Moscow State Engineering Physics Institute (Technical University), Met. Sci. Dept., Kashirskoe shosse 31, Moscow 115409 Russia

A new X-ray method was developed to determine the density of dislocations with different Burgers vectors in alpha-Zr grains of any orientations, presented in the texture of channel and shell tubes of Zr-alloys. The method uses Fourier analysis of X-ray line profiles, registered by successive positions of the sample in the course of texture measurements. Calculated values of the dislocation density are plotted in texture pole figures, so that the condition of the crystalline lattice in grains of the tube proves to be connected with distributions of their basal and prismatic axes. Within the same tube the dislocation density varies, at least, by several times depending on the grain orientation. Volume fractions of grains with different values of c- and a-dislocation density are presented in diagrams, showing an actual substructure inhomogeneity of the tube. Features of these diagrams reflect the spectrum of mechanisms, participating in processes of structure formation. Obtained data testify that estimation of the dislocation density in tubes of Zr-alloys by standard methods results in accidental values and do not characterize the real condition of tubes, which can be adequately described only by taking into account their regular substructure inhomogeneity.

General Abstract Sessions: Solidification Processing

Sponsored by: TMS
Program Organizers: Thomas P. Battle, E. I. DuPont de Nemours & Company, Inc., White Pigments and Mineral Products, Edgemoor, DE 19809 USA; TMS, Warrendale, PA 15086 USA

Wednesday AM Room: 212
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Ralph E. Napolitano, Iowa State University, Mat. Sci. and Eng., Ames, IA 50011 USA; George Spanos, Naval Research Laboratory, Phys. Metall. Brch., Code 6320, 4555 Overlook Ave., S.W., Washington, DC 20375-5000 USA

8:30 AM

The Solidification Velocity of Nickel-Based and Titanium-based Alloys: *Alex S. Altgilbers*[1]; William H. Hofmeister[2]; Robert J. Bayuzick[2]; [1]Federal-Mogul Lighting Products, Prod. Eng., P.O. Box 87, 325 Sewell Dr., Sparta, TN 38583 USA; [2]Vanderbilt University, Chem. Eng., 24th Ave. S. & Garland, 106 Olin Hall, Nashville, TN 37212 USA

The solidification velocity has been measured as a function of undercooling for the Ni-Ti, Ni-Si, Ni-Sn, Ti-Ni, and Ti-Al systems. These alloy systems were selected to cover a wide range of partition coefficients and solute diffusion coefficients. This data has been used to obtain a better understanding of the solidification kinetics of dilute alloy systems. The measurements revealed a plateau in solidification velocity that occurs at intermediate undercoolings, which is separate and distinct from the plateau that has been previously observed at high undercoolings. The analysis performed in this investigation determined that the intermediate plateau is a result of the solute additions. The results were used to develop an addition to the theory developed by Boettinger, Coriel, and Trivedi(1) to more accurately predict the solidification velocity as a function of undercooling. In particular, a function is introduced that provides a bridge between the BCT pure material calculation and the BCT alloy calculation. (1)W.J. Boettinger, S.R. Coriell, and R.Trivedi, "Application of Dendrite Growth Theory to the Interpretation of Rapid Solidification Microstructures": in Rapid Solidification Processing: Principles and Technologies, IV, R.Mehrabian and P.A.Parrish, eds., Claitor's Publishing Division, Baton Rouge, LA, 1988, pp.13-25.

8:55 AM

Effects of Electromagnetic Vibrations on the Microstructure of Hypoeutectic Gray Iron: *Alireza Radjai*[1]; Kenji Miwa[2]; Kazuo Yasue[2]; Sung-Chul Lim[2]; [1]Japan Science and Technology Corporation, Nat. Indust. Rsch. Inst. of Nagoya, Mats. Proc. Dept., 1-1 Hirate-Cho Kita-ku, Nagoya, Aichi 462 Japan; [2]National Industrial Rsch. Inst. of Nagoya, Mats. Proc. Dept., 1-1 Hirate-cho, Kita-ku, Nagoya, Aichi, Japan

Electromagnetic vibrations induced in a molten metal by simultaneous application of alternating electric and stationary magnetic fields may lead to the formation and collapse of cavities and therefore refine the solidification structure. Previous investigations conducted on Al-Si alloys have shown that the two parameters of frequency and intensity of vibrations have crucial roles on the extent of structural refinement brought about by cavitation phenomenon. In the present research it has been tried to study the effects of these two parameters on the structural refinement of hypoeutectic gray iron. Employing an apparatus based on a super-conducting magnet, enabling a frequency of up to 50 kHz and a magnetic field of up to 10 T, a thorough investigation has been conducted over wide ranges of the two parameters. The results have shown similar trends in this case too, showing extensive structural refinement in specific ranges of the frequency and intensity of vibrations.

9:20 AM

Nucleation Kinetics Analysis by Repeated Solidification: *J. L. Sebright*[1]; G. Wilde[2]; J. H. Perepezko[1]; [1]University of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., 1500 Engineering Dr., Madison, WI 53706 USA; [2]Institut für Nanotechnologie, Forschungszentrum Karlsruhe, 76021 Karlsruhe, Germany

The direct assessment of a large number of nucleation events for a droplet provides information about the specific heterogeneous nucleation kinetics by avoiding the convolution of the undercooling response by numerous droplets of dissimilar nucleation characteristics. In the present work, over 800 independent nucleation events were recorded on a single 6N Au droplet that was encased in pyrex. The repeated undercooling measurements are evaluated in terms of a statistical distribution function that can be directly related to the characteristics of the nucleant. The nucleant site density and contact angle calculated from the distribution function are in agreement with the same parameters calculated from continuous cooling experiments performed on the same sample at different cooling rates. The addition of 500 ppm oxygen to the sample atmosphere caused a reversible 90K undercooling reduction from an average of 210K. The support by the NSF (DMR-97-12523) is gratefully acknowledged.

9:45 AM

Microstructural Evolution in Aluminum (Al-6061) Laser Welds: *George Spanos*[1]; Deug W. Moon[1]; C. R. Feng[1]; [1]Naval Research Laboratory, Phys. Metall. Brch., Code 6320, 4555 Overlook Ave., S.W., Washington, DC 20375-5000 USA

Microstructural, compositional, and microhardness variations throughout partial and full penetration laser welds of an aluminum alloy (Al-6061) were investigated and correlated to results from Professor Mazumder's group at The University of Michigan (UM) on real-time monitoring of weld pool dynamics. Features such as weld pool morphology, chevrons on the top surfaces of the welds, and bands within the welds are correlated with: (1) the macrostructure, as determined by optical microscopy, (2) the microstructure, as determined by transmission electron microscopy (TEM), (3) compositional variations, as determined by energy dispersive spectroscopy, and (4) color microhardness maps. Results on the three dimensional nature of and compositional variations within discrete growth bands, changes in microstructure at the TEM level of resolution, and variations in microhardness are all discussed in light of earlier work at UM that suggested frequent growth arrest and re-melting caused by fluctuations in the flow field, which may be related to keyhole dynamics.

10:10 AM Break

10:20 AM
Carbon Additions and Phase Stability in High Refractory Content Single Crystal Nickel-Base Superalloys: *Sammy Tin*[1]; Tresa M. Pollock[1]; [1]University of Michigan, Mats. Sci. and Eng., 2300 Hayward St., Ann Arbor, MI 48109 USA

Recent advances in alloy design have led to the development of multicomponent single crystal nickel-base superalloys which are being implemented in advanced turbine engines where service temperatures can range from T/TM= ~0.8-0.9. To delay incipient melting and improve high-temperature creep properties, the levels of Re, W and Ta additions have gradually increased in these alloys. With increasing levels of refractory alloying additions, phase stability in these alloys becomes a major concern due to limited knowledge of solubility limits and precipitation kinetics in these complex multicomponent systems. Due to the strong segregation characteristics of Re and W, the composition of the alloy must be carefully balanced to prevent the precipitation of detrimental secondary phases, such as TCP and SRZ, during solution heat treatments or service conditions. Recently, carbon additions (up to 0.1wt. %) have been shown to affect the segregation behavior of the constituent elements during solidification. Extended heat treatments of a large set of as-cast experimental single crystal superalloys have shown that the change in the partitioning behavior of the refractory alloying elements inhibits the precipitation of TCP and SRZ. The results of these experiments will be discussed with respect to the possible mechanisms by which carbon influences phase stability.

10:45 AM
Effects of Residual Oxygen on Nucleation of Zirconium: *Melissa J. Wert*[1]; William H. Hofmeister[1]; Robert Bayuzick[1]; [1]Vanderbilt University, Mats. Sci. & Eng., 2400 Highland Ave., 412A Olin Hall, Nashville, TN 37235 USA

In the present work, distributions of undercoolings for Zirconium samples of increasing Oxygen content were obtained using the electrostatic levitator (ESL) at NASA-MSFC. Over 100 undercooling/recalescence cycles were obtained for each specimen, allowing statistical analysis of the results using the Skripov approach as modified by Hofmeister et al to determine nucleation rate kinetic parameters. Histograms were defined for each sample and kinetic parameters were calculated using a form of the classical nucleation rate equation. The effects of residual Oxygen on nucleation of Zirconium using statistical analysis techniques of undercooling experiments will be presented.

11:10 AM
Anisotropy of Crystal-Melt Interfacial Energy: *Ralph E. Napolitano*[1]; [1]Iowa State University, Mat. Sci. and Eng., 104 Wilhelm, Ames, IA 50011 USA

The anisotropy of solid-liquid interfacial energy is measured experimentally using two methods. The interface is stabilized in a thermal gradient and the shape is measured in the vicinity of a reentrant grain boundary. A theory for such coupled- groove constrained equilibrium shapes is presented. These coupled groove solutions are used to extract the anisotropy parameters from the shape measurements. In addition, the Wulff plot is independently estimated for a binary alloy by measuring the equilibrium shape of droplets in a uniform temperature field.

11:35 AM
Dynamic Solute Redistribution of Al-Mg Alloy in Gas Arc Welding: *Eisaku Tokuchi*[1]; Kimioku Asai[1]; [1]Musashi Institute of Technology, Mech. Eng., 1-28-1 Tamazutumi, Setagaya-ku, Tokyo 158 Japan

Experimental investigation was conducted to elucidate the solute redistribution behavior of rapid solidification using Al-Mg alloy 5052 and additionally Al-2%Mg and Al-4%Mg alloy. TIG spot welding was performed in the center of those sheets. Thermal behavior of the welds was carefully measured with CA thermocouple, and the interface progress was microscopically quantified by applying chemical treatment to the fractography in the high-speed breaking technique. The XPS depth profiling in conjunction with Ar-ion sputtering could not accurately depict the solute concentration of the liquid film in the fractured section because of the high-temperature oxidation. Therefore, the fractured sections were made under the water solution containing surface active agents to prevent the electric diffusions. The discussion will be focused on the effective method for diminishing segregations or preventing the hot cracking in the weld rapid-solidification. All the data were obtained at Musashi Institute of Technology in 1994 and 1995 as a graduate program.

General Abstract Sessions: Composites

Sponsored by: TMS
Program Organizers: Thomas P. Battle, E. I. DuPont de Nemours & Company, Inc., White Pigments and Mineral Products, Edgemoor, DE 19809 USA; TMS, Warrendale, PA 15086 USA

Wednesday AM — Room: 229
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Nikhilesh Chawla, Arizona State University, Chem. & Mats. Eng., Tempe, AZ 85287; Mark L. Weaver, University of Alabama, Metall. and Mats. Eng., Tuscaloosa, AL USA

8:30 AM
Load Sharing in Tungsten Continuously Fiber Reinforced Kanthal MMC's: *Bjorn Clausen*[1]; Mark A.M. Bourke[2]; [1]Los Alamos National Laboratory, LANSCE-12, P.O. Box 1663, MS H805, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-8, P.O. Box 1663, MS H805, Los Alamos, NM 87545 USA

Neutron diffraction permits the measurement of phase specific lattice elastic mean phase strains in bulk samples, and therefore it is a valuable tool in the investigation of load sharing in MMC's. Using the in-situ loading capabilities at LANSCE we measured the development of phase strains during tensile loading of tungsten continuous fiber reinforced Kanthal MMC's. The tungsten/Kanthal system has drawn attention for its good high temperature characteristics. The work includes results for samples with various volume fractions of fibers. In the initial elastic region, the two phases are subjected to uniform deformation due to the constraints of the long fiber geometry. The onset of plasticity in the Kanthal matrix results in divergens in the elastic phase strains. Furthermore, the elastic and plastic anisotropy of the Kanthal matrix have been determined from the neutron measurements. The results have been compared to finite element models and self-consistent polycrystal deformation models.

8:55 AM
Stress Intensity Factor Approach for Estimation of Fracture Behavior of Cenospheres Filled Polymers: *Nikhil Gupta*[1]; Eyassu Woldesenbet[1]; [1]Louisiana State University, Mech. Eng. Dept., 2508, CEBA Bldg., Baton Rouge, LA 70803 USA

Fracture of cenosphere leads to generation of hollow space (void) in filled polymers. This hollow space can be considered as a macrocrack in the material. With the deformation of spherical void into ellipsoidal shape, stress intensity factor increases. Present study deals with the change in the stress intensity factor due to deformation of the void to obtain an estimate of stress level in the material. Effect of filler volume fraction coupled with the stress level in the material is used to develop understanding of fracture behavior of the filler containing composites.

9:20 AM
Synthesis and Property Evaluation of Orthorhombic Alloy-Based Particulate Composites: *Masuo Hagiwara*[1]; Satoshi Emura[1]; [1]National Research Institute for Metals, Third Res. Grp., Sengen 1-2-1, Tsukuba, Ibaraki 305-0047 Japan

An orthorhombic Ti-23.5Al-25.3Nb (at.%) alloy powder with 6.5 mass% TiB particulates was produced by gas atomization process. The as-atomized powder having a diameter of between 45 μm

and 150 μm was consolidated by hot isostatic pressing at 1,373K for 10.8 ks with 200 MPa pressure, rolled into 12-mm square bars at 1,423K and heat-treated at 1,523K for 3.6ks followed by slow cooling. The extremely fine needle-shaped TiB particulates with a diameter less than 1 μm and a length from 1 μm to 10 μm were dispersed uniformly in the O+B2+α2 matrix. Room temperature ultimate tensile strength of about 1,300 MPa with elongation of 2 % was obtained. The room temperature high cycle fatigue strength, high temperature tensile strength, Young's modulus and creep properties were superior to those of the unreinforced matrix alloy.

9:45 AM

Composite Anode Development by Novel Techniques: *Mark Alexander Haldane*[1]; Patricia Deanne Cameron[1]; T. H. Etsell[1]; [1]University of Alberta, Chem. and Mats. Eng., 536 Chem. and Mats. Eng., Edmonton, Alberta T6G 2E1 Canada

This paper compares two techniques for producing ceramic-metal (cermet) anodes for solid oxide fuel cells (SOFC's). The first technique involves the precipitation of nickel onto yttria stabilized zirconia (YSZ) powder via autoclave reduction. The nickel coated YSZ powder is then pressed and sintered to form an anode disk. This disk consists of a continuous metallic nickel matrix surrounding a ceramic network. In the second technique, a metal anode is applied to the electrolyte, and a modified form of electrochemical vapor deposition (EVD) is used to apply a thin coating of YSZ over the surface of the metal. The resulting anode is metal with a thin coating of ceramic. Electrodes resulting from these two unique processing methods will then be examined. These results will potentially lead to a better understanding of what elements are most important to SOFC anode performance.

10:10 AM Break

10:20 AM

Cyclic Loading and Residual Strains in Cu/25%Ag Composites: *Kazunari Maeda*[1]; Heinz Nakotte[1]; Sung Chang[1]; Gabe Garcia[2]; Sean Barley[2]; Jim Richardson[3]; Ke Han[4]; J. D. Embury[4]; Bjorn Clausen[4]; Mark A.M. Bourke[4]; [1]New Mexico State University, Phys. Dept., MSC3D, Las Cruces, NM 88003 USA; [2]New Mexico State University, Mech. Eng. Dept., Las Cruces, NM 88003 USA; [3]Argonne National Laboratory, Intense Pulsed Neutron Source, Argonne, IL 60439 USA; [4]Los Alamos National Laboratory, Los Alamos, NM 87545 USA

The development of high-performance pulsed magnets requires wire materials with good electrical conductivity and higher mechanical strength than can be achieved by pure copper. Cold-drawn Cu/Ag composites are widely discussed as possible candidates. However, an understanding of the co-deformation behavior and the evolution of residual strains are needed since these parameters affect the short- and long-term fatigue performance. Here, we report on neutron-diffraction studies of Cu/25%Ag wires cold-drawn to a strain of 2.6 with cross sections of 4*6 mm^2. The composites were exposed to cyclic mechanical loading (up to 20% of the elastic limit), and the development of residual strains was studied up to 10^6 cycles (at which the wires were found to catastrophically fail). We find that most of the compressive and tensile strain develops in the Ag fibers, and the results are compared with the predictions of anisotropic finite-element modeling.

10:45 AM

Properties of Aluminum Matrix Composites Processed by a New ECAP Process: *Yoshinori Nishida*[1]; [1]National Industrial Research Institute of Nagoya, Comp. Mats. Lab., Hirate-cho, Kita-ku, Nagoya 462-8510 Japan

Equal channel angular pressing became attractive, because it is possible to apply large strain to the billets without change of the original shape. However, as the strain by one pass of the ECAP is not large, about ten passes are necessary to obtain fine grain. By the conventional ECAP process, the pressed out sample must be inserted into the die again and the operation is continued about ten times. Then, the conventional one is not efficient and it is not easy to control samples temperature. To solve those problems, we proposed a new ECAP process, which is characterized by the rotary die. By the new ECAP, we can continue ECAP procedure without taking out the sample from the die and temperature control is easy and precise because the sample is in the cavity of the rotary die. The new ECAP process was applied to aluminum matrix composites (7075/SiCw, 7075/Al2O3 short fiber) to obtain fine microstructure. The properties of the ECAP processed aluminum matrix composites are discussed.

11:10 AM

Estimations of the Interfacial Fracture Energy of a Cu/Cr/PI System by the T-Peel Test: *Jae-Yong Song*[1]; Jin Yu[1]; [1]Korea Advanced Institute of Science and Technology, Mats. Sci. and Eng., 373-1, Kusong-dong, Yusong-gu, Taejon 305-701 Korea

T-peel tests were conducted to determine the metal/polymer(m/p) adhesion strength of a Cu/Cr/PI structure with interface precracks between Cr and PI, used in electronic packaging. Effects of the biased RF plasma pretreatment and the metal layer thickness on the peel strength(P) were investigated, and the energy dissipated by plastic bending(Ψ) and the interfacial fracture energy(ϕ) are estimated. During the steady state peeling, the peel angle(Γ) and the maximum curvature at peeled film bases were directly measured by using an optical camera, from which Ψ can be deduced from the elastic/plastic analysis of Kim and Aravas. P, Ψ and ϕ values vary with the plasma density(ρ) and the metal layer thickness. The interfacial fracture energy between Cr and PI which increases with ρ but is independent of m/p layer thickness were deduced. Later, Γ values were compared with the theoretical analysis by Moidu et. al. and Wei and Hutchinson.

11:35 AM

Production and Properties of Sintered Cr-MgO Composites: Jason K. Morgan[1]; Wesley S. Rollings[1]; *Mark L. Weaver*[1]; [1]The University of Alabama, Metall. & Mats. Eng., P.O. Box 870202, A129 Bevill, Tuscaloosa, AL 35487-0202 USA

This paper presents the results of measurements of the basic mechanical properties of sintered Cr and Cr-MgO composites are reported over the temperature range 25°C-900°C. The materials were characterized by compression tests, three-point bend tests and miniaturized disk bend tests coupled with metallographic characterization, and fractrographic studies. Results for the temperature dependence of the yield strength, fracture strength, and fracture toughness are presented. The deformation and fracture mechanisms in the above temperature range are discussed on the basis of microstructural studies by optical, scanning electron, and transmission electron microscopy.

High Temperature Coatings - IV: Metallic/Intermetallic Coatings and Oxidation

Sponsored by: Materials Processing and Manufacturing Division, ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee, Surface Engineering Committee

Program Organizers: Narendra B. Dahotre, University of Tennessee Space Institute, Cen. for Laser Appl., Tullahoma, TN 37388 USA; Janet Hampikian, GA Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Wednesday AM Room: 219
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Brian Gleeson, Iowa State University, Dept. of Mats. Sci. and Eng., Ames, IA 50014; Igor Zhitomirsky, McMaster University, Dept. of Mats. Sci. and Eng., Hamilton, Canada

8:30 AM

Development of Protective Coatings for High-Temperature Metallic Materials: *R. Keith Bird*[1]; Terryl A. Wallace[1]; Sankara N. Sankaran[2]; [1]NASA Langley Research Center, Met. and Therm. Struct., Mail Stop 188A, Hampton, VA 23681-0001 USA; [2]Ana-

lytical Services & Materials, Inc., 107 Research Dr., Hampton, VA 23666 USA

Temperature-resistant metallic materials are key components to many land-based and space-based systems where high-temperature performance is critical. Replacement of conventional alloys with advanced alloys that offer enhanced properties and/or reduced weight offers potential for improved structural performance. Temperature-resistant superalloys, such as PM1000 and 602CA (a relatively new nickel alloy) were investigated for structural applications where the service temperature may attain 1800°F, with Inconel 617 serving as a baseline for comparative purposes. In addition, gamma titanium aluminide was investigated for structural applications where service temperatures are in the 1600°F regime. Due to the extreme operating environments, these metallic materials will require coatings for thermal control and oxidation protection. Ultrathin, lightweight protective coatings have been developed and evaluated to reduce oxidation, increase emittance, and reduce the catalytic efficiency for recombination of dissociated species for these candidate structural materials. This paper describes the mechanical properties of the alloys after exposure to oxidation environments in the coated and uncoated conditions. In addition, the emittance and catalysis behavior of the coated materials is described.

8:55 AM Invited

Evaluation of Iron-Aluminide CVD Coatings for High-Temperature Corrosion Protection: B. A. Pint[1]; *P. F. Tortorelli*[1]; J. A. Haynes[1]; I. G. Wright[1]; [1]Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6156 USA

Under high-temperature oxidation conditions, an aluminide coating forms an alumina scale that normally is less affected by the presence of sulfur or water vapor than those based on chromia or silica. As part of an evaluation of aluminide coatings formed by a variety of processes, the oxidation and sulfidation behavior of iron-aluminide surface layers formed by chemical vapor deposition on steels was studied. Coated specimens of Fe-9Cr-1Mo steel and type 304L stainless steel were exposed at 800°C to dried air, air+10% water vapor, and a hydrogen/hydrogen sulfide/water vapor gas mixture. Initial results showed significantly lower corrosion rates for the aluminized specimens when compared to the uncoated steels. However, iron aluminides have a high coefficient of thermal expansion and this may represent a long-term problem related to mechanical integrity under thermal cycling.

9:20 AM

Rare Earth Oxide Coatings for Life Extension of Chromia Forming Alloys: *Lalgudi Ramanathan*[1]; Stela M. de Carvalho Fernandes[1]; [1]Instituto de Pesquisas Energeticas e Nucleares, Mats. Sci. and Eng., Travessa R-400 Cidade Universitaria, Sao Paulo 05422-970 Brazil

The influence of adding small quantities of rare earth elements to high temperature alloys on improvements in oxidation rate and oxide scale adhesion is well known. Traditionally, the rare earths (RE) have been added to the alloys either in elemental form or as an oxide. The RE can be also applied as coatings to alloy surfaces. Several methods can be used to apply RE coatings to alloy substrates and the sol-gel process is considered to be more efficient and is known to generate the smallest oxide particles. This paper presents the different techniques for producing rare earth oxide coatings and discusses the effects of applying RE oxide sols prepared by different methods on the morphology of the coating and consequently on the oxidation behavior of a Fe-20Cr alloy at 1000°C. The influence of the type of rare earth (Ce, Y, Dy, Pr, La, Nd) and additive on gel morphology and overall oxidation behavior will be presented and discussed.

9:40 AM

Role of Temperature in the Surface Oxidation Chemistry of IN-738 Superalloy: *Sudipta Seal*[1]; Leyda Bracho[1]; Vimal Desai[1]; [1]UCF, AMPAC and MMAE, 4000 University Blvd., Orlando, FL 32816 USA

Super-alloys are used in high temperature industrial applications because of their strength, high resistance to high-temperature oxidation and hot corrosion, and longer durability than other conventional alloys. Although superalloys possess these characteristics, they are still subjected to high temperature degradation when exposed to aggressive environments. The current research includes the high temperature oxidation behavior of IN-738 at 850, 900, and 950°C in dry air for a period of 300 consecutive hours. The oxidation kinetics is parabolic in nature. Due to its parabolic nature, the lattice diffusion is prominent in the various oxide formations. This leads to selective oxidation of various elements present in the alloy. The decrease in oxidation rate observed at higher temperatures is due to more alumina formation. The surface oxide formation is investigated using XPS, AES, XRD, SEM, and EDS. It is expected to find external oxides rich in chromium, aluminum, nickel, and titanium. The thickness of the oxide and the gamma' depleted layers are expected to increase with temperature.

10:00 AM Break

10:15 AM

Oxidation Kinetics and Morphology of Laser Surface Engineered Hard Coating on Aluminum: *Narendra B. Dahotre*[1]; Lalitha R. Katipelli[1]; [1]University of Tennessee Space Institute, Dept. of Mats. Sci. & Eng., Center for Laser Applications, MS 24, 411 B.H. Goethert Parkway, Tullahoma, TN 37388 USA

Laser Surface Engineering (LSE) technique was employed to deposit refractory TiC coating on 6061 Al alloy. Oxidation behavior of the coating was studied for long term exposure at high temperature. Morphology and phase transformation of the oxidation product and kinetics of the oxidation process were also studied. Due to composite (TiC/Al)nature of the coating, variation in the type of product, nature and kinetic rate of oxidation was observed. The coating parameters such as thickness and TiC or Al content influence the oxidation behavior indicating that LSE can be utilized to synthesize the coating with tailored properties.

10:35 AM

The Role of Metallic Coatings on the Structure, Wetting and Mechanical Properties of Metal-Ceramic Interfaces: Natalia Sobczak[1]; *Rajiv Asthana*[2]; [1]Foundry Research Institute, 73 Zakopianska St., 30-418 Krakow, Poland; [2]University of Wisconsin-Stout, Techn. Dept., 326 Fryklund Hall, Menomonie, WI 54751 USA

The paper reviews and previews recent research in the field of metallic films used as technological coatings on ceramic substrates to improve the wettability and/or compatibility in different metal-ceramic systems. The experimental data on the sessile drop wettability tests are analyzed from the viewpoint of type, thickness, and quality of a metallic film, wettability test temperature, and alloying additions to contacting metal or ceramic materials. A new 'push-off' test that allows shearing the solidified sessile-drop samples with less than 90 degree contact angle was used to measure the interfacial shear stress between the ceramic substrate and the metal under various conditions of film thickness, film quality, wettability test temperature, and alloying. Experimentally determined values of shear strength on coated and uncoated ceramic substrates are summarized to improve our understanding of wetting-structure-bonding relationship in chosen metal-ceramic systems.

10:55 AM

High Temperature Oxidation of Beryllium Modified Mo5Si3: *Robert Joseph Hanrahan*[1]; Jason C. Cooley[1]; William L. Hults[1]; Dan J. Thoma[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Techn. Div., MST-6, TA3 MS G770, Los Alamos, NM 87545 USA

In high temperature oxidation it is usually accepted that protective oxides are only formed on alloys containing Cr, Al, or Si. Beryllium compounds also usually form a protective layer of BeO. In many compounds beryllium also exhibits very rapid diffusion allowing protective oxide formation at relatively low concentrations of Be. This behavior also can result in early formation of beryllium containing oxides rather than transient phases observed in the case of alumina forming alloys such as NiAl. Be additions have also been investigated as a route to improving the oxidation resistance of chromia forming alloys and titanium aluminides. In all of these cases Be additions have resulted in varying degrees of improvement. In this investigation we have studied the effects of Be additions on the oxidation mechanism of Mo5Si3 modified by addition of Be alone

and with both Be and boron, with particular emphasis on the early stages of oxidation and the effects of moisture.

11:15 AM

Oxidation Resistant Coatings for Nb Silicide Composites: *J.-C. Zhao*[1]; Melvin R. Jackson[1]; [1]General Electric Company, Corp. Res. & Dev., P.O. Box 8, Schenectady, NY 12301 USA

Nb silicide-based composites show high promise as next-generation gas turbine airfoil materials with significantly higher operating temperatures than Ni-based superalloys. The oxidation resistance of these composites is a significant challenge considering the very high potential operating temperatures. Coating development is critical for the application of these composites in the gas turbine hot-section environment. This presentation will describe our effort in developing coatings for Nb silicide composites. The behavior of thermal barrier coatings (TBC) on Nb silicide composites will also be described.

11:35 AM

Microhardness and Scratch Resistance of Cr/CrN Coatings on Steels: *Ray Y. Lin*[1]; Jin Seok[1]; Ming Chen[2]; [1]University of Cincinnati, Mats. Sci. and Eng., M.L. #12, Cincinnati, OH 45221-0012 USA; [2]AFRL/MLBT, Bldg. 654, 2941 P St, WPAFB, OH 45433-7750 USA

Nanocrystalline chromium and chromium nitride composite coatings on steels have been prepared with a reactive magnetron sputter deposition technique. X-ray diffraction analysis of the coated samples indicates that nanocrystalline Cr and CrN coexisted in the coatings. Cross section microstructural examinations with a scanning electron microscope show excellent coatings with no void at the interface. Microhardness and scratch tests were used to characterize the coating. A model has been developed to determine the coating hardness using the measured composite hardness and the substrate only hardness. While the steel hardness is only around 130 kgf/mm2, the coated steel hardness is about 165 kgf/mm2 with 1.9 Ym Cr/CrN coatings. This corresponds to a calculated coating hardness of about 1200 kgf/mm2. It has been found that the higher the nitrogen content, the higher the coating hardness. Scratch test results also confirm the microhardness data on the effect of nitrogen contents.

International Symposium on Deformation and Microstructure in Intermetallics: Creep

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Physical Metallurgy Committee, Jt. Mechanical Behavior of Materials

Program Organizers: Katherine C. Chen, University of California, Polytechnic State University; Peter M. Hazzledine, UES, Inc., Dayton, OH 45432 USA

Wednesday AM Room: 220
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: ; Jorg M.K. Wiezorek, University of Pittsburgh, Pittsburgh, PA USA

8:30 AM Invited

Pairwise Cutting of Ordered γ Particles During High Temperature and Low Stress Creep of Superalloy Single Crystals: *Michael J. Mills*[1]; Gunther F. Eggeler[2]; Srinivasan Rajagopalan[1]; [1]The Ohio State University, Mats. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Ruhr Universitat Bochum, Lehrstuhl Werkstoffwissenschaft, IA 1-125, Universitaetsstrasse 150, Bochum 44800 Germany

The cutting of ordered γ particles is believed to be a rate controlling mechanism in Ni-based superalloys under conditions of high temperature and low stress creep. In double shear creep tests, the pairwise cutting of the γ'-phase in CMSX4 has been observed to occur by a/2<011> dislocations with different Burgers vectors, forming an a<010> superdislocation. HRTEM studies show a dissociated core structure (comprised of two a/2<011> dislocations separated approximately 25 Å) for the a<010> dislocation along the <101> edge orientation. A combined climb and glide process of the superpartials is presumably responsible for the overall motion of the a<010> dislocation. a<010> dislocations (with no resolved shear stress for glide or climb) have also been observed in the γ'-phase during creep studies on NASAIR-100 superalloys loaded along the <001> orientation. A model is presented predicting minimum creep rates, based on the above observations.

9:00 AM Invited

Mechanistic Understanding of Creep in Gamma-Base Titanium Aluminide Alloys: *Fritz Appel*[1]; [1]GKSS Research Centre Geesthacht, Instit. for Matls. Rsch., Max-Planck-Str., Geesthacht D-21502 Germany

Two-phase titanium aluminides with a fully-lamellar microstructure exhibit a relatively good creep resistance, typically reducing strain rates at given test conditions by at least one order of magnitude, when compared with their duplex counterparts. Nevertheless, lamellar materials often exhibit high primary creep rates and significant structural changes under long-term creep loading, which are unacceptable for the intended applications. The mechanisms controlling these properties are not yet fully understood, partly due to the complexity and fine scale of the defect processes involved. The paper's main objective is to elucidate the intimate correlation between interface-related defect processes and the creep properties of lamellar materials. The analysis bases on long-term creep tests and transmission electron microscope observations utilizing weak-beam darkfield, high resolution and in situ techniques. The major areas of the study are: (i) the structure and stress state of lamellar interfaces, (ii) interface-related nucleation of dislocations and mechanical twins, (iii) the role of misfit-compensating ledges and dislocations in phase transformations and recrystallization. The implications of the findings will be discussed with respect to alloy design towards improved creep strength.

9:30 AM Invited

Relationship Between Tensile and Primary Creep Properties of Near Gamma-TiAl Intermetallics: *Jonathan Beddoes*[1]; Linruo Zhao[2]; [1]Carleton University, Mech. & Aeros. Eng., 1125 Colonel By Dr., Ottawa, Ontario K1S 5B6 Canada; [2]Structures, Materials & Propulsion Laboratory, Inst. for Aeros. Res., Nat. Res. Coun., Ottawa, Ontario K1A 0R6 Canada

This paper correlates the tensile and primary creep behaviour of near g-TiAl compositions in various microstructural states. Minimizing the total primary creep is key for several envisaged applications. Primary creep consists of two major components-the instantaneous strain and a subsequent primary transient. The primary creep behaviour at 760°C and 207 or 276 MPa of investment cast, P/M and XD? binary, ternary and multi-component compositions in the duplex and various fully lamellar conditions is evaluated. The contribution to primary creep of the instantaneous strain and subsequent primary transient is dependent on the microstructural state and can be correlated to tensile yield behaviour at 760°C. Microstructural variables such as grain size, lamellar interface spacing and third phase precipitates can significantly influence the primary creep behaviour. The results are discussed in terms of the microstructural factors controlling tensile and primary creep deformation.

10:00 AM Break

10:20 AM Invited

Microstructure and Creep Behavior of Directionally Solidified TiAl-base Alloys: *David R. Johnson*[1]; Ho Nyun Lee[2]; Shinji Muto[3]; Takamitsu Yamanaka[3]; Haruyuki Inui[3]; Masaharu Yamaguchi[3]; [1]Purdue University, School of Materials Engineering, 1289 MSEE Bldg., West Lafayette, IN 47907-1289 USA; [2]KAIST, Dept. of Mats. Sci. and Eng., Taejon 305-701 Korea; [3]Kyoto University, Dept. of Mats. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan

Tensile creep tests were conducted on directional solidified TiAl alloys to discern the effect of lamellar spacing, lamellar orientation, and alloying. A seeding technique was used to align the TiAl/Ti_3Al lamellar structure parallel to the growth direction for alloys of Ti-47Al, Ti-43Al-3Si, Ti-46Al-0.5Si-0.5X (X=Re, W, Mo, and Cr), and Ti-46Al-1.5Mo-0.2C. Different heat treatments were used to vary the lamellar spacing of selected alloys and tensile creep tests

were performed at 750°C using applied stresses of 180, 210, and 240 MPa. The amount of primary creep can vary by as much as a factor of 5 just from changes in the lamellar spacing. Silicide and carbide precipitates as well as refractory metal additions significantly reduced the secondary creep rate. Differences in the microstructures of these alloys will be highlighted and correlated with creep and tensile test results.

10:50 AM Invited

The Anomalous Strain Rate Dependence of the Work Hardening Rate in Single Crystal Ni3 (Al, Ta): *Michael D. Uchic*[1]; W. D. Nix[2]; [1]Air Force Research Laboratory, Mets. Dev. and Mats. Process. Brnch., 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA; [2]Stanford University, Dept. of Mats. Sci. and Eng., Stanford, CA 94305 USA

The mechanical properties of many metals are fairly insensitive to changes in strain rate, but in general, increasing the strain rate results in modest increases the yield and ultimate strengths, the work hardening rate (WHR), and ductility. This study examines the unusual dependence of the work hardening rate on the applied strain rate at low temperatures in the anomalous flow regime for Ni3Al. During tension creep testing of <123> oriented single crystals of Ni75Al24Ta1 at 100°C, it was observed that samples strained at a very high ratesótrue strain rates of approximately 10-2 s-1ódisplayed a lower work hardening rate, approximately one-half the value obtained at a slower strain rate (10-5 s-1). This result is even more remarkable in that this behavior was not observed in similar experiments performed at either 20 or 200°C. However, the creep apparatus used for these experiments was not an ideal instrument with which to explore this unusual rate dependence. In order to better characterize this atypical dependence of the work hardening rate on the applied strain rate, a constant displacement rate test system has been used to test single crystals of Ni3Al from 20-200°C at three different crystallographic orientations (<123>, <111>, and <001>) and at both fast (10-1 and 10-2 s-1) and more conventional strain rates (10-5 s-1). In this study we also characterize the microstructure of selected samples by TEM, and introduce model for work hardening in Ni3Al which examines the hardening process as competition between the exhaustion and annihilation of mobile dislocations, which is dependent the on temperature, strain rate, and crystallographic orientation.

11:20 AM

The Controlling Creep Mechanisms in TiAl-Base Alloys in the Service Conditions: *Wei-Jun Zhang*[1]; Seetharama C. Deevi[1]; [1]Chrysalis Technology, Inc., Res, Cen, 4201 Commerce Rd., Richmond, VA 23234 USA

TiAl-base alloys are candidate materials for gas turbine and automotive engine alppications. In many cases, the TiAl components will be exposed to a relatively lower stress level of 100-200 MPa at 700-815°C. Unfortunately, most of the studies on creep of TiAl were carried out in relatively higher stress regime and little work was done on the limiting creep mechanisms in TiAl alloys at lower stress level. In this paper, the creep deformation substructure of TiAl-(Nb, W) alloys were examined using TEM after creep tests at the stresses of 100, 140 MPa and 200MPa at 760°C. The contribution of dislocation glide and climb and lamellar interface diffusion was discussed. The results suggest that solid solution of refractory element is more effective in improving the creep resistance of TiAl alloys than refinement of the lamellar spacing in the service conditions.

11:40 AM

Creep and Aging Behavior of a Fully Lamellar Gamma Titanium Aluminide Alloy Containing Carbon and Silicon: *Karthikeyan Subramanian*[1]; Gopal Viswanathan[1]; Y. -W. Kim[2]; Michael J. Mills[1]; [1]The Ohio State University, Dept. of Matls. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]UES, Inc., Dayton, OH 45432 USA

A fully lamellar γ-TiAl alloy containing carbon and silicon (K5SC alloy) has been found to have superior creep resistance. The aging characteristics, α_2 dissolution and precipitation of B2 phase, carbides and silicides have been studied by electron microscopy techniques. Precipitate formation was seen along the interlamellar boundaries and is the result of supersaturation of C and Si in γ formed from the dissolution of metastable α_2. B2 formation is more extensive in alloys without C and Si (K5 alloy) and results from chemical inhomogeneities in the distribution of alloying elements (Nb, Cr and W) in α_2. High temperature creep tests indicate that K5SC alloys have a minimum creep rate an order of magnitude lower than the K5 alloys. Stress jump tests along and microscopic evidence of substructure formation in the K5 alloys, lead us to believe that this alloy creeps in a pure metal fashion. The absence of substructure formation in the K5SC alloys suggests a possible solid solution st rengthening effect. Transmission electron microscopy on crept samples of the K5SC alloy show extensive interaction of dislocations on lamellar interfaces and precipitates, indicating a complementary precipitation strengthening mechanism.

International Symposium on Shape Casting of Aluminum: Science and Technology: Advances in Heat Treatment and Mechanical Behavior

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Aluminum Committee, Non-Ferrous Metals Committee, Solidification Committee, Jt. Mechanical Behavior of Materials
Program Organizers: John E. Allison, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48124-2053 USA; Dan Bryant, Chester, VA 23836-3122 USA; Jon Dantzig, University of Illinois, Department of Mechanical & Industrial Engineering, Urbana, IL 61801-2906 USA; Ray D. Peterson, IMCO Recycling, Inc., Rockwood, TN 37854 USA

Wednesday AM Room: 224
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: J. Daniel Bryant, Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA; Yumin Ruan, Alcoa Technical Center, Alcoa Center, PA USA

8:30 AM Keynote

Heat Treatment, Friend or Foe?: *Salvador Valtierra*[1]; Raymundo Gomez[1]; Miguel Angel Cisneros[1]; David Gloria[1]; [1]Nemak Corporation, P.O. Box 100 Bosques del Valle, Garza, Garcia, NL 66221 Mexico

Aluminum silicon alloys are by far the most important commercial casting alloys, because of superior casting characteristics, producing a good range of physical and mechanical properties. This is an ideal material for the automotive industry in its search for lighter-weight, high quality and low cost components that meet the current environmentally and fuel economic regulations. With auto industry casting requiring nowadays, aircraft industry properties heat treatment is an important factor used to enhance the mechanical properties of the aluminum castings. This involves optimizing both, solution heat treatment and aging treatment. Unfortunately heat treatment is an expensive process and will affect not only cost, but also it can Negatively influence the machinability of the casting and its dimensional stability, and Positively influence the mechanical properties, the fatigue performance and the thermal conductivity of the casting. The present approach is to use all the tools available to fully take advantage of an expensive process that definitively is a friend, but can become a foe even without anybody realizing it.

9:15 AM Invited

A Quality Shape Casting: *Sander A. Levy*[1]; [1]AluBest Consulting, 3625 Milbury Run St., Richmond, VA 23233-7670 USA

The fastest growing portion of the utilization of aluminum in automotive applications is in the field of shape castings. Such applications require an excellent casting. The requirements include mechanical properties, grain size, silicon particle size and shape (modification), and low porosity and inclusion levels. Thirty years ago "mag wheels" were only available as aftermarket equipment. Qual-

ity was extremely poor and elongation levels were only a few percent. Over the years techniques were developed to improve the quality of cast aluminum wheels. Currently the elongation values are generally greater than 10 percent and many millions are sold as original equipment. The measures, which were adopted to quantify and improve these important properties, will be reviewed.

9:45 AM

On the Use of Work Hardening Characteristics to Predict the Ideal Tensile Strength and Elongation of Cast Al-7%Si-Mg Alloys: *Murat Tiryakioglu*[1]; John Campbell[2]; James T. Staley[3]; [1]Western Kentucky University, Dept. of Manuf. Sci., Bowling Green, KY 42101-3576 USA; [2]University of Birmingham, School of Metallurgy and Materials, IRC in Materials for High Performance Applications, Edgbaston, Birmingham B15 2TT UK; [3]Retired, Durham, NC USA

Cast Al-7%Si-Mg alloys are widely used in aerospace and automotive applications due to their excellent castability. Despite their wide use, the effect of different factors on tensile properties is not completely understood. This is probably due to the fact that there exists a defect hierarchy in these alloys. Recent research has shown that work hardening characteristics of these alloys can be used to assess the effect of microstructural defects on the tensile properties in these alloys. For this study, Al-7%Si alloys with three different Mg levels (0.20, 0.40 and 0.60 wt.%) were investigated with artificial aging times at 200°C ranging from 0 to 128 hours. Tangent modulus-true stress curves showed that failure occurs due to the presence of defects in the structure, before the Considere criterion is met. The Kocks-Mecking work hardening model and the Voce true stress-true strain equation were used to predict the tensile stress and elongation in the absence of defects in the structure. Relationships that can be used to predict tensile strength and elongation as a function of yield stress are presented.

10:15 AM Break

10:30 AM

A New Quality Factor to Assess the Tensile Properties of Cast Al-Si-Mg Alloys: An Energy Approach: *Murat Tiryakioglu*[1]; James T. Staley[2]; John Campbell[3]; [1]Western Kentucky University, Dept. of Manuf. Sci., Bowling Green, KY 42101-3576 USA; [2]Retired, Durham, NC USA; [3]University of Birmingham, Sch. of Metall. and Mats., IRC in Mats. for High Perfor. Appl., Edgbaston, Birmingham B15 2TT UK

Al-7%Si alloys with three different Mg levels (0.20, 0.40 and 0.60wt.%) were investigated with artificial aging times at 200°C ranging from 0 to 128 hours. The Voce equation was found to best describe the true stress-true strain relationship in these alloys. The plastic energy to fracture (E) was calculated for all specimens using the true stress-true strain curves. It was found that the plastic energy to fracture can be approximated by (YS+TS)el/2, where YS is yield stress, TS is tensile strength and el is elongation. An approach to predict the defect-free properties of these alloys was intoduced by the authors. Using this approach, plastic energy to fracture of defect-free samples (E_{df}) was calculated. The new quality factor can then be defined as $Q=(E/E_{df})$.

11:00 AM

Quantitative Microstructure Property Relationships for Tensile Properties in a Cast 319 Aluminum Alloy: *Jacob W. Zindel*[1]; James W. Boileau[1]; Kelly A. Kofeldt[1]; Larry A. Godlewski[1]; John E. Allison[1]; [1]Ford Motor Company, Ford Res. Lab., Mail Drop 3182 SRL, P.O. Box 2053, Dearborn, MI 48121 USA

Microstructural parameters such as grain size, dendrite arm spacing (DAS), eutectic silicon morphology, precipitate type and size, and porosity are all possible factors affecting the tensile properties of cast 319-type alloys. Solidification time has a strong effect on many of these parameters and is commonly used to define the microstructural condition of the material. Since castings have a range of solidification times, they have a corresponding range in microstructural features. The purpose of this study was to isolate and determine the relative effects of these parameters. The results of the study indicate that yield strength is not controlled by grain size or DAS. Yield strength is affected only by the amount of Cu that dissolves into the aluminum during solution treatment. Solidification time does not have a direct effect but does control the amount of time required to dissolve the Cu in the solution treatment portion of the heat treatment. Elongation and ultimate tensile strength are inversely related to solidification time.

11:30 AM

Simulation of Processing, Microstructures and Mechanical Properties in a Cast Engine Block–An Application of Virtual Aluminum Castings: *Ravi Vijayaraghavan*[1]; Mei Li[1]; Jacob W. Zindel[1]; John E. Allison[1]; [1]Ford Motor Company, Mats. Sci., MD 3182, 2101 Village Rd., Dearborn, MI 48108 USA

Computational methods continue to play an increasingly important role in the optimization of materials, processes and products in the castings industry. A major thrust of cast aluminum research at Ford is the development of computational tools to reduce component development time and cost and to optimize cast aluminum engine blocks and cylinder heads. The vision is to create and test virtual aluminum castings, reducing the need for physical prototypes. This talk presents a specific example of an application of the Virtual Aluminum Castings approach. The goal is to predict yield strength in a prototype engine block. First we develop boundary conditions for accurate thermal analysis of a cast aluminum (319 alloy) engine block. Once an accurate thermal history is obtained, this is then used in conjunction with a micromodel to predict the fraction of as-cast Al2Cu phase present in the microstructure. Finally, the microstructural information is used to predict yield strength after solution treatment and aging.

Lead-Free Solder Materials and Soldering Technologies V: Mechanical Properties, Fatigue, Creep

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Sung Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598 USA; Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Hareesh Mavoori, Lucent Technologies, Bell Laboratories, Murray Hill, NJ 07974 USA; Ronald W. Smith, Materials Resources International, North Wales, PA 19454 USA

Wednesday AM — Room: 227
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Hareesh Mavoori, Lucent Technologies, Bell Laboratories, Murray Hill, NJ 07974 USA; K. N. Subramanian, Michigan State University, Dept. Mats. Sci.& Mech., East Lansing, MI USA

8:30 AM Invited

Creep of Lead-Free Solders at 75°C: *William John Plumbridge*[1]; Colin Richard Gagg[1]; Shellene Peters[1]; [1]The Open University, Mats. Eng., Walton Hall, Milton Keynes MK7 6AA UK

Implementation of lead-free solders requires detailed knowledge of their mechanical behaviour. This paper reports the creep behaviour of three lead-free alloys: Sn-0.5Cu, Sn-3.5Ag and Sn-3.5Ag-0.5Cu at 75°C, and compares their response to Sn-37Pb. The Sn-0.5Cu alloy behaves similarly to the Sn-Pb over the range of rupture lives considered (up to 1000h). The silver-containing alloys exhibit much greater creep resistance, typically a hundred fold and a thousand-fold for the binary and ternary respectively. These alloys are less ductile but their creep strains to failure are generally above 10%. Their minimum creep rates are at least one hundred times slower. When testing at the same homologous temperature, the silver-containing alloys retain their superiority. The relationship between applied stress and steady-state creep rate is best described by a power law equation although the steady state domain generally occupies

less than 30% of life. Microstructural changes induced by creep are described.

8:55 AM

Assessment of Low-Cycle Fatigue Life of Sn-3.5mass%Ag-X (X=Bi or Cu) Alloy by Strain Range Partitioning Approach: *Yoshiharu Kariya*[1]; Tomoo Morihata[2]; Eisaku Hazawa[2]; Masahisa Otsuka[2]; [1]The Open University, Mats. Eng. Dept., Walton Hall, Milton Keynes, Buckinghamshire MK7 6AA UK; [2]Shibaura Institute of Technology, Mats. Eng. and Sci. Dept., Shibaura 3-9-14, Minato-ku, Tokyo 1088548 Japan

The strain range partitioning method is used to predict creep fatigue life. The basic concept of this method is that the entire reversed inelastic strain range can be partitioned into four generic components identified with creep, plasticity and the manner in which the components of strain in the tensile half of the cycle are reversed by the compressive half of the cycles (i.e. plasticity reversed by plasticity, plasticity reversed by creep, creep reversed by plasticity and creep reversed by creep). The variations in the component type result in possible differences in the life even if the magnitude of the strain range is same. The adaptability of the strain partitioning approach to creep fatigue of Sn-Ag system solders is examined. Sn-3.5Ag, Sn-3.5Ag-1.0Cu and Sn-3.5Ag-5.0Bi show the four partitioned strain range-life relationships. The details of the four partitioned strain range-life relationships and the damage mechanism of each alloy will be presented.

9:15 AM Invited

Constitutive and Damage Model for a Lead Free Solder: Shengmin Wen[1]; Leon M. Keer[1]; Morris E. Fine[2]; [1]Northwestern University, Civil Eng. Dept., 2145 Sheridan Rd., Tech A236, Evanston, IL 60201; [2]Northwestern University, Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

A unified creep plasticity theory with incorporated damage is presented. The theory uses dislocation energy density concept together with dislocation dipoles formation within PSB's to model the crack initiation process both inside grains and in grain boundaries. Following previous work at Northwestern percolation theory due to increase of microcrack density under cyclic loading was used to model damage. The model is science based, thus should be applicable to different size scaled solder joints. It is also within the framework of phenomenological mechanics so it's easy to implement into any commercially available computational software package. It is a particularly promising approach when an adequate reliability prediction method is needed when the joint size falls into the micron sized range. The model will be applied to experimental data from isothermal and thermomechanical fatigue tests on a eutectic Sn-Ag solder.

9:35 AM

Creep Properties of Sn-3.5Ag-Bi Solders: *S. W. Shin*[1]; D. K. Joo[1]; W. K. Choi; Y. S. Lee[1]; Jin Yu[1]; [1]Korea Advanced Institute of Science and Technology, Mats. Sci. and Eng. Dept., 373-1 Kusong-dong Yusong-gu, Taejon 305-701 South Korea

Lead-free solders is a hot issue in the microelectronic packaging industries. Two strong candidates for the lead free solders are Sn-3.5Ag-Bi and Sn-3.5Ag-Cu alloys. However, the data on mechanical properties of these alloys are rather rare in literature. In this study, Sn-3.5Ag-xBi alloys with varying Bi content(up to 6 wt%) were made and their reactions with the Ni and Cu metallization layers were discussed during the reflow temperature of 240°C. Then, variations of the wetting angle, morphologies of the solder interface, and consumption rates of Ni and Cu with the reflow time were investigated and interfacial phases were identified. Mechanical properties of the Sn-3.5Ag-xBi alloy were investigated by conducting single lap shear fracture toughness and creep tests, and effects of Bi content were analyzed from the perspective of the intermetallic compound formation and the interface composition. And the failure locus analysis were conducted using AES, XPS, etc.

9:55 AM

Comparison of Stress Relaxation Behavior of Lead Free Solder Joints: *Susheel G. Jadhav*[1]; *K. N. Subramanian*[1]; T. R. Bieler[1]; J. P. Lucas[1]; [1]Michigan State University, Dept. of Mats. Sci. and Mech., East Lansing, MI 48824 USA

Stress relaxation is an important process that occurs during the thermomechanical fatigue of the solder joints. In industrial applications, the solder joints are subjected to long dwell periods at the extreme temperatures. Stress relaxation that takes place during these dwell periods has significant implications for the reliability of the solder joints. Stresses that arise due to coefficient of thermal expansion mismatch tend to be relaxed by creep deformation within the solder region in the joint. The distribution of this creep strain within the solder joint is strongly dependent upon the constraints imposed by the joint. In this study, the stress relaxation of eutectic 96.5Sn-3.5Ag, Sn-Ag composite (Sn-Ag solder containing Cu6Sn5 reinforcements added in-situ), 95.5Sn-4Ag-0.5Cu is compared at room temperature and at 150° centigrade. Attempts are also made to understand the various deformation mechanisms operative during the stress relaxation process in the realistic solder joints.

10:15 AM Break

10:30 AM

Creep Properties of Eutectic Sn-Ag Solder and Sn-Ag Composite Solders Containing Intermetallic Particles: *Sunglak Choi*[1]; Fu Guo[1]; T. R. Bieler[1]; K. N. Subramanian[1]; J. P. Lucas[1]; [1]Michigan State University, Mats. Sci. & Mech. Dept., 3536 Engineering Bldg., East Lansing, MI 48824-1226 USA

Solder joints used in automotive under-the-hood application are subject to thermomechanical fatigue (TMF) due to severe temperature fluctuations. During cycling, creep deformation by stress relaxation is very significant during the holding period at both high and low temperatures contributing to damage. This study investigates the creep properties between 22 and 150°C of several Sn-Ag solders with and without intermetallic particles, and how aging affects creep resistance. Using the eutectic Sn-Ag solder as a basis for comparison, Sn-Ag based composite solders containing intermetallic particles such as Cu6Sn5, Ni3Sn4, or FeSn2 were used. Creep strain rates were reduced by as much as 1000 times at room temperature, but in contrast, slightly higher creep rates were observed in some composite samples at higher temperatures. The effects of intermetallic particles on creep properties of Sn-Ag composite solders will be discussed and compared to the eutectic Sn-Ag solder.

10:50 AM

Mechanical Behavior of Sn-Ag Solder Joints under Reversed Stress State: J. Howell[1]; S. L. Choi[1]; *K. N. Subramanian*[1]; [1]Michigan State University, Dept. of Mats. Sci. and Mech., 3536 Engineering Bldg., East Lansing, MI 48824-1226 USA

During service CTE mismatches present in the solder joint impose reversed stress states during heating and cooling portions of temperature excursions. The solder joints also undergo stress relaxation during the holding times at the temperature extremes. Double shear lap specimens consisting of 3/8"X3/8"X1/2" copper rods joined with Sn-Ag solder of realistic thickness were used for the present study, since traditional specimen geometry employed in creep/stress relaxation studies do not facilitate stress reversal. Flow and mechanical behavior of such solder joints were investigated under reversed stress conditions in order to develop a basic understanding of thermomechanical fatigue. Effects of holding time between successive loading, and extent of deformation and rate of straining during each loading, were also evaluated.

11:10 AM

Mechanical Cyclic Fatigue of Lead-Free Interconnects of a Flex-Type BGA: *Jenq-Dah Wu*[1]; S. H. Ho[1]; P. J. Zheng[1]; S.C. Hung[1]; Jim C.L. Wu[1]; [1]Advanced Semiconductor Engineering, Inc., R&D LAB., 26, Chin 3rd Rd., Nantze Export Processing Zone, Kaohsiung 811 Taiwan

Several studies have been ongoing to assure the reliability concern of lead-free interconnects, which include mechanical stress-strain behavior, ball shear strength, and thermal cyclic fatigue. The focus of this paper is on the fatigue life of the Pb-free solder joints under cyclic bending test. It is well known that during the operation life of portable electronic products such as cellular phone, game station and PDA, the PCB is subjected to a random vibration from the number dialing strikes. Therefore, board level reliability of the lead-free solder interconnects becomes very critical as the application of Pb-free packages into products. In this work, bending cyclic experi-

ment of a flex-type BGA is performed to evaluate the structural performance of the lead-free joints. Two different composition of Pb-free solder balls, i.e. Sn/Ag and Sn/Ag/Cu are examined, meanwhile, traditional Sn/Pb joints are also tested as a benchmark. It is observed that both lead-free joints have much better performance than the eutectic one in mechanical fatigue life.

11:30 AM

Observation of Crack Initiation and Propagation in Sn-3.5 Ag Solder Joints: *Bruce Alan Cook*[1]; Iver E. Anderson[1]; Joel L. Harringa[1]; Robert L. Terpstra[1]; James C. Foley[1]; Ozer Unal[1]; [1]Ames Laboratory, Iowa State University, Metallurgy & Ceramics, 253 Spedding, Ames, IA 50011-3020 USA

As implementation and acceptance of Pb-free solders gains worldwide momentum, identification and analysis of failure mechanisms become increasingly important. Moreover, microstructural refinements designed to improve shear strength require an understanding of crack nucleation and propagation modes. In an effort to identify the relationship between microstructure and shear strength in lead-free solders, joints prepared from one such composition, Sn-3.5Ag, were evaluated for evidence of failure during interrupted asymmetric four point bend tests. Shear banding in the Sn-rich matrix and crack nucleation in the vicinity of the intermetallic interface were observed at low displacements. Evidence of plastic flow in the matrix was seen at higher shear loadings. Support received from USDOE-BES, Materials Science Division (contract no.W-7405-Eng-82).

11:50 AM

Effects of Cu, Ag, and Sb on the Creep-Rupture Strength and Thermal Fatigue Behavior of Lead-Free Solder Alloys: *Kazuya Miyahara*[1]; Johji Kunii[1]; Noboru Wade[2]; Seiji Yamada[2]; [1]Nagoya University, Dept. Molecular Design and Eng., Nagoya 464-8603 Japan; [2]Topy Industrial, Ltd., R&D Lab., Toyohashi 441-8510 Japan

The materials used in the present research are a Sn metal and Sn-0.5%Cu, Sn-3.5%Ag, Sn-3.5%Ag-0.5%Cu and Sn-0.5%Cu-0.3%Sb alloys. The effects of Cu, Ag,and Sb on the creep-rupture strength and thermal fatigue behavior of lead-free solder alloys have been investigated. Creep tests were performed at the stress and temperature range of 3 to 12 N/mm2 and 353 to 403K, respectively. Thermal fatigue tests were conducted at a temperature range of 233 to 393K and repeating cycles of heating and cooling were up to 2000. The origin of thermal fatigue crack formation was discussed from the point of view of the maximum thermal stress evaluated by an FEM calculation.

Lightweight Alloys for Aerospace Applications: Advances in Joining

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee

Program Organizers: Kumar Jata, Air Force Research Laboratory, Mats, & Manuf, Direct,, WPAFB, OH 45433 USA; Nack J. Kim, Center for Advanced Aerospace Materials, Pohang 790-330 Korea; Eui W. Lee, Naval Air Warfare Center, Code 4342, MS5, Patuxent River, MD 20670 USA; William Frazier, Naval Air Warfare Center, Aircraft Division, Patuxent River, MD 20670-1908 USA

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Session Chair: Mary C. Juhas, Ohio State University, Columbus, OH USA

8:30 AM Invited

Electron Beam Diffusion Bonding of Titanium Aluminides: *Philip L. Threadgill*[1]; B. G.I. Dance[1]; [1]TWI, Friction and Forge Welding Processes, Granta Park, Great Abington, Cambridge CB1 6AL UK

Conventional diffusion bonding of gamma TiAl can take 0.5 to 1 hour minimum to achieve, as the use of high temperatures restricts the forces which can be applied without deforming the material. By using an electron beam to selectively heat only the bond area, higher stresses can be used, resulting in a bond time of about 1-2 minutes, with only minimal distortion. Since the bonds are made at temperatures below the alpha transus, there is very little change to the microstructure, and the undesirable effects of rapid cooling from above the alpha transus which are found in most other thermal joining processes are avoided. The paper describes the results of a number of experiments on various grades of gamma TiAl, and discusses in detail the advantages, disadvantages and prospects for this novel process.

9:00 AM

Effect of Friction Stir Welding on the Superplastic Behavior of Weldalite Alloys: *H. Salem*[1]; A. Reynolds[2]; J. Lyons[2]; [1]American University in Cairo, Dept. of Mech. Eng., Cairo, Egypt; [2]University of South Carolina, Dept. of Mech. Eng., 300 Main St., Columbia, SC 29208 USA

Al-Cu-Li alloys offer attractive property combinations of low density, high specific strength and modulus and exceptional cryogenic properties. This makes them excellent candidates for a variety of applications. Weldability of the aluminum alloys becomes of great concern when pressurized fuel tanks are manufactured for space launch systems. Friction Stir Welding (FSW) of sheet aluminum alloys has proven its potential advantage over the other fusion welding processes in certain applications. In the current research, the effect of friction stir welding (FSW) on the superplastic behavior of Weldalite 049 and 2095 dynamically recrystallized, superplastic, rolled sheets is investigated. Uniaxial superplastic behavior of the alloys is characterized before and after FSW. Microstructural evolution is assessed through light optical microscopy and transmission electron microscopy.

9:30 AM

Properties of 2297-T8 Al-Li Friction Stir Butt and Lap Joints: *William J. Arbegast*[1]; Gil Braun[2]; Anthony Reynolds[3]; Kumar V. Jata[4]; [1]Lockheed Martin Space Systems Michoud Operations, Prog. and Techn. Dev., P.O. Box 29304, New Orleans, LA 70189; [2]Lockheed Martin Space Systems, Astron. Oper., Denver, CO; [3]University of South Carolina, Mech. Eng. Dept., Columbia, SC; [4]Air Force Research Laboratory, MLLM, 2230 Tenth St., WPAFB, OH 45433

Friction Stir Welding is being developed as a replacement for riveted joints in aerospace and aircraft structures. A typical wingbox assembly has been fabricated from 0.25" thick details machined from 2.4" thick 2297-T8 Al-Li plate and joined by the FSW process. The method of fabrication of this 2297 wingbox assembly and the 7075 pathfinder assembly are described. The tensile and lap shear strengths, and, the S/N fatigue (tension and bending) properties are presented for those 2297 Al-Li butt, fillet, “T”, and “L” joint configurations typical of this wingbox. Comparisons are made to those properties expected of related riveted joints. Metallurgical analysis of the various joint configurations is discussed with correlation of joint microstructure to joint performance.

9:55 AM

Characterization of Reinforcing Particle Size Distribution in a Friction Stir Welded Al-SiC Extrusion: *Sarah C. Baxter*[1]; Anthony P. Reynolds[1]; [1]University of South Carolina, Dept. of Mech. Eng., 300 Main St., Columbia, SC 29208 USA

Friction stir welding (FSW) is a new technique that shows great promise for improving the quality of welds in high strength aluminums. Relative motion between a rotating, non-consumable tool and the work-piece produces a solid state weld via in situ extrusion and forging. In this work, friction stir welds were made on a 7093-25% SiC+15% Al extrusion. Statistical image analysis was applied to metallographic sections of the as extruded and as welded material, to investigate and characterize the material microstructure within the weld. Of particular interest is the change in particle size distributions across/through the weld. Strength measurements were also made of the resulting joints.

10:15 AM

Investigation of Gamma Titanium Aluminide GTA Welding Parameters and its Influence on the Weld Pool Characteristics: *Mario F. Arenas*[1]; Viola L. Acoff[1]; [1]University of Alabama, Metall. and Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487 USA

Gamma titanium aluminides are currently receiving considerable attention due to their excellent properties such as low density, high-temperature strength, superior stiffness and acceptable corrosion resistance. A widespread utilization of these alloys will require the ability to weld these materials to themselves and to other materials. Previous investigations have demonstrated the feasibility of using gas tungsten arc welding for joining ×-TiAl. However, a systematic study addressing the effect of the welding variables on the weld pool characteristics is lacking. In this study, a series of welds using a stationary torch (spot welding) and a moving torch have been made on gamma TiAl specimens using various welding currents, arc gaps, torch speeds, and electrode tip angles. The width, depth and area of the melt zone were measured from weld sections by optical microscopy and were then evaluated as a function of welding parameter. The results of this study provide the basis for a heat transfer model analysis of the welding process.

10:35 AM

An Investigation of Microstructure in a Ti-6Al-4V Friction Stir Weld: *Mary C. Juhas*[1]; G. B. Viswanathan[1]; Larry Lehman[2]; Hamish Fraser[1]; [1]The Ohio State University, Dept of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210; [2]Edison Welding Institute, Columbus, OH 43221

The friction stir welding process has recently been extended from lower temperature materials such as Al and Cu alloys to higher temperature structural alloys such as steels, nickel base alloys and titanium alloys. The challenges of producing high quality joints in these materials far exceed those encountered in the routine production of aluminum alloy welds. These process challenges will be reduced and possibly overcome through the diligent development of an appropriate tool material, but also through of a fundamental understanding of the micromechanisms of deformation during welding. The focus of this work was to characterize the microstructures that evolve in Ti-6Al-4V friction stir welds with the aim of determining the underlying basic mechanisms associated with joining. The various regions of the welds have been characterized using scanning and transmission electron microscopy. A variety of microstructures result, not only between the stir zone and base material, but also within the stir zone of some welds. It is intended that these microstructural observations can be related to process variables with the eventual aim of optimizing the mechanical properties of the weld.

11:00 AM

Microstructural Evolution in Aluminum Alloy Friction Stir Welds: *Mary C. Juhas*[1]; Peter C. Collins[1]; Larry Lehman[2]; Hamish Fraser[1]; [1]The Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]Edison Welding Institute, Columbus, OH 43221 USA

Friction stir welding of aluminum alloys has reached a high level of sophistication in the relatively short period of time since its invention in 1991. Although high quality welds are produced routinely in a variety of industrial sectors, it is unclear that the underlying mechanisms of microstructural evolution are completely understood in many cases. The extension of the friction stir welding process to complex joint designs, other low temperature materials and higher temperature alloys will require a basic knowledge of how the microstructure evolves as a function of the thermal excursions and the ranges and rates of deformation experienced during processing. The present study involves an in-depth analysis of microstructural features in the stir zone, thermomechanically-affected zone, and heat affected zone including the regions just ahead of the tool. Characterization techniques include optical microscopy and scanning and transmission electron microscopy (SEM, TEM). A full microhardness map has been generated to compare the observed microstructures with an estimate of their corresponding mechanical properties. A dual beam focused ion beam (FIB) has been used to very precisely slice subsized TEM foils (~10 μm x 10 μm) from site-specific locations corresponding to features within the microhardness map.

11:25 AM

Friction Stir Weld Edge Seal for Corrosion Prevention in 7475-T761 Lap Joint Structures: *Edmond R. Coletta*[1]; William J. Arbegast[1]; Bryan A. Jensen[1]; [1]Lockheed Martin Space Systems Company-Michoud Operations, Prog. and Techn. Dev., P.O. Box 29304, New Orleans, LA 70189 USA

Friction stir welding (FSW) of aluminum alloys is being considered as a viable replacement for riveting in aerospace and aircraft structures. Many of the possible rivet replacement applications involve lap joint configurations, which are readily joined with the proper FSW process. However, once these lap joint structures are welded a tight interface remains on either side of the weld. The FSW Edge Seal eliminates this free surface and the likelihood of crevice corrosion. This study examines the effects of pin tool orientation and process parameters upon the edge seal weld quality on a AA7475-T761 lap joint. Characterization of both baseline and edge seal welds was completed through metallography, mechanical lap shear testing, and salt fog chamber corrosion testing.

11:45 AM

Process Development of Friction Stir Lap Joints in AA7075 and AA2297 Alloys: *Zhixian Li*[1]; William J. Arbegast[1]; Anthony Reynolds[2]; Kumar V. Jata[3]; [1]Lockheed Martin Space Systems Company, Prog. and Techn. Dev., Michoud Oper., P.O. Box 29304, New Orleans, LA 70189; [2]University of South Carolina, Mech. Eng. Dept., 300 South Main St., Columbia, SC 29208; [3]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., Wright-Patterson AFB, OH 45433

Friction stir welding (FSW) has shown potential applications in airframe structures to replace riveted joints by taking advantage of various FSW joint configurations such as lap joints and fillet joints. In the present study, both AA7075-T6 sheet and AA2297-T8 sheet were lap-joined via friction stir welding using different pin tool configurations and lengths. Effects of pin tools and processing parameters on joint microstructures and mechanical properties will be presented. The different responses of AA7075 and AA2297 to FSW lap joining will be addressed as well.

Magnesium Technology 2001: Physical Metallurgy

Sponsored by: TMS: Light Metals Division, Magnesium Committee and Reactive Metals Committee; International Magnesium Association; and ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee

Program Organizers: John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA; David Creber, Alcan International Ltd, Kingston R&D Center, Kingston, Ontario K7L 5L9 Canada; Russell H. Jones, Battelle Pacific Northwest National Laboratory, Richland, WA 99352 USA; Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Ramaswami Neelameggham, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Eric A. Nyberg, Pacific Northwest National Laboratory, Materials Processing Group, Richland, WA 99352 USA; Mihriban O. Pekguleryz, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Kevin Watson, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H7R 1G5 Canada

Wednesday AM	Room: 203-205
February 14, 2001	Location: Ernest N. Morial Convention Center

Session Chair: Mihriban Pekguleryuz, Noranda Technology Centre, Pointe Claire, Quebec, H9R 1G5 Canada

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8:30 AM

Application of Digital Image Analysis Technique to Quantify

Cast Microstructure of Mg Alloys: *Arunkumar Balasundaram*[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Mats. Sci. and Eng., 771, Ferst Dr., Atlanta, GA 30332 USA

Die cast Magnesium alloys are being used to manufacture automotive components because of their unique combination of good strength and low density. These alloys contain both shrinkage and gas microporosities. Characterization of shrinkage and gas microporosities is expected to be useful to understand the processing-properties-microstructure correlations. However, the existing Image Analysis techniques do not permit a separate quantification and characterization of the shrinkage and gas microporosities. In this contribution, a unique Digital Image Analysis technique has been developed which can now separately quantify and characterize the shrinkage and gas microporosities in Magnesium alloys. The resulting data has been correlated with their mechanical properties.

8:55 AM

Ductility and the Skin Effect in High Pressure Die Cast Mg-al Alloys: *A. L. Bowles*[1]; J. R. Griffiths[2]; C. J. Davidson[2]; [1]The University of Queensland, Cooperative Res. Cen. for Cast Metals Manuf. (CAST), Dept. of Min., Mine. and Mats. Eng., Brisbane, Australia 4072; [2]CSIRO Manufacturing Science and Technology, P.O. Box 883 Kenmore, Australia 4069

The prominent microstructural features of high pressure die cast Mg-Al alloys are presented. The high pressure die cast Mg-Al alloys examined typically exhibit a finer grain size, a region near the casting surface termed the *skin* that has a finer structure, a distinctive band of porosity below the skin and a central region with a high fraction of large primary magnesium dendrites. Direct measurement of the as-cast grain size of AZ91D and AM60B has been obtained by EBSD analysis. The depth of the skin region in these alloys has been quantified by several techniques: microhardness traverses; the location of porosity bands; and the variation of the fraction of large primary a grains. The use of these features to define a skin depth is discussed.

9:20 AM

Microstructure and Microchemistry of Creep Resistant Magnesium Alloys: *Eric A. Nyberg*[1]; Dan J. Edwards[1]; Russell H. Jones[1]; [1]Pacific Northwest National Laboratory, Mats. Res./Mat. Proc., P.O. Box 999; MSIN P8-35, Richland, WA 99352 USA

Magnesium is being considered for automotive components to reduce weight. Some of these components require creep resistance at temperatures of 150°C and above. A number of Mg alloys have been developed to meet these needs including AE42, which contains rare earth elements, and calcium containing alloys such as Al-Zn-Ca and AM60+Ca. Detailed explanations for the creep resistance are not available, although reduced grain boundary sliding has been mentioned. This study was undertaken to correlate the microchemistry and microstructure of these creep resistant alloys with the goal of determining the role of these additions. A high-resolution analytical TEM (JEOL 2010F) with 200 keV accelerating voltage was used for these evaluations. A JEOL 840 SEM was also used. The alloys examined included die cast AE42 and ZAC 8506 and semi-solid molded ZAC8506. AZ91D was used for comparison. The microchemistry and microstructure were correlated with the compressive creep and the bolt load retention properties at 175°C.

9:45 AM

The Relationship Between Microstructure and Creep Behavior in AE42 Magnesium Die Casting Alloy: *Bob R. Powell*[1]; Vadim Rezhets[1]; Michael P. Balogh[2]; Richard A. Waldo[2]; [1]General Motors, Res. & Dev. Center, Mats. & Proc. Lab., Mail Code 480-106-212, Warren, MI 48090-9055 USA; [2]General Motors, Res. & Dev. Cen., Chem. & Environ. Scis. Lab., Mail Code 480-106-320, Warren, MI 48090-9055 USA

Microstructural analysis of die cast AE42 reveals a correlation between microstructure and creep strength. A lamellar phase $Al_{11}E_3$ which dominates the interdendritic microstructure of the alloy partly decomposes above 150°C into Al_2E and Al (forming $Mg_{17}Al_{12}$). The creep strength decreases sharply with these phase changes. A mechanism for the decrease in creep strength of AE42 is proposed whereby reduced presence of lamellar $Al_{11}E_3$ and/or the presence of $Mg_{17}Al_{12}$ contribute to the observed poor creep strength at the higher temperature. The increased solubility of Al in Mg at higher temperature may also promote the decomposition of $Al_{11}E_3$.

10:10 AM Break

10:20 AM

Beta Phase (Mg17Al12) Precipitation Kinetics of Magnesium Alloys: *S. R. Agnew*[1]; E. A. Payzant[1]; S. Viswanathan[1]; [1]Oak Ridge National Laboratory, Mets. and Ceram. Div., Oak Ridge, TN 37831-6115 USA

High temperature X-ray diffraction has been used to identify the kinetics of beta phase (Mg17Al12) precipitation kinetics in the primary die cast magnesium alloys, AZ91D and AM60B. The volume fraction of beta phase has been measured as a function of time and temperature (225°C to 325°C). This information could provide useful input for understanding aging or elevated temperature service behaviors. In addition to phase composition changes, the lattice constant of the primary magnesium phase is shown to dilate as aluminum precipitates out of solution. This effect has been identified as a possible explanation for the tension/compression asymmetry observed in the creep behavior of these alloys. The volume fraction of beta phase before and after creep testing in tension and compression was also measured to verify this hypothesis.

10:45 AM

TEM Study of Zn Influence on Precipitation in Mg-Al-Zn Alloys: *Menachem Bamberger*[1]; Ludmila Shepeleva[1]; Evgeny Manov[1]; [1]Technion, Mats. Eng. Dept., Haifa 32000 Israel

Magnesium alloys have attracted increasing interest as structural materials for automotive and airspace applications thanks to their low density. The two key factors, which limit their usage, are relatively low mechanical properties and poor corrosion resistance. The first one can be solved by using precipitation hardenable alloys, such as Zn rich Mg-alloys. The microstructure of Mg-Al-Zn based alloys containing 0.7%, 5.5%, and 6.5%Zn in as-cast state and after heat treatment at 1500°C for 25 to 2000h was investigated using TEM. The matrix/precipitate orientation relationships, sizes, shapes and the number of precipitates per unit volume are described. The transition from discontinuous lamellas to spherical precipitates was observed. The relationship between the continuous precipitate morphology and the hardness is discussed.

11:10 AM

Origins of Variability in the Mechanical Properties of AM60 Magnesium Alloy Castings: *Arun M. Gokhale*[1]; Gautam R. Patel[1]; [1]Georgia Institute of Technology, Sch. of Mats. Sci. and Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Successful applications of cast magnesium alloys for structural applications require production of castings that exhibit reproducible mechanical and environmental response. Therefore, a thorough understanding of correlations between variability in the microstructure and the mechanical response of cast magnesium alloy components is of interest. In the present study, tensile tests were performed on the specimens machined from the same region of a group of die cast AM60 alloy components. In this group of specimens, the room temperature ductility varied from 4% to 13%, and the tensile strength varies from 25 to 34 ksi. The microstructure contains defects such as internal oxide films, shrinkage porosity, and gas (air) porosity. These defects affect the fracture path and contribute significantly to the variability in strength and ductility. The total area fraction of defects and discontinuities on the fracture surfaces of the tensile test specimens has been quantitatively correlated with the ductility and tensile strength.

11:35 AM

Experimental and Computational Study of Bolt Load Retention Behavior of Magnesium Alloy AM60B: Cornelius Temmel[1]; Ken C. Liu[1]; Sean R. Agnew[1]; Adrian S. Sabau[1]; Qingyou Han[1]; *Srinath Viswanathan*[1]; [1]Oak Ridge National Laboratory, Mets. and Ceram. Div., Oak Ridge, TN 37831-6083 USA

An important requirement for the use of magnesium automotive powertrain housings is adequate bolt load retention (BLR). An experimental set-up using an instrumented bolt was used to test the BLR behavior of AM60B alloy at 150°C. In addition, creep test data collected at 150°C over a range of stresses (20-80 MPa) were

used to develop constitutive models of creep behavior. Simulations of the BLR test were performed using the finite element analysis software Abaqus. The impacts of the various constitutive model parameters, including transient and steady state regimes, and the effect of thermal expansion during the heat-up, are evaluated and compared with the experimental BLR data.

Materials Processing Fundamentals V

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Process Fundamentals Committee, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: P. N. Anyalebechi, ALCOA, Ingot & Solidification Platform, Alcoa Center, PA 15069-0001 USA; A. Powell, MIT

Wednesday AM Room: 218
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Patrick R. Taylor, University of Tennessee, Moscow, ID 83843-2461 USA

8:30 AM

The Effect of Phase Decomposition in Quaternary Chromite Spinel on the Extraction of Chromium as Sodium Chromate during Oxidative Alkali Roasting: *Vilas D. Tathavadkar*[1]; M. P. Antony[1]; Animesh Jha[1]; [1]University of Leeds, Dept. of Mats., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK

Natural chromite mineral is a solid solution of spinel end members namely: chromite (FeCr2O4), hercynite (FeAl2O4), magnetite (Fe3O4), magnesio-chromite (MgCr2O4), (MgAl2O4), and magnesio-ferrite (MgFe2O4). The phase transformation in chromite minerals under the oxidising conditions during soda-ash roasting plays an important role. The conversion efficiency of chromium (Cr3+) to a water-soluble sodium chromate (Cr6+) salt depends upon the decomposition kinetics of the spinel phase. In this paper, we have investigated the chromite phase equilibria during alkali roasting and its effect on chromium conversion. The thermogravimetric (TGA) and roasting experiments of chromite mineral were carried out. The samples collected at various interval/temperature during experiment were systematically investigated in view of phase equilibria using X-ray diffraction analysis and electron microscopy techniques. A separate investigation was also carried out to study the phase changes in chromite mineral in same heating cycle in the absence of sodium carbonate. The thermodynamics of phase transformation has also been discussed using sub-regular solution model of Sack and Ghiorso. The effect of the oxygen partial pressure and temperature on the phase constituents of the chromite during roasting has been discussed. The microstructural changes were examined by SEM, and the changes in elemental compositions of different phases were analysed by electron probe micro analyser (EPMA). It is evident from the experimental results that spinel phase in chromite undergoes a spinodal decomposition under the influence of oxygen partial pressure and temperatures. The cation vacancies generated during this transformation helps chromium to diffuse towards the reaction interface during roasting. The phase transformation in spinel phase also increases the reactivity of chromite, and the roasting reaction starts at an early stage as compared to the decomposition reaction taking place in an argon atmosphere.

8:55 AM

On-Line Monitoring of Reduction Processes for Improved Control of Iron- and Steel-Making: *Cheryl Su-Lean Lim*[1]; Brian David Sowerby[1]; Stephen Rainey[1]; [1]Commonwealth Scientific & Industrial Research Organisation, Dept. of Mins., Private Mail Bag 5, Menai, NSW 2234 Australia

New technologies such as direct reduction and smelting reduction processes are increasingly being used in iron- and steel-making in addition to conventional blast furnace technology. Critical parameters for control of these processes include the degree of metallisation, and carbon and silica levels. On-line monitoring of these parameters is difficult because measurements often need to be made on hot ores (at or higher than ~1000°C) in circumstances where representative sample streams are not readily accessible. A nuclear analyser has been developed which provides continuous, non-intrusive, real-time measurements of iron, oxygen, carbon and silicon levels in hot ores undergoing reduction. The analyser has been tested in a plant using a direct smelting process; accuracies of 1% relative, 0.5 wt.% (25% relative) and 1.0 wt.% (9% relative) were obtained for the degree of metallisation, carbon and silica respectively. The underlying method, capabilities and potential applications of the analyser will be discussed.

9:20 AM

In Situ Synthesis of CaZrO3/MgO Porous Composites with 3-D Network Structure: *Yoshikazu Suzuki*[1]; Peter E.D. Morgan[2]; Tatsuki Ohji[1]; [1]National Industrial Research Institute of Nagoya, Synergy Ceram. Lab., 2268-1, Simo-shidami Moriyama-ku, Nagoya, Aichi 463-8687 Japan; [2]Rockwell Science Center, Thousand Oaks, CA 91360 USA

By using reactive sintering of highly-pure natural dolomite (CaMg(CO3)2) and synthesized zirconia mixed powders with LiF additive, porous CaZrO3/MgO composites with three-dimensional network structure have been successfully synthesized. Equimolar mixture of dolomite and zirconia powders doped with 0.5 wt% LiF was cold isostatically pressed at 200 MPa and sintered at 1100-1400°C for 2 h in air. Because of the liquid formation via LiF doping, strong necks were formed between constituent particles before the end of pyrolysis of dolomite, which resulted in the formation of 3-D network structure. During and after the formation of network structure, CO2 evaporated to form homogeneous open-pore structure. The pore-size distribution was very narrow (with pore size about 1 micron), and the porosity was easily controllable (e.g. ~40-60%) by changing the sintering temperature. The porous composites can be applied as filter materials with good structural and chemical stability at high temperatures.

9:45 AM

An Investigation on the Carbothermic Reduction of Arsenic and Antimony Bearing Chalcopyrite Ore: *Maria Lucelinda Ferreira Alcântara da Cunha*[1]; Animesh Jha[2]; [1]New University of Lisbon, CENIMAT, Dept. of Mats. Sci., Fac. of Sci. and Tech., Caparica 2825-114 Portugal; [2]University of Leeds, Dept. of Mats., Clarendon Rd., Sch. of Proc., Environ. and Mats. Eng., Leeds LS2 9JT UK

The lack of control of As and Sb compounds from copper smelters and converters has rendered some chalcopyrite containing arsenopyrite, stibnite and tetrahedrite minerals unusable for metal extraction. In the present investigation, we have studied the distribution of arsenic and antimony between the metal and the matte phase during the carbothermic reduction of Portuguese chalcopyrite concentrate ore (As 0.19 wt.%, Sb 0.04 wt.%). The advantage of carbothermic reduction reaction is to manipulate the redox potential to preferentially segregate these two elements either into metallic phase or in the matte phase. The reduction reaction has been studied between 850°C and 1100°C, and the phase formed have identified by X-ray powder diffraction and scanning electron microscopy technique. The distribution of As and Sb in the metal and matte phase is also studied as a function of lime and calcium sulphate concentration of the pellet over the above temperature range. The importance of this research for using recycled matte, lean and rich minerals with As and Sb is also explained.

10:10 AM Break

10:20 AM

Behaviour of Cu2O Particles in Copper Smelting Waste Heat Boiler Conditions: *Esa J. Peuraniemi*[1]; [1]Helsinki University of Technology, Lab. of Mats. Proc., P.O. Box 6200, Espoo FIN-02015 Finland

Primary copper production using suspension smelting techniques like Outokumpu Flash Smelting always causes some dust formation. Flue dust particles are formed when sulphide feed material reacts violently with oxygen inducing particle disintegration and

total desulphurisation of fragments. In the process, flue dust follows the off-gases to the waste heat boiler (WHB) where its sulphation takes place because of prevailing conditions. Sulphation releases heat and causes also a drop in particle melting temperature and further, particle sticking to the walls and heat exchange banks. Sulphation of synthetic Cu2O particles were examined using a laboratory scale fluid-bed reactor. This study aims to a better understanding of flue dust behaviour in a WHB and, thus, to a better operational practise and design of a WHB. The effect of pSO2/pO2 -ratio and temperature on the sulphation kinetics were tested. Standard chemical analysis, light optical and scanning electron microscopy with energy dispersive spectroscopy were used to examine the samples.

10:45 AM

Effect of Converting Slag Recycling into Teniente Converter on Copper Losses: *Andrzej Warczok*[3]; Gabriel Riveros[1]; Roberto Mackay[2]; Domingo Cordero[2]; Gerardo Alvear[3]; [1]Universidad de Chile, Dept. de Minas, Av. Tupper 2069, Santiago Casilla 2777 Chile; [2]CODELCO, El Teniente, Millan 1040, Rancagua, Chile; [3]Instituto de Innovación en Minería y Metalurgia S.A., Av. Del Parque 4980, Santiago, Chile

Intensive smelting of copper concentrate in Teniente Converter (TC) leads to the production of high grade matte and highly oxidized slag. Smelting slag is processed in Teniente vascular slag cleaning furnace. The slag composition and forms of copper affect the slag cleaning process and final copper content. Converting of high grade matte in Pierce-Smith converter faces some difficulties in proper slagging of iron oxides. Overblowing in copper making stage allows for slag liquidation but leads to high content of copper oxide in converter slag. Converter slag recycling into TC results in the increase of the participation of oxidic copper. Separate treatment of converter slag and its impact on the forms of copper in TC slag has been analyzed. Thermodynamic properties of a system and kinetic limitations pointed out the negative effect of converter slag recycling on the forms of copper and copper recovery in slag cleaning stage. Experimental results of slag cleaning tests in a crucible scale as well as microscopic examination of laboratory and industrial slag samples supported the above analysis and permitted to formulate recommendations of separate processing of converter slag. Keywords: slag, magnetite, electric furnace.

11:10 AM

Leaching Study of a Zinc Concentrate Ore by Factorial Design: Luisa Maria Abrantes[1]; Monica Luisa Afonso[1]; *Maria Lucelinda Cunha*[2]; [1]ICAT Universidade de Lisboa, Faculdade de Ciências, Campo Grande, Lisboa 1749-016 Portugal; [2]Cenimat, Dept. of Mats. Sci., Fac. of Sci. and Tech./New Univ. of Lisbon, Caparica 2825-144 Portugal

Environmental control is becoming more and more stringent. As conventional process for zinc production are pollutant routes, alternative processes have become an important subject of investigation. In this regard, the leaching processes have aroused a considerable interest, namely the electrochemically induced approach. The behavior of a zinc concentrate ore on the leaching process is influenced by several parameters. Chloride concentration and pH of the leaching solution, time and temperature of reaction and the applied potential were studied in order to evaluate their influence on the zinc recovery. The efficiency of the leaching process for zinc extraction is performed by factorial design. The experiments were carried out by 2^3 and 2^2 factorial designs of the factors. The results show that all the factors, except the time of reaction have a significant influence on the optimization process, within each experimental region.

Materials & Processes for Submicron Technology: Device Characterization and Integration

Sponsored by: Electronic, Magnetic & Photonic Materials Division, ASM International: Materials Science Critical Technology Sector, Thin Films & Interfaces Committee

Program Organizers: N. (Ravi) M. Ravindra, New Jersey Institute of Technology, University Heights, Newark, NJ 07102-1982 USA; Mark Anthony, University of South Florida, College of Eng., Tampa, FL 33620 USA; Ashok Kumar, University of South Florida, Dept. of Mech. Eng., Tampa, FL 33620 USA; Sailesh Merchant, Lucent Technologies, Orlando, FL 32819 USA; Mahesh Sanganeria, Novellus Systems, Inc., San Jose, CA 95134 USA

Wednesday AM — Room: 226
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Sailesh M. Merchant, Lucent Technologies-Bell Laboratories, 9333 S. John Young Pkway, Orlando, FL 32819-8698 USA; Dentcho Ivanov, Microelectronics Res. Cen., New Jersey Inst. of Techn., Newark, NJ 07102 USA

8:30 AM Invited

Local Material Chemistry, Mechanical Properties and Reliability of Integrated Circuits 1: Correlation Between Burn-in Fallout Rates and the Mechanical Properties of Final Passivation: *Yaw S. Obeng*[1]; Irene Li[1]; Joko Seputro[1]; Jennifer A. Jusczak[1]; Jonathon Lobbins[1]; Seung H. Kang[1]; Jia Sheng Huang[1]; Anthony S. Oates[1]; [1]Bell Laboratories, Lucent Technologies, 9333 South John Young Parkway, Orlando, FL 32819 USA

The dielectrics used in IC fabrication play crucial roles in the device performance and reliability. For example, the mechanical properties of the dielectrics control stress evolution in encapsulated metal lines. In the talk, we use a variety of analytical techniques (Nanoindentation, FTIR, Optical Spectrometry, Stress Temperature Hysterisis) to examine the relationships between local chemistry and the mechanical properties of the dielectric films. We will also discuss the interactions between the dielectrics and the metal stacks. The consequences of the interaction between the dielectrics and the metal stacks on the reliability of the IC devices will be illustrated by the correlation between lot rejection rate at burn-in and the dynamic mechanical properties of the final passivation(CAPS) used. Specifically, there is strong dependence of lot rejection rates (LRR) on the effective Young's modulus of the CAPS; the LRR increases with Young's modulus.

9:00 AM Invited

NMOSFET and PMOSFET Characteristics of Hafnium Doped SiO2 Gate Dielectrics: Veena Misra[1]; Manoj Kulkarni[1]; [1]North Carolina State University, Dept. of Elect. Eng., Box 7911, Raleigh, NC 27695 USA

High-K gate dielectrics are needed to continue the successful downscaling of Si CMOS devices beyond 70nm. Recently, a lot of attention has been directed towards Group IVB (Hf, Zr) metal oxides and metal silicates, owing to their thermodynamic stability and large barrier heights. In this work, we present the interfacial characteristics of HfSixOy films via NMOS and PMOS transistor characterization. HfSixOy films were deposited at room temperature using reactive sputtering of HfSi2 target in O2 ambient. The Hf content of these films was ~3% as determined from XPS. These Hf doped SiO2 films were studied as gate dielectric and were found to be stable on Si. These films produced a bi-layer structure with an effective dielectric constant of ~8. The bottom layer in this stack was not pure SiO2. Excellent C-V, low leakage current and negligible hysteresis was observed. Two-level charge pumping current provided an average Dit value of 4.35x1010/eV/cm2. Mobilities, although slightly lower than the universal SiO2 mobility model, were higher than those reported on other high-K materials. The films remained amorphous up to 900°C indicating their compatibility with conventional processing. In conclusion, these medium-K dielectric materials offer a technologically relevant gate-stack for insertion into 50nm CMOS devices.

9:30 AM

Combined Low-Frequency Noise and Resistance Measurements for Void Extraction in Deep-Submicrometer Interconnects: *Lip Wei Chu*[1]; Wai Kin Chim[1]; Kin Leong Pey[2]; [1]National University of Singapore, Elect. Eng., Center for Integrated Circuit Failure Analysis and Reliability (CICFAR), Blk E3 #-04-08, 4 Engineering Dr. 3, Singapore 117576 Singapore; [2]Chartered Semiconductor Manufacturing, Ltd., Fab 3 Yield Eng., 60 Woodlands Industrial Park D, St. 2, Singapore 738406 Singapore

Electromigration stress can give rise to voids that increase the resistance and localised thermal stress in interconnects. Estimation of the extent of voiding can provide information on the material quality and the amount of degradation that has resulted from the electrical stress. In this paper, a model is proposed which can be used to estimate the effective void volume in deep-submicrometer interconnects. The model uses a combination of low-frequency noise and resistance measurements, and also considers the thermal coefficient of resistance (TCR) in calculating the change in resistance of the interconnect line. A deconvolution scheme was employed to extract the 1/f noise component from the noise measurements to improve the accuracy of the extraction algorithm. To verify the accuracy of the model, the focused ion beam (FIB) technique was used to mill holes (to simulate voids) of known dimensions. The model was further applied to an electromigration stress study of aluminum and copper interconnects as a means of testing its validity for stress-induced voids. The proposed technique is a useful reliability tool for void detection in deep-submicrometer interconnects.

9:50 AM

Electromigration Characterictics of Multilevel Dual Damascene Electroplated Copper Interconnects: *H. S. Rathore*[1]; D. B. Nguyen[1]; Alexander Swinton[1]; [1]IBM Micrelectronics, 2070 Route 52, East Fishkill Facility, Hopewell Jct., NY 12533 USA

Copper metallization has been successfully implemented in sub-quarter micron high performance products. Copper has been chosen for the interconnect metallization due to its enhanced electromigration and lower resistivity. In this paper we will discuss the key reliability failure mechanisms; electromigration, its kinetics (current and temperature acceleration) and stress migration. The technology assessed was a CMOS base technology with up to six copper wiring levels built at a minimum pitch of 0.63 um. The copper interconnects were deposited using three different techniques; Chemical Vapor Deposition (CVD), Physical Vapor Deposition (PVD), and Electro-Plating (EP). The interconnects were patterned using a dual damascene process. The copper lines were encapsulated by conductive metallic liners and insulated by silicon nitride. The sample line length was 400.0 lm and interconnection to higher and lower levels by dual damascene vias. We will show that the process of choice is electroplated copper with experimentally determined activation energy of 1.0 eV (DH) and a current acceleration of 1.1 (n).

10:20 AM Break

10:40 AM Invited

Anomalous Scaling Effect of Tungsten/Titanium Nitride/Titanium to Silicon Electrical Contact Resistance for Sub-Quarter Micron Electronic Devices: *Jun-Ho Choy*[1]; Young-Soo Kim[1]; Tae-Keun Hwang[1]; Duk-Hee Lee[1]; Jae-Hyung Kim[1]; Jin-Tae Choi[1]; Sang-Bum Han[1]; [1]Hyundai Electronic Industries, Memory R&D Div., 1 Hyangjung-dong, Hungduk-gu, Cheongju, Choongbuk 361-725 Korea

This paper reports the anomalous scaling effect of tungsten/titanium nitride/titanium to n+ and to p+ silicon electrical contact resistance used in the DRAM(Dynamic Random Access Memory) devices, upon post heat treatment following rapid thermal silicidation annealing. The high temperature post heat treatment, accompanied by the memory cell capacitor process, may become necessary as the minimum feature size shrinks to 0.18um and below, in order to avoid high aspect ratio contact hole process. A test vehicle with various test structures was fabricated using sub-quarter micron CMOS technology. In the process, sputter deposition using ionized metal plasma was employed for titanium deposition. The electrical measurements on contacts of sizes ranging from 0.46 to 0.18um reveal that the increase in the resistance upon post heat treatment becomes larger as the contact size decreases. TEM results on these contacts show that the silicide film agglomeration proceeds more severely as the contact size decreases. The analysis reveal that the silicide film agglomeration, rather than the dopant deactivation or profile change, plays a major role in increasing the contact resistance. Based on the morphology observed, a numerical simulation on the shape evolution of the silicide film is performed in order to explain the size-dependent degradation of the contact resistance. The numerical results show that the poor coverage of the film at the edge for small contacts accelerates the reduction rate of contact area. In addition, the contact size is found not to have considerable effect on the reduction rate, only affecting the area at equilibrium.

11:10 AM Invited

Process Integration of W Local Interconnect for Sub-Quarter Micron VLSI: *Weidan Li*[1]; Zhihai Wang[1]; Wilbur Catabay[1]; [1]LSI Logic Corporation, Wafer Proc. R&D Div. , 3115 Alfred St., Santa Clara, CA 95054 USA

Tungsten local interconnect (W LI) is now widely used in the advanced VLSI to serve as the interface between transistors and multi-layer Cu wires. Area reduction of ~10% was observed comparing with the conventional W contact process. Since W LI is physically very close to transistors, it may cause significant impact on transistors. In order to successfully integrate W LI into the VLSI technology, new processes and process sequences have to be introduced. In this paper, we discuss about the films used in W LI layer and their impact on transistors, the processes to deposit and pattern the films, and the other process integration issues. The transistor performance and reliability, interconnect performance, and the process robustness will be used as matrix for the disccussion.

Modeling of High Temperature Alloys: Process Modelling

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Shailesh Patel, Special Metals, Huntington, WV 25705-1771 USA; Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA

Wednesday AM — Room: 221
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: David Furrer, Ladish, Research, Cudahy, WI 53110 USA

8:30 AM Opening Remarks:

Lesh Patel; Special Metals Corporation

8:35 AM

Model Integration of Hammer Forging of Superalloys: *Gangshu Shen*[1]; [1]Ladish Co., Inc., 5481 South Packard Ave., Cudahy, WI 53110-8902 USA

Computer controlled hammer technology can provide repeatable hammer blows and taylor the energy input into the workpiece during each of the hammer blows. Additionally, the computer program for each part runs a reproduceably engineered, pre-programmed sequence each time and records actual velocity, energy and operation timing for each forged component. This new capability in hammer forging allows process and microstructural model integration into the process design cycle. In this study, hammer forging of selected superalloys was run in the computer controlled hammer. The process and microstructure modeling of the hammer forging was performed. The results indicate (1) hammer forging can be run under accurate reproducible control; (2) the hammer forging process can be simulated accurately using DEFORM; (3) the microstructure of hammer forged parts can be accurately predicted.

9:00 AM

Numeric Simulation of Microstructural Evolution During Open Die Cog Forging: *Bruce Fergus Antolovich*[1]; [1]Special Metals

Corporation, 4317 Middle Settlement Rd., New Hartford, NY 13413 USA

Recent advances in finite element code efficiency combined with the well established continued computer speed increases have resulted in industrially useful finite element analysis applications to modelize the complete thermo-mechanical history of an ingot undergoing cog forging. In conjunction with appropriate recrystallization models, numerical simulation of grain size evolution has become a very useful tool. Accurate microstructural modeling has served to 1) improve product quality, 2) reduce the time required for and cost of industrial trials and 3) develop more efficient conversion programs. Recrystallization is a complex phenomenon in which final grain size distributions depend primarily upon material, temperature, hold time, strain, strain rate and initial grain size distribution. Regardless of which particular set of recrystallization behavior laws is chosen, a significant amount of testing is required to determine recrystallization constants. Specialized specimens such as "double-cone" compression specimens can significantly reduce the required amount of testing. Examples of rotary forge cogging are used to illustrate the accuracy of grain size evolution prediction for INCO 718 being forged from a 355mm octagon to a 266 round cross section. Furthermore, techniques to improve grain size control in the absence of detailed recrystallization data are illustrated with practical examples of parametric variation of available forge control functions.

9:25 AM

Thermal Process Modeling of P/M Superalloys: *Keh-Minn Chang*[1]; Jian Mao[1]; Dave Furrer[2]; Suhas Vaze[3]; [1]West Virginia University, Mech. and Aeros. Eng., P.O. Box 6106, Morgantown, WV 26506 USA; [2]Ladish Co., Inc., Cudahy, WI; [3]Concurrent Technologies Corporation, Johnstown, PA USA

The excellent high-temperature strength of P/M superalloys is derived from the precipitation of ordered and coherent Ni3Al phase from Ni solid solution matrix. The mechanical properties of P/M superalloy parts are strongly dependent upon the microstructural characteristics (the size, the density, and the distribution) of precipitates during heat treatments. The quenching step after solution heat treatment is particular critical, because the cooling rate must be fast enough to avoid the formation of extensive cooling precipitates. To optimize the quenching step without expansive trials, thermal process modeling would be the best approach to determine heat treatment parameters. A cooperative research effort has been performed through constitutive modeling of on-cooling microstructures and properties of selected P/M superalloys. A generic physical metallurgy based on microstructural model is developed, which allows the prediction of on-cooling microstructures of precipitation at different cooling rates. In couple with the thermal profile simulation, this constitutive modeling can provide great help to forging vendors on optimizing thermal process parameters for specific requirements of a given P/M superalloy component.

9:50 AM Break

10:10 AM

Virtual Production Supply Chains for Gas Turbine Disc Manufacture: *Chris A. Dandre*[1]; [1]University of Wales Swansea, Singleton Park, Swansea, UK

Engine manufacturers are encouraging the development of virtual production supply chains for the manufacture of gas turbine disc forgings. Such capabilities will enable efficient microstructural and mechanical property optimisation throughout the thermomechanical processing route in support of the "cradle-to-grave" design philosophy. A major advance in this direction will be the availability of datafiles that contain a complete description of the evolved microstructure throughout the workpiece. Furthermore, the ability to transfer such datafiles from one simulation to the next will form the basis of virtual production supply chains. However, there are many obstacles that prevent immediate implementation of virtual production supply chains. This paper addresses these limitations in view of recent developments in the field of microstructural process modelling. Furthermore, new discoveries are presented that highlight the need to develop virtual production supply chains.

10:35 AM

High Temperature Forming Process Simulation: *Dave Lambert*[1]; John Walters[1]; Christian Fischer[1]; [1]Scientific Forming Technologies Corporation, 5038 Reed, Columbus, OH 43220-2514 USA

Hot forming processes are complex in nature, involving adiabatic heating, die chill and microstructural changes. In addition, hot forming processes often comprise numerous operations in the process manufacture of high quality, critical service components. In many cases, interactions between casting, ingot breakdown, extrusion, closed-die forging, heat treatment and machining can significantly affect the cost-effective production of near-net shape components. While it may be difficult and time intensive to develop a complex forging, extrusion or heat treatment regime using trial and error, it is almost impossible to understand the thermal and metallurgical interactions taking place during and between multiple operations, without powerful process modeling tools. For a number of years now, process modeling has been developed for both deformation and heat treatment analyses. Presently, the application of computer simulation is extremely widespread in the design and development of hot forming processes. Most major companies are now routinely involved in process simulation and its usage is continuing to increase.

11:00 AM

Isothermal Forging Process Design of PM Rene 95 Alloy Component Using Coupled Thermoviscoplastic FEM Analysis: *Maicang Zhang*[1]; Lina Zhang[1]; Jianxin Dong[1]; *Xishan Xie*[1]; [1]University of Science and Technology Beijing, High Temp. Mats. Res. Labs., Beijing, China

For PM superalloy components, hot isostatic pressing and isothermal forging are the practical working processes in aviation industry during the past years. However, isothermal forging is a rather expensive and complicated working procedure, especially the design of the isothermal forming dies and the modifications of the forming equipment. As a kind of useful CAD/CAM tool, the development of numerical simulation technique makes the hot working processes design more convenient and less expensive, avoiding a large quantity of trial and error designs for technicians or engineers in manufacturing processes. In this paper, isothermal forging process for PM Rene 95 alloy component was designed by means of coupled thermoviscoplastic FEM analysis. The constitutive relationship for dense PM Rene 95 alloy was established phenomenologically based on isothermal compression tests at constant strain rate. Two types of die materials including TZM (a Mo-Ti-Zr-C system alloy) and K21 (a nickel base casting superalloy) were considered in the simulating process. For each kind of die materials, two cases of isothermal forging processes (open die and closed die forming) were selected to evaluate the process feasibility of isothermal forming for PM Rene 95 alloy disk. During the forming process, the crossbeam velocity of hydraulic press varied as a function of instantaneous reference height of workpiece in order to make the workpiece deform at a pseudo-constant strain rate. The prediction details like the distribution of equivalent flow stress, equivalent strain and instantaneous strain rate in deformed workpiece, the temperature in both die and workpiece were all presented. The results are as follows: For die material of TZM alloy, isothermal forging at 1050º is feasible both in open-die and closed die forging. However, for die material of K21 alloy, it is not practicable even forming at 1000º because of the die failure due to heat effect of deformation. Hence, if the isothermal forging die system was worked under vacuum condition, TZM alloy is the first choice for die material. Otherwise, K21 superalloy may be selected and the die is exposed in the air and the hot die forming process, or the economical working process, may be adopted. Key words: Process design, Isothermal forging, PM Rene 95 alloy, Coupled thermoviscoplastic FEM

11:25 AM

Prediction of Interface Friction Factor using Upsetting Tests: *Palla Sivaprasad*[1]; C. H. Davies[1]; [1]Monash University, Dept. of Mats. Sci., Clayton, Victoria, Australia

In metal forming operations the interface friction between tool and work piece plays an important role in deciding the processing schedule and also the quality of the product. Generally ring compression tests are employed to predict the interface friction factor.

The test involves forging of a flat ring shaped specimen to a specified thickness strain, and estimating the interface friction factor based on the change in the internal diameter of the ring specimen and a set of calibration curves. In contrast to this, the present investigation aims to predict the interface friction factor from the bulging in a conventional upset test on a solid cylindrical specimen. In such a test, the degree of bulging observed is a function of the friction between die and work piece. The main advantage of this approach is that upset tests conducted for evaluation of flow behaviour of the material would be also used to predict the interface friction factor. Towards this end, a general-purpose large plastic strain finite element package, Marc 2000 is employed. The constant cross head speed conditions of the experiments are specified in the boundary conditions, and the effects of material properties such as work hardening, flow softening and the strain rate sensitivity on the characteristics of a simulated bulge are analysed. Calibration curves thus generated are employed to predict the interface friction factors of upset specimens under various experimental conditions. Interface friction factors are also evaluated using the ring compression test, and the results are used to validate the current approach.

Properties of Nanocrystalline Materials: Modeling and Microstructure

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Jt. Mechanical Behavior of Materials, Chemistry & Physics of Materials Committee
Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Horst W. Hahn, Techische Hchschule Damstadt, Darmstadt D-64287 Germany; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552 USA

Wednesday AM Room: 223
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Larry Kabacoff, Office of Naval Research, VA USA; Linda Horton, Oak Ridge National Laboratory, Oak Ridge, TN USA

8:30 AM Invited
A Model for the Yield Stress of Nanocrystalline Materials: *Chandra Shekhar Pande*[1]; Robert A. Masumura[1]; [1]Naval Research Laboratory, 4555 Overlook Ave. SW, Washington, DC USA

A model is proposed for the yield stress of nanocrystalline materials based upon Coble creep. Using Coble creep with a threshold stress for finer grains and conventional Hall-Petch strengthening for larger grains, an analytical relation is derived for the yield stress as function of grain size for the whole range of grain sizes from large to very small. A grain size distribution is incorporated into the analysis to account for a distribution of grain sizes occurring in most specimens. This result is compared with experimental data from Cu and NiP and shown to be in good agreement. Experimental and computational evidence for the support of the model is also discussed.

8:55 AM Invited
Grain Boundary Structure and Plasticity in Nanocrystalline FCC Metals: *Helena Van Swygenhoven*[1]; [1]Paul Scherrer Institute, GFA-ASQ, CH-5232 Villigen-PSI Switzerland

Molecular dynamics simulations of nanocrystalline Ni and Cu metals with mean grain sizes between 3 and 20 nm are performed. An analysis of the structure and energetics of the grain boundaries and triple junctions is presented using direct visualisation, energy calculations and local crystalline order. Grain boundaries are essentially similar to those found at the micron- scale, i.e similar structural units are found, providing evidence against the view of grain boundaries in nano-crystals as liquid-like interfaces. A change in deformation mechanism is observed: at the smallest grain sizes all deformation is accommodated in the grain boundaries and grain boundary sliding, a process based on mechanical and thermally activated single atomic jumps, dominates the contribution to deformation. At larger grain sizes, a combination of sliding and intragrain dislocation activity is observed. The conditions for dislocation emission, the role of the atomic structure of the grain boundaries as well as the mechanism on atomic level of grain boundary sliding are discussed and results are compared with experimental data found in literature.

9:20 AM Invited
Atomistic Simulations of Dislocation-Interface Interactions in the Cu-Ni Multilayer System: *Satish I. Rao*[1]; [1]UES, Inc., 4401, Dayton-Xenia Rd., Dayton, OH 45432 USA

Experimental results show that a nanolayered composite structure made of two kinds of metallic materials strengthens dramatically as the layer thickness is reduced. This strengthening can be attributed, in epitaxial systems, to four kinds of dislocation-interface interactions: modulus, lattice parameter, gamma surface and slip plane mismatches between adjacent layers. The modulus mismatch produces a force between a dislocation and its image in the interface. The lattice parameter mismatch generates coherency stresses and mismatch dislocations which interact with a mobile dislocation. The gamma surface mismatch results in core energy changes as the glide dislocations approach the interface. Slip plane mismatch across the interface requires mobile dislocations in the screw orientation to cross-slip. In this manuscript the embedded atom method (EAM) is used to study atomistically, all four types of dislocation-interface interactions in the Cu-Ni multilayer system. It is shown that the blocking strength of the Cu-Ni interface due to all four types of interactions is significant and range in value from 0.004-0.05G.

9:45 AM Invited
Nanocrystalline Solids–Synthesis, Structural Features, Diffusion and Ordering: H.-E. Schaefer[1]; K. Reimann[1]; W. Straub[2]; U. Brossmann[3]; R. Würschum[4]; [1]Universität Stuttgart, Institut für Theoretische und Angewandte Physik, Pfaffenwaldring 57, 70550 Stuttgart, Germany; [2]Now: SAP AG Walldorf, Neurottstr. 16, 69190 Walldorf, Germany; [3]GKSS Forschungszentrum, Abt. Werkstoffphysik, 21502 Geesthacht, Germany [4]Forschungszentrum Karlsruhe GmbH, Institut für Nanotechnologie, Postfach 3640, 76021 Karlsruhe, Germany

The potentials for novel properties of nanocrystalline solids arise from the small crystallite size and the high number of interfaces. In the present overview we will report on atomic resolution electron microscopy (ARM), positron annihilation and tracer diffusion studies for investigating the orientational distribution of crystallites, structural variations of interfaces with temperature and the atomic transport in nanostructures. By atomic resolution electron microscopy (JEOL ARM 1250) the orientation of crystallites and the orientation correlationship of neighbouring crystallites can be observed together with the interfaces between these crystallites. From the present studies we conclude that in gasphase prepared n-Pd a random orientation correlationship and high-energy grain boundaries prevail [1]. The present state of the investigation of thermal structural effects in interfaces of thermally stabilized nanocrystalline metals by positron annihilation techniques will be discussed. The temperature variation of the positron lifetime may indicate thermal formation of free volumes at elevated temperatures. Data on atomic diffusion in dense nanocrystalline materials available from self-diffusion (^{59}Fe in n-Fe [2]) and substitution-atom diffusion (^{59}Fe in n-Pd [3]) mainly coincide with the values expected from an extrapolation of conventional grain boundary diffusion. Studies on the nanocrystalline $Fe_{73.5}Si_{13.5}B_9Nb_3Cu_1$ composite show much higher ^{59}Fe diffusivities in the Fe_3Si crystallites than in the amorphous precursor [4] due to an enormously high thermal vacancy concentration in Fe_3Si. The ^{18}O diffusion in nanocrystalline monoclinic ZrO_2 [5] is by orders of magnitude higher in the interfaces than in the crystallites which may have consequences for the development of fast ion conductors. Finally the ordering processes in the nanocrystalline compounds FeAl and NiAl will be discussed [6]. [1] K.Reimann and R.Würschum, J.Appl. Phys. 81, 7186 (1997) [2] H.Tanimoto, P.Farber, R.Würschum, R.Z.Valiev, and H.-E.Schaefer, Nanostructured Materials 12, 681 (1999) [3] R. Würschum, Habilitationsschrift, Universität Stuttgart, 1997 [4] R. Würschum, P.Farber, R.Dittmar, P.Scharwaechter, W.Frank, and H.-

E.Schaefer, Phys. Rev. Letters 79, 4918 (1997) [5] U.Brossmann, R.Würschum, U.Södervall, and H.-E. Schaefer, J. Appl. Phys. 85, 7646 (1999) [6] K.Reimann and H.-E.Schaefer, Nanostr. Mat. 12, 633 (1999)

10:10 AM Break

10:30 AM

Grain Boundaries of Nanocrystalline Grains Studied by Molecular Dynamics Simulations: *Karsten Albe*[1]; Yinon Ashkenazy[2]; Robert S. Averback[2]; Horst Hahn[1]; [1]TU Darmstadt, Fachbereich Materialwissenschaften, Petersenstr. 23, Darmstadt 64287 Germany; [2]University of Illinois, Mats. Res. Lab., 104 S Goodwin Ave., Urbana, IL 61801 USA

Structure and dynamics of grain boundaries are important aspects in understanding the superplastic behavior of nanocrystalline materials. Computer simulations studies can provide useful information about the underlying atomic processes, which are not well understood. By means of molecular dynamics simulations we have investigated the dynamics of boundaries between finite grains and 2-dim. periodic surfaces as a model system. The interfaces are composed of regions with perfect crystal allignment and grain boundary dislocations (GBD). If no external forces are applied we observe the GBD array moving along the slip planes, thus creating a perfect match in the lower layers. For crystalites with small dimensions complete fast reorientation can be observed. A discussion of this effect in the context of dislocation-interface is presented. Finally, we present results, where grain boundary gliding with applied external forces has been simulated.

10:50 AM

A Comparison of Nanocrystalline Microstructures Developed by Different Deformation Processes: *Darcy A. Hughes*[1]; [1]Sandia National Laboratories, Ctr. for Matl. and Eng. Sci., P.O. Box 969, MS9405, Livermore, CA 94550-0969 USA

Very large strain deformation produces finely divided microstructures of deformation-induced high angle and dislocation boundaries. Quantitative transmission electron microscopy is used to measure the structure that develops, including boundary spacing and misorientation angle, as well as the local crystal orientations of the volume within the boundaries. The dislocation structure between the boundaries is also assessed. It is observed that the spacings between boundaries depend on the strain level ranging on average from 100 to 50nm at strains from 5 to 10. Individual spacings may be as small as 5nm. These structures are then compared to structures obtained by other techniques that create nanocrystalline structures. The relationship of the structures to mechanical properties is also discussed. This work supported by the U.S. DOE, under contract no. DE-AC04-94AL85000.

11:10 AM

Effect of Electrodeposition Parameters on Microstructure and Mechanical Properties of Nanocrystalline Nickel: *Fereshteh Ebrahimi*[1]; Zunayed Ahmed[1]; Kristin L. Morgan[1]; [1]University of Florida, Mats. Sci. and Eng. Dept., P.O. Box 116400, 180 Rhines Hall, Gainesville, FL 32611 USA

Electrodeposition is a viable method for fabricating metallic nanostructures. We have used a sulfamate-based solution and the galvanostatic method to produce nanocrystalline nickel deposits. This study reports the effects of current density and substrate on the microstructure and strength of nickel nanocrystals. The microstructure was characterized using x-ray diffraction, SEM, and TEM techniques. The mechanical properties were evaluated by tensile testing. It was found that there exists an optimum current density that yields the minimum crystallite size in nickel. The results of this study indicated that annealed copper substrates with a strong (100) rolling-plane resulted in deposits with a finer grain size than when a cold rolled copper with a strong (110) rolling-plane texture was used. The yield strength and strain-hardening rate increased with a decrease in grain size and its distribution width. Nodules and pores reduced the maximum uniform strain and hence the tensile strength of the deposits.

11:30 AM

Microstructure Development of Al-Based Amorphous Alloys with Pb: *Robert I. Wu*[1]; Zhenfu Dong[1]; John H. Perepezko[1]; [1]University of Wisconsin-Madison, Mats. Sci. and Eng., 1509 University Ave., Madison, WI 53706 USA

The high number density (10^{21}~10^{22} m^{-3}) of Al nanocrystals (~20nm in diameter) that can be developed by controlled primary crystallization of amorphous Al-RE-(TM) melt-spun ribbons yields an effective dispersion strengthening. An approach to increase the number density of the Al-nanocrystals has been developed through the incorporation of insoluble Pb throughout the amorphous matrix. The effectiveness of Pb in catalyzing the nucleation of Al-nanocrystals is revealed by studying the wetting behavior between Al-nanocrystals and Pb particles utilizing HRTEM. In addition, compositional analysis and thermal analysis reveal that the presence of crystalline Pb in the amorphous phase has altered the elemental diffusion path during crystallization of a-Al and appears to enhance the thermal stability of the microstructure. The support of the ARO (DAAG55-97-1-0261) is gratefully acknowledged.

11:50 AM

Microstructure and Properties of Ultrafine-Grained Pure Ti Processed by ECAP and Cold Deformation: *Vladimir V. Stolyarov*[1]; [1]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Mats., K. Marksa 12, Ufa 450000 Russia

Equal channel angular pressing (ECAP) has been used to refine the grain size of commercially pure (CP) Ti as well as other metals and alloys. CP-Ti is usually processed at about 400°C because it lacks sufficient ductility at lower temperature. The warm processing temperature limits the capability of the ECAP technique in improving the strength of CP-Ti. We have employed cold deformation following warm ECAP to further improve the strength of CP-Ti. Ti billets were first processed for 8 passes via ECAP route BC, with a clockwise rotation of 90° between adjacent passes. The grain size obtained by ECAP alone is about 260 nm. The billets were further processed by cold deformation (cold extrusion, and/or rolling) to increase the crystalline defects such as dislocations. The strength of pure Ti was improved from 380 MPa to around 1000 MPa by the two step process. This presentation reports the surface quality, microstructures, microhardness, tensile properties, and thermal stability of these Ti billets processed by a combination of ECAP and cold deformation.

Sampling, Sensors & Control for High Temperature Metallurgical Processes: Aluminum Reduction Technology-Sampling & Sensors

Sponsored by: Light Metals Division, Extraction & Processing Division, Materials Processing and Manufacturing Division, Aluminum Committee, Pyrometallurgy Committee, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: Adrian Deneys, Praxair, Inc., Tarrytown, NY 10591 USA; Derek Fray, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3Q2 UK; Matt Krane, Purdue University, Department of Materials Engineering, West Lafayette, IN 47907; Markus Reuter, Delft University of Technology, Applied Earth Science, Delft 2628 RX The Netherlands; Fiona Stevens McFadden, University of Auckland, Chemistry and Materials Engineering, Auckland, New Zealand

Wednesday AM — Room: 230
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Fiona Stevens McFadden, University of Auckland, Chem. and Mats. Eng., Auckland, New Zealand; Adrian C. Deneys, Praxair, Applications Res. and Dev., Tarrytown, NY 10591 USA

8:30 AM

Control of Temperature in Aluminium Reduction Cells-Challenges in Measurements and Variability: *Fiona Jean Stevens McFadden*[1]; Daniel Whitfield[1]; Barry J. Welch[1]; [1]University of

Auckland, Chem. and Mats. Eng. Dept., Auckland 92019 New Zealand

The temperature of the electrolyte is one of the key process variables in a reduction cell, as it has a strong influence on cell current efficiency. The variance in temperature, a measure of control performance, is typically higher than desired and smelter operators would generally speaking like to see an improvement. Measurement of temperature is made difficult by a number of factors such as the corrosive nature of electrolyte and the spatial and temporal variation resulting from the semi-batch, semi-continuous nature of the process. Difficulties also arise in the interpretation of the temperature measurement and feedback to control action, as temperature change can result from a change in excess aluminium fluoride, alumina concentration and/or superheat. Depending upon the cause of temperature variation the appropriate control action may vary.

9:00 AM

Development and Application of a Novel Sensor for Combined Bath Temperature and Cathode Voltage Drop Measurements in Aluminium Reduction Cells: Bernd Rollofs[2]; *Peter White*[1]; Rik Kelchtermans[1]; Neal Wai-Poi[2]; Paul Verstreken[1]; [1]Heraeus Electro-Nite International N.V., Prod. Mgmt., Centrum Zuid 1105, B-3530, Houthalen B-3530 Belgium; [2]Corus Voerde, Reduction, Huttenwerk Voerde, Scheleusenstrase D-46562, Voerde D-46549 Germany

Cathode voltage drop measurements are conducted by most smelters on a periodic basis. Whilst the measurement is necessary to optimise target voltage, most smelters consider the present methods to be cumbersome, labour intensive and the accuracy can be questionable due to a heavy reliance upon human interpretation. A new technique of combining cryolite bath temperature measurement with CVD measurement has been developed which provides a novel method of measurement that significantly reduces, and in some cases, eliminates the labour cost associated with conventional CVD methods. This paper describes the development of the sensor, presents field tests to assess the sensor reproducibility and illustrates the recent application of the sensor into routine use at the Corus Voerde aluminium smelter in Germany. The benefits of this new technique, as perceived by the smelter are identified. Future possibilities for the sensor including monitoring of sludge and ridge build up are also discussed.

9:25 AM

Fuzzy Pattern Recognition of Temperature for Aluminum Electrolyte: *Zeng Shuiping*[1]; [1]North China University of Technology, Instit. of Auto., Shijinshan District, Beijing City, Beijing 100041 China

This paper develops a Fuzzy Pattern Recognition model for aluminum bath temperature based on a lot of data of temperature measurement in alumina reduction process. According to the these data the paper concludes the some fuzzy rules and makes inferences by max-min methods. In the end, the output, i.e., temperature of the system is defined by centroid defuzzification. Two methods are adopted in this pattern recognition. One is by fuzzy relation matrix; and the other is by Matlab Fuzzy Logic Toolbox. Verification results show the average relative error is less than 0.4%, which indicates the model can recognize the temperature of Al-electrolyte well. Therefore, if the method is applied in aluminum production, it can reduce the cost and the labor intensity of temperature measurement, and much benifits to the cell-control system.

9:50 AM

Real Time Alumina Distribution Measurement in Industrial Cells: *Richard G. Haverkamp*[1]; [1]Massey University, Inst. of Techn. and Eng., Private Bag 11222, Palmerston North 1015 New Zealand

The distribution of alumina in a smelter cell has been measured in real time. Measurements are performed with a hand-held or stand-mounted probe connected to a self-contained power supply and laptop computer adapted without shielding to work in a high magnetic field environment. Either individual measurement of alumina concentration or continuous alumina concentration measurements can be made. This gives another tool for monitoring cell electrolyte flows and alumina concentration gradients. These measurements can be used, for example, in conjunction with simultaneous temperature measurements to show the transport and dissolution of the alumina within the cell after feeding and to identify regions that are poorly fed by alumina. The system has been adapted to work in a range of electrolyte compositions with each calibration applicable within a limited range of cryolite ratio.

10:15 AM Break

10:25 AM

Development of Techniques for Measuring the Composition of Low Temperature Electrolytes: *Olivier Crottaz*[1]; Jennifer Purdie[1]; Vittorio de Nora[1]; [1]Moltech S.A., 9 Rte. de Troinex, Carouge, Ge 1227 Switzerland

Low temperature, cryolite-based electrolytes for the Hall-Héroult process continue to be of interest to the Aluminium industry. In a conventional Hall-Héroult cell, lower temperature operation could increase energy efficiency and improve materials performance. Low temperature electrolytes may also facilitate the operation of an inert anode, since the solubility of many metal oxides decreases with temperature. The ability to measure bath composition (AlF3, Al2O3 and other components) quickly and accurately will be critical to successful operation of low temperature electrolytes. Liquidus temperature becomes more sensitive to small changes in concentration as AlF3 concentration increases. Measurement and control of Al2O3 concentration would be particularly important to operation with inert anodes. In this paper the development of techniques for analysis of high AlF3 electrolytes for low temperature operation is discussed. The techniques include both laboratory analysis methods, and the use of available sensors for instantaneous measurement of superheat and alumina concentration, which together might allow a more rapid estimation of bath composition.

10:50 AM

Development of a Sensor to Measure Velocity in High Temperature Liquid Metals: *Stavros A. Argyropoulos*[1]; [1]University of Toronto, Dept. of Metall. and Mats. Sci., 184 College St., Toronto, Ontario M5S 3E4 Canada

This paper will describe the development of a sensor to measure localized velocity in high temperature liquid metals. This sensor utilizes a sphere which is immersed in a moving liquid metal. By measuring the melting time of sphere, the liquid metal velocity can be inferred. The elementals for the development of this sensor were carried out in a commercial purity aluminum bath and in liquid low carbon steel. In developing the sensor, we investigated a range of bath temperatures, as well as the impact of different sphere diameters. Results showed that the sphere melting time was related linearly to the magnitude of flow velocity for the range of velocities of 0-40 cm/sec and for bath superheats up to 100°C. How this sensor can be adapted to be used in other high temperature reactive liquid metals and liquid slags will be shown. In addition, a modification of this technique to detect direction of velocity in these high temperature hostile fluids will be described.

11:15 AM

Novel Solid State Sensor for Mg in Molten Al: *Girish Madhav Kale*[1]; [1]University of Leeds, Min. and Miner. Eng., Sch. of P, E & M Eng., Clarendon Rd., Leeds, West Yorkshire LS2 9JT UK

A novel and completely solid state sensor for measuring dissolved magnesium in molten aluminium for demagging and alloying operation has been developed employing two different novel solid electrolytes. Novel bi-phasic reference electrode materials have been used in designing the Mg-sensor. The solid state sensor has been tested between 963 to 1003K in molten Al-Mg alloys. The sensor was found to respond rapidly to change in concentration of Mg in molten alloy between 0.0003 to 0.03 weight fraction of Mg in Al. The present paper will discuss the preparation of solid electrolyte materials, electrical characterisation of the solid electrolyte materials by ac-impedance spectroscopy, preparation of the reference electrode materials, fabrication of sensor and testing of sensor in the laboratory.

11:40 AM

Sensitivity Analysis of the Thermal Detection of the Freeze Profile in an Aluminium Reduction Cell: Laszlo Istvan Kiss[1];

Rung Tien Bui[1]; Paul Desclaux[2]; *Pascal Boily*[1]; [1]University of Quebec at Chicoutimi, DSA, 555 boul de l'Universite, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International, CRDA STE, 1955 Boul Mellon, Jonquiere, Quebec G7S 3H5 Canada

The potentials of the thermal detection of the freeze profile were studied by laboratory experiments and by sensitivity analysis. In the experimental set-up, the freeze was represented by a sand layer placed on top of a carbon slab. Temperatures were measured in different points inside the carbon slab. The shape of the isothermal surface of the sand layer was identified by a numerical procedure based on the solution of the inverse heat conduction problem. The material properties, geometrical parameters, boundary conditions that are used in the inverse solution, influence the performance of the identification procedure. The sensitivity values of the freeze detection were determined for a wide range of the influencing parameters like the temperature of the interface, thermal conductivity of the freeze and cathode side-block (aging) as well as for non-zero parietal heat fluxes.

Second Global Symposium on Innovations in Materials Process & Manufacturing: Sheet Materials: Secondary Processing, Texturing and Tribology

Sponsored by: Materials Processing and Manufacturing Division, Powder Materials Committee, Shaping and Forming Committee, Solidification Committee

Program Organizers: Mahmoud Y. Demeri, Ford Motor Company, Manufacturing System Department, Northville, MI 48167 USA; Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; John Papazian, Northrup Grumman, Bethpage, NY 11714 USA; Klaus Siegert, University of Stuttgart, Inst. for Met. Form. Tech., Stuttgart D-70174 Germany

Wednesday AM Room: 228
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Henry Piehler, Carnegie Mellon University, Pittsburgh, PA 15213 USA; Amit Ghosh, University of Michigan, Ann Arbor, MI USA

8:30 AM

Evaluating Friction in Sheet Metal Stamping–Principles and Methods of Tribology: *Gregory M. Dalton*[1]; [1]TribSys, Inc., 1400 Barrydowne Rd., NORCAT Bldg., Sudbury, ON P3A3V8 Canada

During the last decade of the 20th century efforts to reduce the dependence on metal stamping lubricants saw the development of water-based lubricants, dry film lubricants, and even thixotropic lubricants. While some manufacturers sought the holy grail of metal stamping-stamping without lubricants, for most processes this was not possible. Early hopes that synthetic lubricants would replace chlorinated EP additives have not materialized. Increased use of zinc-coated steel and aluminum has added to the confusion. The outcome of these fractious efforts is a confused end user. Consequently, lubricant suppliers and users are turning to laboratory testing to evaluate lubricant performance. A wealth of published papers showing the importance of friction on metal stamping performance have encouraged lubricant, steel, and aluminum producers as well as OEMs and Tier 1 suppliers to purchase equipment begin their own friction testing programs. As metallurgists, chemists, and other non-tribologists enter the realm of tribology there are some fundamentals that must be understood to ensure the data and analysis are meaningful.This paper examines the important Tribological principles in friction testing and reviews the basic differences in common tribotesters. Data from these tests are examined and compared. Conclusions are drawn and procedures for establishing a friction-testing program are outlined.

8:55 AM

Quantification of Deformation Induced Surface Roughening Using Spectral Methods and Fourier Filtering: *Y. Choi*[1]; H. R. Piehler[1]; L. G. Hector[2]; A. D. Rollett[1]; [1]Carnegie Mellon University, Mats. Sci. & Eng., Pittsburgh, PA 15213-3890 USA; [2]Alcoa Technical Center, Alcoa Center, PA 15069-0001 USA

Surfaces of metal sheets initially contain high frequency, low amplitude perturbations imparted during the rolling process. When plastic deformation is imposed, these free surfaces increase the amplitude of these perturbations and produce various kinds of morphological features including randomness, periodicity and waviness. The key to understanding the evolution of topographical features on these deformed surfaces involves characterizing those features using appropriate parameters. In this study, various roughness characterization tools are used to investigate the 3-D surface roughening of 6022-T4 Al sheets deformed in plane strain. Areal Power Spectral Density (APSD), Areal Auto-Correlation Function (AACF) and Radial Spectral Density (RSD) are employed to characterize roughness texturing and periodicity. The newly introduced peak-to-valley mean-height roughness (Rmh) is used to characterize roughness amplitude in order to overcome the deficiencies of the root mean square roughness (Rrms). A new approach, dividing the roughening pattern into several regimes, is also used to quantify the perturbations that provide the major contributions to surface roughening. The Fast Fourier Transform (FFT) algorithm is used to filter 3-D roughness into these different roughness regimes.

9:20 AM

Effect of Asymmetric Rolling Parameters on Texture Develpoment in Aluminum Sheets: *Dong Nyung Lee*[1]; Keun-Hwan Kim[2]; [1]Seoul National University, Mats. Sci. and Eng., Shinrim-dong, Seoul 151-742 Korea; [2]POSCO, Technical Research Laboratories, Pohang 790-785 Korea

Asymmetric rolling, in which the upper and lower roll radii are different, imposes shear deformation textures in sheets through the thickness. A component of ND <111> in the shear deformation textures can improve the plastic strain ratios of aluminum sheets. The shear deformation texture can vary with the ratio of shear to normal strain increments. As the ratio increases from zero to infinity, the texture moves from the plane strain compression texture (beta fiber) to the ideal shear deformation texture consisting of {001}<110>, {111}<110>, and {111}<112>. The ratio increases with rolling reduction per pass in asymmetric rolling. However, it is practically difficult to the rolling reduction per pass to be high enough to obtain the ideal shear deformation texture. Imposing the positive and negative shear deformations on the sheet by reversing the shearing direction can give rise to the ideal shear deformation texture. The effect of asymmetric rolling on texture will be presented and discussed in this paper.

9:40 AM

The Influence of Grain Size on the Roughening Behavior of Al-Mg Alloys: *Mark R. Stoudt*[1]; Richard E. Ricker[1]; [1]NIST, Mats. Sci. & Eng. Lab., 100 Bureau Dr., Stop 8553, Gaithersburg, MD 20899-8553 USA

The discontinuous plastic flow exhibited by numerous aluminum alloys presents significant challenges to the use of aluminum alloys in many automotive applications. The inhomogeneous morphology of the surface asperities generated during metal forming increases the friction between mating die surfaces resulting in accelerated die wear and progressively reduced shape accuracy in the metal stampings. The roughness data present in the literature are generally inadequate for use in finite element simulations of die wear from aluminum forming. One important question that needs to be addressed more thoroughly is what are the relative influences of grain size and plastic strain on the surface roughening behavior? In response, surface profilometry measurements were performed on 1 mm sheets of substitutionally strengthened Al-Mg alloys pulled in uniaxial tension to controlled levels of plastic strain. The results of these evaluations will be presented and discussed.

10:00 AM Break

10:20 AM

Optimizing Fiction Between Die and Sheet Metal: *Stefan Wagner*[1]; [1]University of Stuttgart, Insti. for Met. Form. Tech., Holzgartenstrasse 17, Stuttgart 70174 Germany

In deep drawing and drawing of car body parts the friction conditions are of great influence on process limits, on the robustness of the production process and on the quality of the produced parts. Beside the used lubricant, the friction conditions are influenced by the topography of the sheet metal surface and by the topography of the tool surface. This paper deals with the influence of sheet metal surfaces, coatings on sheet metal surfaces, tool surfaces, coatings on tool surfaces and with the influence of the lubrication on the frictional behaviour.

10:40 AM

Deformation Response of Sheet Stock Machined from AA 7050 T7451 Plate: T. J. Turner[1]; E. J. Harley[1]; *Matthew P. Miller*[1]; Armand J. Beaudoin[2]; William A. Cassada[3]; [1]Cornell University, Mech. and Aeros. Eng., 194 Rhodes Hall, Ithaca, NY 14853 USA; [2]University of Illinois, Mech. and Indust. Eng., 1206 W. Green St., MC-244, Urbana, IL 61801 USA; [3]Reynolds Metals Company, Corp. Res. & Dev., 13203 N. Enon Church Rd., Chester, VA 23831-3122 USA

High speed machining of AA 7050 aluminum plate offers a means of producing structural assemblies with section thickness similar to conventional sheet materials. However, grain morphology and textural anisotropy lead to different deformation response, as compared to sheet materials. In the present work, a set of tensile experiments were performed using specimens machined from AA7050 T7451 plate. Both strength and ductility showed direction dependence. These property variations were further associated with deformation-induced surface roughening. In general, specimens tested in the rolling direction (RD) showed greater ductility that specimens tested in the transverse direction (TD). Specimens tested in the transverse direction developed "troughs" aligned along the road. Observed hardening rate data for the tensile tests is contrasted with predicted behavior using a polycrystal plasticity model.

11:00 AM

Diffusion Bonding Steel: *David C. Van Aken*[1]; [1]University of Missouri-Rolla, Metall. Eng., 1870 Miner Circle, Rolla, MI 65409-0340 USA

Diffusion bonding of low carbon steel is obtained by using a powder metallurgy technique where the joint is filled with a mixture of iron powders and iron carbide or iron nitride. Plasma spraying of the iron carbide onto the joint area is also possible. Upon heating above the eutectoid temperature, the iron carbide or iron nitride compounds decompose to create austenite in the joint. Decomposition is driven by the rapid diffusion of the interstitial (C or N) into the base metal. Bonding experiments were performed between 870°C and 930°C in an argon atmosphere and parts were subsequently air-cooled. Metallographic examination of the joint shows a ferrite and pearlite microstructure through out the joint with an obvious diffusion gradient into the base metal. High strength joints can be obtained by this technique and when tested in tension, failure occurs through the base metal.

11:20 AM

Methodology of Design of Pulsed Electromagnetic Joining of Tubes: *Sergey F. Golovashchenko*[1]; [1]Ford Research Laboratory, Manufact. Sys. Dept., 2101 Village Rd., P.O. Box 2053, MD3135, SRL, Dearborn, MI 48121-2053 USA

An intense transient magnetic field is used in magnetic pulse forming, and through interaction with the metal work-piece, pressure in the form of a magnetic pulse is built up to do the work. In this paper, the technology of tube joining by means of filling the circular grooves with tube material is presented. These joints after assembling usually come under the influence of axial tensile or compressive static or dynamic load conditions. In order to achieve the optimal design of the assembling process and to meet the requirements of the axial load carrying capacity of the product, optimization of groove parameters and electromagnetic coil design must be performed for certain electromagnetic pulsed machine. The objective of the optimization process is to reach minimum value of the electromagnetic pressure peak which will provide the required strength of the assembled part. Minimizing the electromagnetic pressure peak extends the life of the electromagnetic coil.

Teaching and Learning Solid State Diffusion

Sponsored by: ASM International: Materials Science Critical Technology Sector, Atomic Transport Committee

Program Organizers: Richard D. Sisson, Worcester Polytechnic Institute, Mats. Sci. & Eng., Worcester, MA 01609 USA; Joe I. Goldstein, University of Massachusetts, Department of Engineering, Amherst, MA 01020 USA; John Morral, University of Connecticut, Deparatment of Metallurgy, Storrs, CT 6260 USA

Wednesday AM — Room: 202
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Richard D. Sisson, Worcester Polytechnic Institute, Mats. Sci. and Eng., Worcester, Massachusetts 01608 USA; John Morral, University of Connecticut, Dept. of Metall. and Mats. Eng., Storrs, CT USA

8:30 AM

Teaching Mass Transport to Undergradaute Students: *David S. Wilkinson*[1]; Gary R. Purdy[1]; [1]McMaster University, Mat. Sci. and Eng. Dept., 1280 Main St. W., Hamilton, Ontario L8S 4L7 Canada

At McMaster we have long taught an undergraduate course (3rd year level) dedicated to mass transport. This course contains a large component of solid-state diffusion theory. However it also deals with transport in fluids. We have developed an approach to the subject which enable students to see the link between drift during diffusion and convective flow in fluids. A new textbook has just been developed which emphasizes this link through the use of a common nomenclature. Other features of this course include a problem-based learning approach with particular emphasis on physical modeling plus the use of Maple V software for demonstrations and simulations.

8:55 AM

A One-Semester Course on Diffusion in Solids: *Martin E. Glicksman*[1]; Afina Lupulescu[1]; [1]Rensselaer Polytechnic Institute (CII-9111), Mats. Sci. and Eng. Dept., CII-9111, 110 8th St., Troy, NY 12180-3590 USA

One author (MEG) has taught the subject of solid-state diffusion for 25 years to professional engineers and graduate and undergraduate students with divergent backgrounds in engineering and physical science. These pedagogical exposures were integrated and distilled into the recently published textbook, Diffusion in Solids: Field Theory, Solid-State Principles and Applications [ISBN 0-471-23972-0], Wiley-Interscience, 2000. This book supports one-semester introductory courses on solid-state diffusion phenomena and their applications, and provides auxiliary readings for typical upper-divisional materials engineering courses in kinetics and processing. A brief overview of the book is included to highlight the scope and approach taken to teach classical (continuum) diffusion theory for solving diffusion problems and its integration with modern solid-state concepts for understanding diffusion. The encouragement to apply numerical methods, and the inclusion of advanced topics such as microstructure evolution and multicomponent diffusion, will be discussed. The development of additional aids as companion teaching materials will also be included.

9:20 AM

Diffusion in a Junior-Level Materials Kinetics Course: *Dennis W. Readey*[1]; [1]Colorado School of Mines, Metall. and Matls. Eng., 1500 Illinois St., Golden, CO 80401 USA

Both atomic and macroscopic aspects of solid-state diffusion are taught in a second semester junior-level course on solid-state kinet-

ics. The conservation of mass equation is derived and several steady-state solutions are developed for precipitation, dissolution, etc. Then the partial differential equation is solved for different cases of approximate initial conditions for finite boundary condition problems that permit single term solutions: homogenization, drying, and decarburization. A similarity variable is used to solve infinite and semi-infinite boundary condition problems. Problem sets include the use of spreadsheets to solve finite-difference equations and random-walk problems.

9:45 AM Break

10:10 AM

Teaching Diffusion: Paris Round-Table: *Jean Philibert*[1]; *Yves Limoge*[1]; Jean-Louis Bocquet[2]; Jean Bernardini[3]; [1]Universite Paris-Sud, Metall. Dept., Bat 410, Orsay, Cedex F-91405 France; [2]CEA, CEREM, SRMP, Gif/Yvette 91190 France; [3]University Aix-Marseille, Metall. Dept., Marseiile, France

In the program of the DIMAT 2000 Conference (Paris, July 17-21, 2000), this abstract is submitted just the week before the Paris Conference, a round table has been organised to exchange ideas, experiences and proposals on the what and how in order to help teachers who have to give a class on diffusion in condensed media. Two main questions were proposed to the attendees:1) What are the essential points to be taught according to the cursus (basic knowledge, professional purpose)? What is the best approach: beginning with the atomic theory or with the macroscopic equations? How to convince the students of the importance of this course? 2) How to teach? Pointing out items that raise difficulties for the students, defining the best choice of practical exercises, assessing the role and importance of computer-assisted teaching. Conclusions appear as recommendations for the teachers in the form of practical advices and of a list of the essential items to be known and understood by the students. A proposal for creating a WEB site devoted to the teaching of diffusion was presented and discussed by the attendees.

10:35 AM

An Integrated Education Program on Thermodynamics, Kinetics, and Materials Design at Penn State: *Zi-Kui Liu*[1]; Long-Qing Chen[1]; Karl Spear[1]; [1]The Pennsylvania State University, Dept. of Mats. Sci. and Eng., Steidle Bldg., University Park, PA 16802 USA

The National Science Foundation has recently decided to fund an educational program at the Department of Materials Science and Engineering, The Pennsylvania State University. The objective of the project is to improve the student's learning experience and educator's teaching experience on two of the core components in the curriculum of materials science and engineering, i.e. Thermodynamics and Kinetics, by integrating fundamental principles and advanced computational approaches. Computer-based education tools will help students connect abstract thermodynamic concepts with the properties of real world materials, and mathematical kinetics with practical materials processing procedures, and thus remove the common stereotype perception among university students that thermodynamics and kinetics are problematical to learn and difficult if not impossible to apply in the real world. In this presentation, the goals and infrastructure of the project will be discussed.

11:00 AM

Teaching Multiphase Diffusion to Chemical Engineers: *F. J. van Loo*[1]; A. A. Kodentsov[1]; [1]Eindhoven University of Technology, Lab. of Sol. Ste. and Mats. Chem., P.O. Box 513, Eindhoven 5600 MB, The Netherlands

There are many ways of teaching "Diffusion", because the subject is essentially classical in nature and relevant to many branches of science. Obviously, the content of the courses depends, largely, on the undergraduates (physicists, chemists, mechanical engineers, etc.) for whom these are intended. In this presentation, we will share our experience in teaching "Diffusion" within a curriculum in our department of Chemical Engineering. The main objective of the course is to present the basic fundamentals on a level appropriate for chemical engineering students at a technical university, who have completed their freshman calculus, chemistry and physics. Since a chemical or materials engineer will be mainly confronted with the formation of new phases (oxidation, substrate/coating interaction, composite materials, etc.) the emphasis is put on the understanding of multiphase diffusion. The role of Thermodynamics (driving force, phase diagrams) and Kinetics (essentially diffusion when quasi-equilibrium has been reached) is made clear. The frame of reference in which diffusion fluxes are measured (Kirkendall- and Matano-plane) are extensively treated because the clue of understanding is hidden there. Apart from the courses given orally the students have to study some articles and they participate in an experimental project which relates to our own research field. Diffusion is not always the central theme in this research, but is at least an important research tool. It is organized in such a way that a group of about 6 students work during 6 "half-days" on a specific topic; other groups are working in the same system but at different composition or temperature. At the end of the course all students (up to about 24) come together to discuss their results. This ends up in a poster, which after some refinement by us is shown at (international) conferences. They all get a copy and are very proud having done real research. We will show a few examples at this "teaching" conference. Having said this, it is our firm belief that the traditional straightforward lecture remains both valuable and important. Our primary role as teachers is to communicate our enthusiasm for chemistry, physics or materials science to our students.

11:25 AM

Teaching Multicomponent Diffusion: *J. E. Morral*[1]; [1]University of Connecticut, Metall. and Matls. Eng. Dept., 97 N. Eagleville Rd., P.O. Box U-136, Storrs, CT 06269-3136 USA

Teaching multicomponent diffusion to undergraduate students can be a formidable task, because of the large number of potential variables, the complexity of equations, and the lack of elementary treatments in textbooks. However there is an approach to teaching the subject that greatly simplifies the topic for both teacher and student. To follow this approach may require some change in the way that diffusion in binary systems is taught, but the benefits are great when explaining fundamental multicomponent equations and how to apply them. Students find that after several lectures they know how to measure diffusivities in higher order systems, design coatings to minimize interdiffusion or eliminate Kirkendall porosity, and to classify concentration profiles and types of boundaries that form, even in complex systems. Explaining diffusion paths and special "multicomponent effects" takes longer and may involve discussing controversial subjects, making them both more suitable for advanced undergraduates and graduate students.

Aluminum Joining-Emphasizing Laser and Friction Stir Welding:
Session 3 - Frictions Stir Welding-Process and Process Development

Sponsored by: Light Metals Division, Aluminum Association
Program Organizers: John A.S. Green, The Aluminum Association, Washington, DC 20006-2168 USA; J. Gilbert Kaufman, Columbus, OH 43220-4821 USA; Thomas J. Lienert, Edison Welding Institute, Columbus, OH 43221-3585 USA

Wednesday PM Room: 214
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Thomas J. Lienert, Edison Welding Institute, Columbus, OH 43221-3585 USA

2:00 PM Keynote
Friction Stir Welding-Tool Developments: *Dave Nicholas*[1]; Simon D. Smith[1]; Wayne Morris Thomas[1]; [1]TWI, Innovat. Unit, Granta Park, Great Abington, Cambridge, Cambridgeshire CB1 6AL UK

By any standard the industrial adoption of friction Stir welding as the preferred joining technique for a range of aluminium alloys represents a remarkable progress of technical development. Furthermore, a wide range of non-ferrous and ferrous materials of various thickness has also been shown to be readily welded by FSW in the laboratory. The design of the tool is the key to the successful application of the process. A number of different high performance tool designs have been investigated. This paper describes recent developments using these enhanced tools from the perspective of existing and potential applications.

2:30 PM Invited
Wiping Metal Transfer in Friction Stir Welding: *Arthur C. Nunes*[1]; [1]NASA/Marshall Space Flight Center, Matls. Process. and Manufact. Dept., ED33, Marshall Space Flight Center, Huntsville, AL 35812 USA

In Friction Stir Welding (FSW) a rotating pin-tool inserted into a weld seam literally stirs the edges of the seam together. The superposition of a rapidly rotating cylinder, a slowly rotating ring vortex, and a uniform translational flow generates a "wiping" flow that appears to model the plastic flow around pin-tool. The wiping model is described and used to explain the results of a number of tracer experiments (traversed slab, line of shot, traversed wires). The wiping process model is compared to the metal cutting process and shown to have much in common, including very high strain rates. The model comprises a starting point for the rational design of the FSW pin-tool.

3:00 PM
Process Development of Friction Stir Lap Joints in AA7075 and AA2297 Alloys: *Zhixian Li*[1]; William J. Arbegast[1]; Anthony Reynolds[2]; Kumar Jata[3]; [1]Lockheed Martin Space Systems Company, Michoud Oper., Prog. and Techn. Dev., P.O. Box 29304, New Orleans, LA70189 USA; [2]University of South Carolina, Dept. of Mech. Eng., 300 S. Main St., Colombia, SC 29208 USA; [3]Air Force Research Laboratory, AFRL/MLLM, WPAFB, OH 45433 USA

Friction stir welding (FSW) has shown potential applications in airframe structures to replace riveted joints by taking advantage of various FSW joint configurations such as lap joints and fillet joints. In the present study, both AA7075-T6 sheet and AA2297-T8 sheet were lap-joined via friction stir welding using different pin tool configurations and lengths. Effects of pin tools and processing parameters on joint microstructures and mechanical properties will be presented. The different responses of AA7075 and AA2297 to FSW lap joining will be addressed as well.

3:25 PM
Development and Application of an Analytical Process Model for FSW: *Michael J. Russell*[1]; H. R. Shercliff[2]; P. L. Threadgill[1]; [1]TWI, Friction and Forge Processes Dept., Granta Park, Great Abington, Cambridge CB1 6AL UK; [2]Cambridge University, Eng. Dept., Trumpington St., Cambridge CB2 1PZ UK

Friction stir welding is being applied to an increasing number of joining applications worldwide, primarily in aluminium alloys. Uptake of the process has been rapid, and has involved a number of prominent engineering companies and high profile projects. FSW has successfully made the transition from a laboratory curiosity to an industrially important technology in a relatively short time. The science of FSW is not as well advanced however, and most development to date has been empirically based. Effective modelling of the process offers significant time and cost savings at this stage, particularly when used as part of a co-ordinated development programme. The key aim in effective modelling is to match the complexity of the solutions used to the level of predictive detail required. In this way relatively simple modelling approaches can be used where appropriate, giving a fast and flexible development tool. This paper presents an analytical process model for FSW, developed for age-hardening Al alloys. The model is described in three main parts: 1. Calculation, and representation, of the energy input of the FSW tool. 2. Modelling of the thermal field produced in the workpiece during FSW. 3. Prediction of the response of the workpiece material during FSW. For each section of the model, comparisons are made between the predictions generated, and experimental measurements. Finally, a case study is presented which illustrates the use of the combined model as a process development tool.

3:50 PM Break

4:05 PM
Flow and Deformation of Material during Friction Stir Welding: Tracy W. Nelson[1]; *Carl D. Sorensen*[1]; [1]Brigham Young University, Mech. Eng., 435 CTB, Provo, UT 84602 USA

Material flow and deformation mechanisms in friction stir welding are complex at best, most likely being dependent on several factors. Understanding the mechanisms of material flow during FSW may aid in the design of tooling or optimization of parameters specific to certain weld properties. Several investigators have made great efforts in characterizing the flow of material during FSW. Although these efforts have provided substantial information regarding material flow, there still exists a tremendous lack of understanding regarding this aspect of FSW. This paper will present the results of marker experiments in aluminum alloys 6061 and 7075. Marker experiments were produced using a stop-action technique and continuous markers. Results indicate that material flow and development of weld nugget are dependent on the base material and processing parameters. Likewise, the degree of mixing within the nugget can be controlled, to some extent, by the ratio of rotational-to-travel speeds during welding. This aspect could have important implications when joining dissimilar aluminum alloys with FSW. Although the exact nature of material flow during FSW is not obvious, several observations will be presented that will aid the FSW community in better understanding this aspect of the process.

4:30 PM
Material Flow Characterization of Al6061 Friction Stir Welds: Xun-Li Wang[2]; David Wang[2]; Bill Chao[3]; Wei Tang[3]; *Zhili Feng*[1]; [1]Emc2, 3518 Riverside Dr., Ste. 202, Columbus, OH 43221 USA; [2]Oak Ridge National Laboratory, Spall. Neut. Sce., Bldg 7964H, Rm. 110 MS 6430, Oak Ridge, TN 37831 USA; [3]University of South Carolina, Dept. Mech. Eng., Columbia, SC 29208 USA

Abstract text is unavailable.

4:55 PM
Characterization of Friction Stir Weld Defect Types: *William J. Arbegast*[1]; Edmond R. Coletta[1]; Zhixian Li[1]; [1]Lockheed Martin

Space Systems Company, Michoud Oper., P.O. Box 29304, New Orleans, LA 70189 USA

Friction stir welding (FSW) of aluminum alloys is rapidly finding acceptance as a low cost replacement for fusion welding and riveting in aerospace and aircraft structures. While the solid state process has a large operating parameter box and can consistently produce defect free joints, certain defect types are possible. In contrast to fusion weld defects, the formation of which are governed by the liquid-solid transformation, friction stir weld defects are governed by metal flow patterns. Potential defect types in butt joints and lap joints are described in terms of the fundamental friction stir weld flow model, pin tool geometry, and processing parameters. The effects of these defects on joint performance are also described.

5:20 PM

Faying Surface Defect Analysis of Friction Stir Welded 2195 Alloy: *Zhixian Li*[1]; William J. Arbegast[1]; Brian Dracup[1]; [1]Lockheed Martin Space Systems Company, Michoud Operations, Program and Technology Development, P.O. Box 29304, New Orleans, LA 70189 USA

AA 2195 plates were butt-joined via friction stir welding (FSW) process with both a nominal and a flat-tipped pin tool. A faying surface defect was observed from the welds made using the flat-tipped pin tool. It was found that the faying surface was an aggregate of second phase particles that lined up to a narrow ribbon, and often it became a preferred fracture path that deteriorate the mechanical properties. Auger Electron Spectroscopy (AES) was utilized to characterize the FSW faying surface. The AES results indicated that the particles of faying surface were rich in Cu, O, and S elements. In order to understand the source of S contamination, two different surface preparation conditions were examined using AES. The detailed microscopic analysis will be presented.

Aluminum Reduction Technology - Stream I: Fundamentals

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: John Chen, University of Auckland, Department of Chemical & Materials Engineering, Auckland, New Zealand; Eric Jay Dolin, USEPA, MC 6202J, Washington, DC 20460 USA; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway

Wednesday PM — Room: 206-207
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Jennifer Purdie, Moltech S.A., 9 Rte. de Troinex, Carouge, Ge 1227 Switzerland

2:00 PM

The Content of Sodium in Aluminium in Laboratory and in Industrial Cells: *J. Thonstad*[1]; S. Rolseth[1]; J. Rodseth[2]; O. Lund[2]; J. Tonheim[2]; V. Danielik[3]; P. Fellner[3]; J. Hives[3]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. and Electrochem., Trondheim 7491 Norway; [2]Hydro Aluminium Karmoy, Havik 4265 Norway; [3]Slovak University of Technology, Dept. of Inorg. Tech., Bratislava 81237 Slovakia

The concentration of sodium in molten aluminium in contact with cryolite-based melts is determined by the equilibrium 3 NaF+Al = 3 $Na_{in\ Al}$+AlF_3. Laboratory data for varying electrolyte composition and temperature are compared with a thermodynamic model. During electrolysis the content of sodium increases with increasing current density, caused by a concentration gradient at the cathode, which gives rise to concentration overvoltage. On the assumption of equilibrium between the electrolyte adjacent to the cathode and the aluminium, the composition of the electrolyte at the interface can be estimated. A new metal sampling technique for industrial cells was introduced to prevent sodium losses during sampling. The content of sodium varied with the alumina feeding cycle, and it decreased with increasing temperature and with decreasing aluminium fluoride. These trends, which apparently are in conflict with thermodynamic data, will be discussed.

2:25 PM

Modeling the Solubility of Alumina in the NaF-AlF3 System at 1300K: *Sanjeev Gupta*[1]; Yunshu Zhang[1]; Yogesh Sahai[1]; Robert A. Rapp[1]; [1]The Ohio State University, Dept. of Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

The experimentally well known alumina solubility in the range of acid to neutral cryolite-base melts has been modeled thermodynamically in terms of several oxyfluoride solutes. For an acidic melt, cryolite ratio (CR)=1.5, the dominant solute is monoxygen Na2Al2OF6. In a less acidic regime, dioxygen Na2Al2O2F4 is dominant, whereas for neutral compositions (CR=3) Na4Al2O2F6 starts to gain importance. The fit of the model to the experimental solubility data is virtually perfect. The values of the equilibrium constants for the formation of the individual solutes are reported.

2:50 PM

Solubility of Iron and Nickel Oxides in Cryolite-Alumina Melts: *Trond Eirik Jentoftsen*[1]; O. A. Lorentsen[1]; E. W. Dewing[2]; G. M. Haarberg[1]; J. Thonstad[1]; [1]Norwegian University of Science and Technology, Dept. of Mats. Tech. and Electrochem., Trondheim N-7491 Norway; [2]648 Pimlico Pl., Kingston, Ontario K7M 5T8 Canada

The solubility of divalent iron and nickel oxides was measured in cryolite-alumina melts at 1020°C. FeO and NiO were found to be the stable solid phases at low alumina concentrations, while $FeAl_2O_4$ and $NiAl_2O_4$ were stable at high concentrations. The alumina concentrations corresponding to the point of coexistence between FeO and $FeAl_2O_4$ and between NiO and $NiAl_2O_4$ were determined to be 5.03 and 3.0 wt% Al_2O_3, respectively. The results are discussed in terms of dissolution mechanisms. Experiments performed at alumina saturation in the range 980-1050°C showed that the solubilities of $FeAl_2O_4$ and $NiAl_2O_4$ increased with increasing temperature. The solubilities of $FeAl_2O_4$ and $NiAl_2O_4$ as a function of the molar cryolite ratio were investigated in alumina-saturated melts at 1020°C. For both compounds maximum solubility was found at a molar ratio of around 5, and the results are discussed with respect to dissolved species. Gibbs energies of formation and apparent partial molar enthalpies of dissolution for $FeAl_2O_4$ and $NiAl_2O_4$ were calculated.

3:15 PM

Coupled Current Distribution and Convection Simulator for Electrolysis Cells: *Knut Bech*[1]; Stein Tore Johansen[1]; Asbjørn Solheim[1]; Torstein Haarberg[1]; [1]SINTEF Materials Technology, Trondheim N-7465 Norway

A simulator for coupled current distribution and convection in electrolysis cells has been developed. The simulator solves the electric and temperature fields in the electrolyte, electrodes and surrounding solids, as well as the rate of anodic gas evolution and the resulting convection in the electrolyte. The simulator is based on the computational fluid dynamics software Fluent 4.5, to which we have added a solver for the electric field including electrochemical overvoltage. The overvoltage model is based on an iterative flux method. Furthermore, the amount of gas released from the anode is a function of the local current density. The resulting gas induced convection depends on gas mass flow and bubble size. The local electric conductivity of the electrolyte is a function of the local gas fraction. Calculations have been performed for various cell geometries with both horizontal and vertical electrodes. Effects of bubble size and cell geometry have been studied. The physical parameters were chosen to match those encountered in aluminium electrolysis, but the simulator is general and can be applied to all kinds of electrolysis cells. Additional calculations of side ledge heat transfer in Söderberg and prebaked cells are presented and compared with measurements.

3:40 PM Break

3:50 PM

Some Surface and Interfacial Phenomena Encountered in Aluminium Electrolysis: *Asbjorn Solheim*[1]; Sverre Rolseth[1]; [1]SINTEF Materials Technology, N-7465 Trondheim, Norway

Surface or interfacial tension causes several effects that are important for the understanding of the Hall-Heroult process. Some of these phenomena are analyzed in the present paper. A formula for the calculation of the meniscus formed at the metal-bath-sideledge boundary is derived. It is shown that large alumina-bath agglomerates may be present at the metal surface due to the interfacial tension. The propagation of capillary and gravity waves at the interface is addressed. Furthermore, it is shown that adsorption of water into commercial alumina conforms with the BET isotherm up to about 40-50 percent relative humidity, followed by the absorption of up to 20 weight percent water by pore condensation at water vapour pressures still below saturation. Marangoni convection can occur due to interfacial tension gradients between the parts of the cathode that are inside and outside the projection of the anode.

4:15 PM

Bath-Metal Interfacial Deformation Due to Gas Induced Flow in Aluminium Cells: *Torstein Haarberg*[1]; Espen Olsen[1]; Asbjørn Solheim[1]; Marc Dhainaut[1]; Pål Tetlie[1]; Stein Tore Johansen[1]; [1]SINTEF Materials Technology, Proc. Metall. and Cer. Div., Alfred Getz vei 2b, Trondheim N-7465 Norway

A model for the steady state deformation of bath-metal interface in aluminium cells is developed. The different factors considered in the model are density difference, gas induced flow pressure and interfacial tension between the two liquid phases. The model is applied in combination with computations of gas driven flow in aluminium cells to predict steady state interfacial deformation between bath and metal for different operating conditions and cell geometries. The predicted effect is verified in water model experiments. Measurements on full scale industrial cells show good agreement with results obtained both in a small-scale water model and with CFD-simulations. Steady state deformations up to ±20 mm have been recorded under normal operating conditions. In addition to the steady state deformation, gas induced non-permanent waves occur superpositioned on the bath-metal interface.

4:40 PM

Voltammetry and Electrode Reactions in AlF_3 Rich Cryolitic Electrolyte: *Richard G. Haverkamp*[1]; Sverre Rolseth[2]; Henrik Gudbrandsen[2]; Jomar Thonstad[2]; [1]Massey University, Instit. of Tech. and Eng., Private Bag 11222, Palmerston North 1015 New Zealand; [2]SINTEF, Matls. Tech. Div., Trondheim N-7465 Norway

Many aluminium smelters use a cryolite type electrolyte containing a cryolite ratio (CR, molar ratio of NaF/AlF_3) of 2.4 with around 5wt% CaF_2 (3.5mol% CaF_2). However there is a tendency by some smelters to move in the direction of higher AlF_3 content electrolyte. In this work we studied the influence of the electrolyte composition (CR 1.2-2.4) on the anodic reactions with fast linear sweep voltammetry. We observed a marked change in the anodic processes at high AlF_3 content. In the laboratory cell during voltammetry with standard electrolyte (CR 2.4) anode effect occurs around 2-5V with a rapid decrease in anode current (4 A/cm^2 to <0.1A/cm^2) which is maintained even at high (>10V) potentials. However with high AlF_3 electrolytes electrolysis took place at high applied potentials (7-10V) to produce large current densities (8-9A/cm^2 at 10V). CaF_2 had the effect of promoting the initiation and stability of the anode effect.

Aluminum Reduction Technology - Stream II: Advanced Control

Sponsored by: Light Metals Division, Extraction & Processing Division, Materials Processing and Manufacturing Division, Aluminum Committee, Pyrometallurgy Committee, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: Adrian Deneys, Praxair, Inc., Tarrytown, NY 10591 USA; Derek Fray, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3Q2 UK; Matt Krane, Purdue University, Department of Materials Engineering, West Lafayette, IN 47907 USA; Markus Reuter, Delft University of Technology, Applied Earth Sciences, Delft 2628 RX The Netherlands; Fiona Stevens McFadden, University of Auckland, Chemistry and Materials Engineering Auckland, New Zealand

Wednesday PM — Room: 230
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Fiona Stevens McFadden, University of Auckland, Chem. and Mats. Eng., Auckland, New Zealand; Matthew John M. Krane, Purdue University, Sch. of Mats. Eng., West Lafayette, IN 47907

2:00 PM

Control Electrochemical Cell Dynamics with Electrode Current Measurements: *James R. Barclay*[1]; [1]Universal Dynamics, #100-13700 International Place, Richmond, BC V6V 2X8 Canada

Knowing the electrical current distribution in a multiple-electrode, electrochemical cell, for example, a prebake aluminum reduction pot, provides significant insights into the cell dynamics. Metal pad movements, bath chemistry variations, anode spikes, bubble phenomena all affect the current distribution and efficiency of the cell. This paper describes a robust current sensing system that graphically shows cell dynamics. The current sensors are suitable for harsh reduction cell environments and are easily installed and maintained. The system can display real time data, analyze historical data and graphically replay cell events.

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Digital Processing of Anode Current Signals: An Opportunity for Improved Cell Diagnosis and Control: Graeme C. Barber[2]; *Jeffrey T. Keniry*[4]; Mark P. Taylor[3]; Barry J. Welch[4]; [1]Alumination Consulting Party, Ltd., 2, Governors Dr., Mt Macedon, Vic 3441 Australia; [2]Consultant, 5, Fullwood Pde, Doncaster East, Vic 3109 Australia; [3]Comalco Aluminium Limited, 12, Creek St., Brisbane, Qld 4000 Australia; [4]University of Auckland, Dept. of Chem. and Mats. Eng., Auckland, New Zealand

While digital signal processing (DSP) is now commonplace in many industrial applications, it has received surprisingly little attention or application in aluminium smelting. Despite advances in data acquisition and storage, line current and cell voltage remain the only signals that are continuously monitored for control of vital cell functions such as alumina feeding, thermal regulation and magnetic stability. But are we using these signals to their maximum potential for diagnosis and control of the process? A full complement of anode signals has been studied from industrial cells using high frequency (50Hz) sampling, with subsequent processing in time and frequency domains using the Fast Fourier (FFT) technique. While the imprinting of the metal surface motion is a well-known observation under low frequency sampling, this work shows that the higher frequency signals associated with bubble formation and release from the anodes also provide an imprint for specific process events and cell behaviour. The potential applications of DSP of individual anode and composite cell signals in diagnosis and control are discussed.

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Application of Advanced Process Control to Aluminium Reduction Cells–A Review: *Fiona J. Stevens McFadden*[1]; Geoffrey P. Bearne[2]; Paul C. Austin[3]; Barry J. Welch[1]; [1]Auckland University,

Chem. and Mats. Eng., Private Bag 92019, Auckland, New Zealand; [2]Comalco Research, P.O. Box 315, Thomastown, Victoria 3074 Australia; [3]University of Auckland, Elect. & Electr. Eng., Private Bag 92019, Auckland, New Zealand

The aluminium electrolysis process is fundamentally unchanged since its advent in the late 1880's. The control of the process has however, developed since then, with the trend being to increased mechanisation and automation. Process computers were implemented in the mid-1960's and currently the process is controlled with a mixture of automated and manual systems. In terms of control algorithms, although there have been refinements in use, the principles of the control strategies have not changed substantially in the last 30 years and control of the process outputs is in general achieved using single-input/single-output control loops. Through the application of advanced process control, which draws on elements from disciplines ranging from control engineering, signal processing, statistics, decision theory and artificial intelligence, performance improvements have been gained in other process industries. For aluminium reduction cells optimal control has been investigated along with artificial intelligence techniques such as fuzzy logic control, expert systems and neural networks for identification, prediction and control. Process simulators (physio-chemically derived dynamic models) have also been developed for the development and tuning of control strategies.

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A Multivariable Control of Aluminum Reduction Cells: *Kevin L. Moore*[1]; Nobuo Urata[2]; [1]Utah State University, CSOIS/ECE, 4160 Old Main Hill, Logan, UT 84322 USA; [2]Kaiser Aluminum and Chemical Corporation, Ctr. for Tech., Prim. Al Bus. Unit, 6177 Sunol Blvd., Pleasanton, CA 94566 USA

This paper considers control of the aluminum reduction process, using a dynamic model developed from the literature and Virtpot, a model developed at Kaiser Aluminum. Analysis shows the process is controllable and observable, but not easily stabilizable using one input, and that short-term changes in measured voltage result primarily from changes in alumina concentration rather than anode-to-cathode distance (ACD). Next, a multivariable control strategy is developed to regulate cell voltage by adjusting feed rate rather than beam movement. We introduce the idea of a feed voltage, obtained by subtracting expected voltage deviations due to ACD changes and beam moves from the filtered voltage. Feed rate is adjusted to compensate for deviations of feed voltage from its target. Simultaneously, beam movements are made to compensate for the difference in expected anode consumption and metal pad rise, based on changes in feed period. Simulations show the effectiveness of the proposed control strategy.

3:40 PM Break

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From Cell Models to a Virtual Potroom: *Laszlo G. Tikasz*[1]; Rung T. Bui[1]; Vincent Villeneuve[1]; Sylvain Doyon[1]; [1]University of Quebec at Chicoutimi, Dept. of Appl. Sci., 555 University Blvd., Chicoutimi, Quebec G7H 2B1 Canada

This paper analyzes the 'virtual cell' concept in which dynamic models of aluminium electrolytic cells are used as training, operation support and research tools in aluminium reduction plants. A systematic approach is proposed to transform a chosen cell model into a virtual cell that exchanges data with its environment like a real cell does. Then, a method is given to clone a virtual cell to form a group of cells or even populate an entire 'virtual potroom'. The virtual cells-like the real ones-are identical by design but individual in performance. Every one of these virtual cells is under automatic control. The controllers, organized into a hierarchical scheme, can be real or virtual. Examples are given for various cell and controller arrangements simulating typical cell operations in a plant.

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Virtual Aluminum Reduction Cell: *V. V. Yurkov*; V. Ch. Mann; T. V. Piskazhova; K. F. Nikandrov

At the TMS Annual Meeting of 2000 the "Model of Process of Electrolysis" was presented. Applying this dynamic mathematical model it was possible to imitate the operation of the industrial cell as the first approximation. After the conference this work was continued. A number of active and passive experiments were conducted. Specially designed diagnostic equipment was used for measuring and recording into a database temperature regimes of the cell different units. At the same time in order to identify the model some calculations on the model were carried out (equations selected and coefficients adjusted). This paper presents the results of the performed work, describes a "virtual cell", created on the basis of the dynamic model and a "virtual control system", which is the mathematical twin of an electrolysis control system operated at KRAZ.

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Development of Fuzzy Expert Control Technique for Aluminum Electrolysis: *Jie Li*[1]; *Fengqi Ding*[1]; Zhong Zou[1]; Minjun Li[1]; *Yexiang Liu*[1]; *Youkang Bian*[2]; Zhiming Wu[2]; *Gang Liu*[2]; [1]Central South University of Technology, Dept. of Metall. Sci. and Eng., Changsha, Hunan 410083 China; [2]Qinghai Aluminum Corporation, Xining, Qinghai 810108 China

In order to upgrade the basic control unit called "cell controller" in process control systems of aluminum electrolysis, a new control algorithm called "Fuzzy Expert Controller (FEC)" was developed. Because all the knowledge and experience that can be collected from the field experts as well as all the information which can be sampled for the analysis and control of cell state are not precise, the FEC was designed as a rule-based system working with rules in which imprecise and precise propositions were mixed freely. Its self-regulation mechanism adjusted the universes of discourse of fuzzy variables according to the change of cell state and the transition of control modes, achieving the object of on-line modifying its operating points and dynamic and static performance. Application results on smelters showed that the control accuracy, robustness and stability were satisfactory, and remarkable effects of production increasing and energy saving were achieved.

Bauxite Residue Treatment: New Development: Session 1

Sponsored by: Light Metals Division, Aluminum Association
Program Organizer: John A.S. Green, The Aluminum Association, Washington, DC 20006-2168 USA

Wednesday PM Room: 217
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chair: John A.S. Green, Aluminum Association, Washington, DC 20006 USA

2:00 PM

Causticization System of Pond Water in CVG-Bauxilum: *Néstor Andrés Velasquez*[1]; *Saúl Escalona*[1]; [1]CVG-Bauxilum, Control de Calidad, Zona Industrial Matanzas, Puerto Ordaz, Bolivar 8015 Venezuela

CVG-Bauxilum alumina plant, is always making efforts in the search of solutions to control the inventory of liquids in its mud disposal ponds. With this objective in mind, it has been conceived an operation which basic concept is to return a maximum flow of pond water to the plant without jeopardising its process. Operation establishes the rearrangement of flows within the various ponds dedicated to sand and mud disposal, leaving the sand pond solely to the collection of excess condensate and liquor plant spillage. Under this conditions a relatively clean pond liquor, that is low in concentration of impurities when compared to the others ponds liquids, is left for the causticization of 200 m3/h where carbonates are reduced to almost zero. This flow is returned to the plant and used in process areas. The benefits are expressed in a more favourable water balance around the ponds and higher caustic recovery.

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Filtering Dike: Cruz de Jesús Silva[1]; [1]CVG-Bauxilum, Manejo de Lodo, Zona Industrial Matanzas, Puerto Ordaz, Bolivar 8015 Venezuela

CVG-Bauxilum discharges, as waste of its refinery of Alumina, a mixture of caustic liquids and fine solids. These solids, known as mud, occupied space that affected the useful life of the storage lagoons. It was decided to build a filtering dike where the two phases of the mixture can separate, preventing the solids to arrive to the ponds. The only available space for the location of the dike was the beach of the pond # 3, with nine meters of mud thickness and a practically null support capacity. Under this condition it was required the usage of sand as construction material, leading to the second advantage since the sand pond was nearly exhausted in its capacity. Seven meters of sand were placed, three of those which were forced to penetrate in the mud for their sustentation and build a drainage system that allows the passage of liquid but not the solids. In three years of operation of the filtering dike the prospective results have been obtained.

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Construction of a Pilot Plant for the Neutralization of Water Pond: Ricardo Alfredo Galarraga[1]; [1]CVG-Bauxilum, Manejo de Lodo, Zona Industrial Matanzas, Puerto Ordaz, Bolivar 8015 Venezuela

CVG-Bauxilum, as part of its plan to find a definitive solution to avoid the continuous increment in the liquid levels in the waste deposit pond, has established a relationship with the Universidad Simón Bolívar to treat the water pond and to be able to produce a clean effluent from the environmental point of view. This institution carried out a novel process which has given excellent results at laboratory levels, obtaining a reduction of the pH of the pond water from 12 down to approximately 8. This pH reduction, accompanied by some suspended solids inferior to the permitted limit for pouring liquids into natural waters currents, has generated the necessity to build and to evaluate results in a pilot plant of semi-industrial scale, projected to begin operations by July 2000. The results are guaranteed by the addition of a solid reactant, the strict control of the temperature conditions, time of residence and pressure, as well as the gassy final addition of CO2.

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Recovery and Utilization of Iron from Red-Mud: *Brajendra Mishra*[1]; Anthony Staley[1]; David Kirkpatrick[2]; [1]Colorado School of Mines, Metall. and Matls. Eng., 1500 Illinois St., Golden, CO 80401 USA; [2]Kaiser Aluminum and Chemical Corporation, P.O. Box 3370, Gramercy, LA 70052 USA

Red mud is the primary waste product of the alkaline extraction of alumina from bauxite ore [Bayer Process]. The red-mud generated from the processing of Jamaican bauxite is rich in hematite. It has been shown that hematite can be carbothermically reduced with a degree of metallization of over 94 pct. Iron can be separated from the reduced product magnetically with limited success. Both calcium and titanium oxides have a tendency to contaminate the separated iron metal. The magnetic separation is significantly improved when the trihydrate residue is reduced. Trihydrate material contains less lime. Calculations have been done to suggest that reduced material could be charged through the tuyeres of an iron blast furnace or smelted to produce pig iron. If smelted, the concentration of titanium oxide in the slag can be significantly high justifying its recovery by an acid-leach process. However, the acid-leach process can not applied in the presence of iron. This work will describe the successful efforts of iron and alumina recovery from red mud. The problems associated with the use of reduced red-mud, as an alternative to direct-reduced iron [DRI], will be discussed. Critical assessment of the recovery sequence chosen for the products will be described based on energy requirements.

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Low-Temperaure Reduction of Ferric Iron in Red Mud: Qinfang Xiang[1]; *Mark E. Schlesinger*[1]; John L. Watson[1]; Xiaohong Liang[1]; [1]University of Missouri-Rolla, Dept. of Metall. Eng., 1870 Miner Circle, Rolla, MO 65409-0340 USA

Previous proposed methods for removing the iron from red mud have focused on either DRI-type processing or blast furnace smelting. A new iron-removal process features low-temperature reduction of ferric iron content to magnetite, followed by magnetic separation. The results of reduction experiments using coal, charcoal, sawdust and bagasse as solid-state reducing agents are described. Other variables included the type of mud used (three U.S. producers), reduction time and temperature, and the mud/reductant mass ratio. Sawdust and bagasse are the better reductants, and complete reduction to magnetite can be achieved at temperatures as low as 350°C. Conversion of the ferric iron to magnetite is strongly dependent on the mud/reductant ratio, suggesting that pyrolysis of the reductant is the controlling factor in the reduction process. Initial experiments on recovery of the magnetite generated by the reduction process will be described.

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Smelting Reduction of Red Mud for the Recovery of Iron and Titania: Slag/Metal Equilibrium Studies: Animesh Jha[1]; Srikanth Srinivasan[2]; Amitava Bandopadhyaya[3]; Thomas C. Alex[3]; [1]University of Leeds, Dept. of Mats., Clarendon Rd., Leeds LS2 9JT UK; [2]National Metallurgical Laboratory Madras Centre, CSIR Madras Complex, Post TTTI, Tharamani, Chennai, Tamilnadu 600 113 India; [3]National Metallurgical Laboratory, Ferr. Proc. Div., P.O. Burma Mines, Jamshedpur, Bihar 831 007 India

The possible utilisation of red mud for the recovery of iron as cast iron and titanium as synthetic rutile has been explored. Laboratory-scale reduction smelting experiments were carried out on mixtures of red mud and iron scrap for this purpose. Both, low TiO2 red mud (ALCAN, UK) as well as high TiO2 red mud (INDAL, India) were tested. Reduction smelting was followed by isothermal slag-metal equilibration in the temperature range 1400-1600ºC. The alloy and slag compositions obtained experimentally were compared with those obtained by thermochemical modeling. The unified interaction parameter formalism was adopted to describe the thermodynamic properties of the alloy phase and a regular solution as well as the modified quasi-chemical model was used for the slag phase. The experimental results as well as theoretical calculations indicate that it is possible to extract the iron as an alloy and segregate most of the titanium to the slag. The titanium distribution between the metal and slag increases with increase in temperature. The slag can subsequently be processed for the production of pigment grade TiO2.B

Carbon Technology: Cathode Performance

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Morten Sorlie, Elkem ASA Research, Vaagsbygd, Kristiansand N-4675 Norway; Les Edwards, CII Carbon, Chalmette, LA 70004 USA

Wednesday PM — Room: 215-216
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Harald A. Oye, Norwegian University of Science & Technology, Dept. of Inorganic Chem., Trondheim N-7034 Norway

2:00 PM Introductory Comments

2:05 PM Invited
A Model of Degradation of Carbon Cathodes by Sodium: *Harry Marsh*[1]; Maria-Antonia Diez[1]; [1]Instituto Nacional del Carbon (INCAR), Oviedo E-33080 Spain

Degradation of cathodes by sodium decreases with increasing graphitizability of the constituent carbon. Modelling degradation has two components: (I) Structural-including (a) the aromaticity of the graphene clusters, (b) the three-dimensional bonding within the graphene clusters, (c) accessible porosity, and (d) 'parallelism' within the graphene clusters. The second component is: (II) Kinetic and Mechanistic-modelling the sequences of degradation of cathode carbon by sodium including: (i) generation of sodium vapour, (ii) penetration (activated diffusion) of sodium into the 'so-called' closed

porosity, (iii) penetration of sodium (intercalation) between the graphene sheets so causing internal expansion of the graphene clusters, and (iv) generation of significant internal stresses within the cathode carbon sufficient to cause degradation. More graphitic carbons have less internal cross-linkages and porosity and can better accommodate reduced internal stresses. Significant differences exist in extents of degradation of carbons which have only small differences in their optical textures.

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Electrolytic Degradation Within Cathode Materials: *Parin Rafiei*[1]; Frank Hiltmann[2]; Margaret M. Hyland[1]; Bryony James[1]; Barry J. Welch[1]; [1]University of Auckland, High Temp. Mats. and Process. Grp., Dept. of Chem. and Mats. Eng., Private Bag 92109, Auckland, New Zealand; [2]SGL Carbon GmbH, Griesheim Plant, Stroofstasse 27, D-65933 Frankfurt, Germany

There are two generally accepted electrochemical processes involving cathode carbon, sodium uptake and aluminium carbide formation. Both processes occur at or below the potential required for aluminium deposition, and therefore can occur at all times in blocks permeated with electrolyte. Since the cathode carbon is at a more cathodic potential than the molten aluminium, the electrolyte filled material can sustain the electrochemical reactions, even though the rate may be very low. A laboratory test has been developed to identify and ascertain the extent of the carbide forming reaction within the carbon matrix of laboratory cathode samples. Results show that the reaction is dependent on the binder pitch, the carbon filler material and the cathode heat treatment. This electrochemical reaction is expected to become more prevalent at higher current densities and the growth of the carbide can potentially contribute to accelerated wear of the cathode.

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Wetting of Carbonaceous Cathode Material in the Presence of Boron Oxide: *Rudolf Keller*[1]; Julian V. Copenhaver[2]; Richard O. Love[3]; Allen Barkley[4]; David Huff[5]; [1]EMEC Consultants, Schreiber Industrial District, Bldg. 242, 2nd Floor, New Kensington, PA 15068 USA; [2]NSA Division of Southwire Company, 1627 State Rt. 271 N, P.O. Box 500, Hawesville, KY 42348 USA; [3]Century Aluminum of West Virginia, P.O. Box 98, Ravenswood, WV 26164 USA; [4]Northwest Aluminum Company, 3313 West Second St., The Dalles, OR 97058 USA; [5]Great Lakes Carbon, Germany

In the electrolytic aluminum production, changing the characteristics of the interface between carbonaceous cathode material and molten aluminum to achieve wetting of the carbon by the metal is desirable. Tests were performed in the laboratory and in industrial cells with cathode material containing boron oxide. When exposed to molten aluminum with a titanium content, wetting by the metal occurred, presumably due to in-situ formation of titanium diboride. The viability of applying this concept to improve the performance of aluminum production cells is being explored with the support of the Department of Energy's Office of Industrial Technologies.

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Usage of a Full 3D Transient Thermo-Electric F.E. Model to Study the Thermal Gradient Generated in the Lining During a Coke Preheat: *Marc Dupuis*[1]; [1]GéniSim, Inc., 3111 Alger St., Jonquiére, Québec G7S 2M9 Canada

Ten years ago, a full 3D transient thermo-electric F.E. model was developed to study the thermal gradient generated in the cathode lining during coke preheat [1,2]. The model development and applications turned out to be very costly because the model had to be run on expensive supercomputers. But nowadays, this type of model can easily be run on inexpensive Pentium III computers. This paper presents a typical model application for a modern prebaked cell running at 400 kA.

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Investigating Cathode Stress-Deformed State in the Aluminum Electrolysis Cell: *Vitaly Pinguin*[1]; G. Arkhipov[2]; [1]Krasnoyarsk Aluminium Plant, Krasnoyarsk Russia; [2]Krasnoyarsk State Acad. of Non-Ferr. Mets. and Gold, Krasnoyarsk, Russia

Improper design of cathode linings can result in early cell failures through destruction of the bottom insulation of the cell. The paper describes a 3D mathematical model used to evaluate the stress deformed state of a cell incorporating the following elements: cathode blocks, collector bars, cast iron, joints and peripheral seams, thermal insulation and refractory layers, compensators for bottom expansion, ramming paste, shell stiffeners, cathode shell type (split or welded) etc. The deformation and stress mechanism was simulated based on the thermal expansion of the cell lining materials and cathode shell, sodium expansion of the cathode and volume increase of the insulation materials due to salt formation. The model was used to analyze the influence of material property, cell design and cell construction changes on the cell stress-deformed state. The paper presents the main requirements to cell design and materials to prolong cell life.

Cast Shop Technology: Degassing and Alkali Metal Removal

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: John F. Grandfield; CSIRO Australia, Preston, Victoria 3072 Australia; Paul Crepeau, General Motors Corporation, 895 Joslyn Road, Pontiac, MI 48340-2920 USA

Wednesday PM Room: 208-210
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: John J. Chen, University of Auckland, Dept. of Chem. & Mats. Eng., Private Bag 92019, Auckland, New Zealand; Malcolm Couper, Comalco Research & Technical Support, 15 Edgars Rd., Thomastown, Victoria 3074 Australia

2:00 PM

Modeling of Rotary Injection Process for Molten Aluminum Fluxing: *Jean-François Bilodeau*[1]; Carl Lakroni[1]; Yasar Kocaefe[2]; [1]Alcan International, Ltd., Arvida R&D Laboratory, P.O. Box 1250 (1955 boul Mellon), Jonquière, Québec G7S 4K8 Canada; [2]Université du Québec à Chicoutimi, Dept. of Appl. Sci., 555 boul Université, Chicoutimi, Québec G7H 2B1 Canada

Over the past five years, industrial implementation of the rotary gas/flux injection technology (RGI/RFI) has proven to be a metallurgical and environmental solution to the traditional chlorine injection with lances. In order to understand, predict and support optimization of the process performance, mathematical modeling of the rotary flux injection process in a holding furnace has been developed. The three-dimensional, two phase flow model calculates the velocities of molten metal and gas in the whole liquid bath, taking into account impeller rotation and gas buoyancy. A chemical reaction controlled by mixing was also incorporated. Indexes of global furnace stirring and reactant dispersion were calculated for holding furnaces of various configurations. A good correlation was obtained between calculated dispersion indexes and measured alkali removal kinetics.

2:25 PM

Efficiency Modeling of Rotary Degasser Head Configurations and Gas Introduction Methods, Part I: Water Tank Tests: *Michael J. Hanagan*[1]; Kevin Carpenter[1]; [1]Blasch Precision Ceramics, Res. and Dev. 580 Broadway, Albany, NY 12204 USA

The use of ceramic materials for rotary degasser heads and shafts is on the increase and a wide variety of head designs and methods of gas introduction (e.g. holes versus porous medium) are now available. This paper attempts to rank the efficiency of various head designs and gas introduction methods by measuring the removal of dissolved oxygen from water at room temperature. Several rotor head designs and gas introduction configurations were tested under a variety gas flow rates and rotational speeds to determine the rates of oxygen removal and the ultimate residual oxygen levels.

2:50 PM

Flow Pattern Detection in a Melt Treatment Water Model Based on Shaft Power Measurements: *Jianchao Zhao*[1]; John J.J. Chen[1]; P. V. Lacey[1]; Tom N.H. Gray[1]; [1]The University of Auckland, Dept. of Chem. & Mats. Eng., Private Bag 92019, Auckland, New Zealand

The rotor power consumption was measured in a full-scale water model of a proprietary molten aluminium treatment unit. The bubble dispersion patterns at various gas flow rate and rotor speed were observed and compared to the changes in the rotor power as obtained from the torque measured on the rotor shaft. A plot of the gassed power to the ungassed power ratio versus the rotor Reynolds Number shows a definite trend which is related to the flow pattern, and this behaviour can be used to provide a non-intrusive method of detecting the flow pattern. This is a general methos and is expected to be applicable in controlling operation conditions. The Power Number-Reynolds Number characteristics of the rotor are also reported. Furthermore, the gassed power was correlated with the ungassed power using the Michel-Miller type of correlation that is commonly used in agitated gas-liquid dispersion systems.

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The Use of a Complex Gaseous Mixture for the Liquid Treatment of Al-Si-Cu Alloys: *Alfredo Flores Valdes*[1]; Jose C. Escobedo Bocardo[1]; Pedro E. Garcia De la Peña[1]; Jose M. Almanza Robles[1]; [1]CINVESTAV, Metall. Eng., Carretera Saltillo-Monterrey Km 13.5 Colonia Molinos del Rey, Ramos Arizpe, Coahuila 25900 Mexico

The liquid treatment of Al-Si-Cu alloys has been performed for many years at an industrial scale, to achieve a high melt quality. In the specific case of elements removal from the melt, many different techniques and chemical products have been employed, with many variations in performance, from very low to very high efficiencies. Na2SiF6 has been reported as one of the most efficient compounds to remove magnesium from the melt. However, it has been stablished that this compound decomposes into NaF and SiF4 prior to react with the magnesium dissolved. In this work, the use of a complex mixture of gases for the liquid treatment of an aluminum alloy is reported, having determined a very good performance of a mixture which contained an small amount of SiF4, as very low porosity, very small inclusions content and low final magnesium contents were achieved.

3:40 PM Break

3:50 AM

Recent Advances in Gas Injection Technology Using Molten Metal Pumps: *Richard S. Henderson*[1]; David V. Neff[1]; Chris T. Vild[1]; [1]Metaullics Systems, 31935 Aurora Rd., Solon, OH 44139 USA

Inert and reactive gases are employed in molten aluminum processing to serve several needs: magnesium reduction, degassing, alkali metal removal, and inclusion flotation. Growing environmental concern and the necessity to develop and manage melt treatment processes for better efficiency have dicated continuing improvements in process technology. This paper focuses on the gas injection pump and its use for metallurgical refining. In particular, recent advances have been made in discharge and nozzle configuration and injection technique to achieve increased gas/metal mixing efficiency, which has resulted in higher productivity coupled with environmental acceptability. Laboratory and field test performance data will be presented, especially as it pertains to scrap melting/metal recycling systems. Gas injection processes in melting and holding furnaces greatly improve process efficiency, metal recovery, and molten metal quality, while lessening the burden on downstream gaseous refining processes.

4:15 PM

Improvements in Cast Shop Processing Using Pyrotek's HD-2000 and PHD-50 Rotary Injector Systems: Peter Flisakowski[1]; *Mickey McCollum*[2]; [1]Pyrotek, Inc., 9503 East Montgomery Ave., Spokane, WA 99206 USA; [2]Pyrotek, Inc., SNIF Systems, 1660 Sperry's Forge Trail, Westlake, OH 44145 USA

Ever increasing demands for high quality aluminum creates the need for continuous improvements in the melt treatment processes. Pyrotek has developed equipment to enhance and improve molten metal treatment in holding furnaces. Pyrotek's HD-2000 and PHD-50 units are designed to replace conventional furnace treatments, such as degassing tablets, porous plugs, steel flux wands and graphite flux tubes. In addition the HD-2000 and PHD-50 can also be used to inject solid fluxes and for metal circulation in furnaces. Both units are fully automated. While the PHD-50 unit offers all the same advantages as the HD-2000, it is a portable unit, and more suited for smaller installations. This presentation discusses several cast shop equipment trials in melting and holding furnaces to demonstrate the benefits of the PHD-50 and HD-2000 treatment systems. The performance of the equipment was compared to conventional treatment methods. Improvements of these conventional methods are documented.

4:40 PM

Crucible Fluxing of Potroom Metal in a Norsk Hydro Cast Shop Effect on Dross Reduction and Increased Metal Recovery.: *Trygve Leinum*[1]; [1]Hydro Aluminium Sunndal, Cast Shop, 6600 Sunndalsora, Norway

Aluminum from electrolysis at Hydro Aluminum Sunndal contains approximately 50 ppm Na. New equipment for fluxing the potroom metal with AlF3 and Argon directly in the tapping crucible, was installed in October 1998. In The Crucible Fluxing Process, a half-life period for Na of 2 minutes is obtained. The Na content is reduced to typically 1-5 ppm before the metal is charged into the casting furnace. The process has caused 20 pct reduction in the amount of dross in the Cast Shop. Today, the dross amounts to less than 1 pct of the metal flow. In addition, the metal recovery from dross has increased from approximately 50 to 75 pct. The net metal loss is reduced from approximately 0,4 to 0,2 pct of the total metal production. The reduction in metal loss (i.e. burn up) may be explained by removal of Na as a catalyst in the oxidizing process.

5:05 PM

Alkali Removal and Reduced Chlorine Use during Furnace Fluxing: *Edward M. Williams*[1]; [1]Alcoa, Ingot and Solidification, 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

A series of furnace fluxing trials was performed at the Reynolds Casting Research Center as part of a DOE funded NICE3 project. The purpose of this work was to characterize the alkali removal efficiency of three different chlorine/inert gas fluxing methods; flux wands, porous plugs and a Pyrotek PHD-50 in-furnace spinning degasser. In addition to determining the relative alkali removal efficiencies of these three methods, the alkali decay curves have been used to develop a model describing the optimum chlorine addition rate during fluxing. This model has been verified for all three fluxing methods by measuring the excess hydrogen chloride in the furnace stack during fluxing using a FTIR emissions monitor. This work has also shown that it is possible to use the FTIR to control the chlorine input rate to the furnace flux such that HCl emissions are minimized and alkali removal rates are maximized.

Chemistry and Electrochemistry of Corrosion and Stress Corrosion: A Symposium Honoring the Contributions of R.W. Staehle:
Stress Corrosion Cracking of Iron and Nickel Based Alloys

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee, Jt. Nuclear Materials Committee

Program Organizer: Russell H. Jones, Battelle Pacific Northwest National Laboratory, Richland, WA 99352 USA

Wednesday PM Room: 222
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Peter L. Andresen, GE Corporate R&D Center, Schenectady, NY 12309 USA; Stephen M. Bruemmer, Pacific Northwest National Laboratory, Mats. Scis. Dept., Richland, WA 99352 USA

2:00 PM

Review of Problems with Material Selections in the Nuclear Power Industry: *Jeffrey A. Gorman*[1]; Roger W. Staehle[1]; [1]Dominion Engineering, Inc., 6862 Elm St., Suite 460, McLean, VA 22101 USA

The objective of this review is to evaluate the history of several of the serious problems that have occurred with materials in the nuclear power industry to identify the lessons that should be learned to prevent recurrence. The problems that will be reviewed include (1) use of Alloy 600 (Inconel 600) for steam generator tubes and reactor coolant system nozzle applications in pressurized water reactors (PWRs), (2) use of normal grade austenitic stainless steels as a major pressure boundary and structural material in (BWRs), and (3) use of Alloy 750-X (Inconel 750-X) for high strength bolting and structural applications in the reactor coolant systems of both PWRs and BWRs. The history of other materials will also be briefly reviewed. This review will concentrate on identifying the technical bases that were used at the time of material selection, what service experience has shown to be the problems with these original technical bases, and what lessons should be learned from these problems.

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Alloy 600 Stress Corrosion Crack Growth Rate Testing and Analytical Electron Microscopy as a Function of Pourbaix Space: *Nathan Lewis*[1]; D. S. Morton[1]; S. A. Allanasio[1]; G. A. Young[1]; [1]Lockheed Martin, P.O. Box 1072, Schenectady, NY 12301 USA

An alloy 600 stress corrosion crack (SCC) growth rate study and corresponding Analytical Electron Microscopy (AEM) investigation has been performed. This effort was conducted to characterize alloy 600 crack growth rate and subsequent fracture morphology as a function of Pourbaix space (i.e., pH and electrochemical corrosion potential -EcP). Three alloy 600 heat treatments were investigated: 1) mill annealed at 980°C (MA), (25 mm grain size, with ~0.2 mm M7C3 carbides, slight Cr depletion), 2) MA+980°C for seven days with water quench (25 mm grain size, ~0.5 mm M7C3 carbides, with no Cr depletion, and 3) MA+980°C for 7 days with water quench +607°C for 7.5 hours (25 mm grain size, -0.5 µm and smaller M7C3 carbides with significant Cr depletion). Extensive elevated temperature testing was conducted at near neutral pH as a function of EcP (controlled via aqueous hydrogen addition) in the low potential SCC regime (LPSCC). Additionally, testing was conducted under 10% caustic and mildly acidic oxidizing test conditions. Rapid crack growth rates (~25 µm/hr at 288°C) were observed for Cr depleted alloy 600 under oxidizing test conditons. However, non-Cr depleted alloy 600 crack growth (~0.4 µm/hr at 307°C) has been observed under caustic test conditons. Under 10% caustic conditions NixFe1-xCr2O4 fills the cracks and carbides are frequently observed to be completely oxidized. A maximum LPSCC alloy 600 crack growth rate (~ 0.5 µm/hr at 338°C) is observed in proximity to the Ni/NiO phase transition. Under these conditions NiO and mixed spinels are observed at crack tips however, carbides are never completely oxidized.

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Hydrogen Embrittlement Mechanism for Low Potential Stress Corrosion Cracking of Nickel Base Alloys: *Meryl M. Hall, Jr.*[1]; D. M. Symons[1]; [1]Bettis Atomic Power Laboratory, Bechtel Bettis, Inc., P.O. Box 79, West Mifflin, PA 15122 USA

Hydrogen enbrittlement (HE) is generally accepted as a mechanism for the embrittlement of nickel base alloys, such as Alloy X-750 and Alloy 690, in hydrogenated pure water in the low temperature range of 298K to 395K. However, the role of hydrogen in the static load cracking of these and other nickel base alloys such as Alloy 600 at the higher temperatures of pressurized water reactor (PWR) operation is controversial. In this paper, the experimental evidence for a HE mechanism of low-potential stress corrosion cracking (LPSCC) is reviewed and a model applicable to both low and higher temperature crack advance due to HE is developed. In the model developed here, corrosion of the exposed metal at crack tips results in the production of hydrogen by the reduction of water. Nascent hydrogen enters through the crack tip where it is trapped and concentrated at grain boundaries in the surrounding strain field. Interstitial hydrogen localizes and intensifies the crack tip strain rate while trapped hydrogen reduces the strain required to initiate fracture. This HE crack advance mechanism is incorporated into a previously reported dislocation creep model for LPSCC with the result that hydrogen diffusion is rate controlling at lower temperatures and the creep rate is rate controlling at higher temperatures. The model is applied to crack growth rate (GCR) data obtained on Alloy X-750 and Alloy 600. Features of the current version of the model are the ability to model the effects on CGR of carbon concentration, yield stress and crack orientation relative to the principal metal working direction. The model also includes explicit descriptions of the effects on CGR of electrochemical potential (hydrogen over potential) and pH.

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The Mitigation of Stress Corrosion Cracking of Boiling Water Reactor Structural Components: *Robert L. Cowan*[1]; [1]GE Nuclear Energy, 2273 St. Charles Ct., Livermore, CA 94550 USA

The Boiling Water Reactor (BWR) utilizes pure water at a temperature of 288ºC as the working fluid to provide steam directly to a turbine. Because of the nuclear fission, the deposition of neutron and gamma energy into the water causes significant radiolysis, producing a steady state concentration of oxygen, hydrogen and hydrogen peroxide. The hydrogen peroxide is not nearly as volatile as either hydrogen or oxygen and the net result is an environment containing 200 to 1000 ppb of oxidant (oxygen plus hydrogen peroxide) and a stochiometric deficiency of hydrogen. The resulting oxidizing environment is capable of initiating and propagating stress corrosion cracking in structural components constructed of stainless steel or high nickel alloys if they are in a metallurgically susceptible condition. This paper will review the basis for the chemical and electrochemical methods developed to successfully mitigate the stress corrosion cracking caused by this phenomenon.

4:00 PM

Electrochemical Study of Type 304 Stainless Steel Corrosion in High Temperature High Pressure Water: *June (James) B. Lee*[1]; Arun K. Agrawal[2]; Roger W. Staehle[3]; [1]JLG Industries, Inc., Mats. & Corr. Eng., 1 JLG Dr., McConnellsburg, PA 17233-9533 USA; [2]CC Technology, 6141 Avery Rd., Dublin, OH 43016 USA; [3]University of Minnesota, 22 Red Fox Rd., North Oaks, MN 55127 USA

Corrosion properties of Type 304 stainless steel was studied in high temperature high pressure water under laboratory simulated boiling water nuclear reactor conditions. The study consisted of steady state open circuit potential measurements in de-ionized water at various oxygen concentrations and electrochemical polarization study of the steel in 0.1 and 0.01N sodium sulfate solutions at 250ºC. The polarization study was also conducted at various dissolved oxygen concentrations. By combining the two studies, an attempt was made to construct a polarization curve of the steel in high purity water at 250ºC.

4:30 PM
Best Ways to Prevent SCC in the Chemical Process Industries: *Katsumi Yamamoto*[1]; [1]JGC Corporation, Techn. & Busin. Dev. Div., 2205 Narita-cho, Oarai-machi, Ibaraki Pref. 311-1313 Japan

Many SCC failures have been reported in chemical process industries. Many countermeasures on SCC have been also reported from various technical fields, such as SCC resistant materials, PWHT (post weld heat treatment) to reduce residual stresses on welds, coating systems to isolate materials from environments, chemical injection to change SCC environments, and others. However, the important factor to select the countermeasure which is the most applicable way is to know the detailed analytical results of SCC failure occurred in actual plants. The author has been solving SCC in chemical process industries based on the combination of laboratory's data and failure analysis. This time, chloride SCC on type 304SS, polythionic acid SCC on stabilized SS at HDS plant and SCC on carbon steels in amine plants will be discussed in details in terms of laboratory's data and failure analysis.

Computational Thermodynamics and Materials Design: Phase Equilibria and Phase Transformation III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Jt. Computational Materials Science & Engineering, Thermodynamics & Phase Equilibria Committee

Program Organizers: Zi-Kui Liu, Penn State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; Ibrahim Ansara, LTPCM-Enseeg, Grenoble, France; Alan Dinsdale, National Physical Laboratory, UK; Mats Hillert, Royal Institute of Technology, Materials Science & Engineering, Stockholm 10044 Sweden; Gerhard Inden, Max-Planck Institute-Duesseldorf, Düsseldorf D-40074 Germany; Taiji Nishizawa, Tohoku University, Japan; Greg Olson, Northwestern University, Dept. MSE, 2225 N. Campus Dr., Evanston, IL 60208 USA; Gary Shiflet, University of Virginia, Dept. of Matls. Sci. & Eng., Charlottesville, VA 22903 USA; John Vitek, Oak Ridge National Laboratory, Oak Ridge, TN USA

Wednesday PM, February 14, 2001 — Room: 201, Location: Ernest N. Morial Convention Center

Session Chair: Gary J. Shiflet, University of Virginia, Mats. Sci. and Eng., Charlottesville, VA 22904 USA

2:00 PM
Modelling and Synthesis of Ceramic Materials Under Hydrothermal Conditions Using Computational Thermodynamics: *Charles S. Oakes*[1]; Kullaiah Byrappa[1]; Wojciech L. Suchanek[1]; Margaret M. Lencka[2]; Richard E. Riman[1]; [1]Rutgers University, Dept. of Ceram. and Mats. Eng., 607 Taylor Rd., Piscataway, NJ 08854 USA; [2]OLI Systems, Inc., 108 American Rd., Morris Plains, NJ USA

Stability fields for of a variety of ceramic oxides (e.g. lead and alkaline-earth titanates and zirconates, lead zirconium titanate solid solutions, alkaline bismuth titanates, and alkaline-earth phosphates) have been estimated over wide ranges of pH, temperature, composition, and ionic strength using a robust thermochemical model for the calculation of activity coefficients and standard state properties. The thermodynamic model also serves as a tool for optimizing material yields. Phase fields have been experimentally verified for each material over wide ranges of synthesis conditions. In this presentation we will present our recent work on phase-pure hydroxyapatite particle synthesis. Thermodynamic computations have been used to define the region of optimal yield within the range 25 to 200ºC, pH's between 2 and 9, and at the vapor-pressures of the aqueous solutions. Experiment has verified that the minimum pH at which 99% of the calcium in the system is precipitated as hydroxyapatite is lowered from 4 at 100°C to 3 at 200°C.

2:25 PM
Computational Thermodynamics and the Kinetics of Lath Martensitic Transformation: *Gautam Ghosh*[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

Renewed interest in designing new materials with lath martensitic microstructure, such as ultrahigh strength steels, martensitic stainless steels, power plant steels for higher operating temperatures, low activation martensitic steels for fusion reactor applications etc. calls for a mechanistic model to predict martensitic transformation kinetics in multicomponent alloys. We will present the role of computational thermodynamics in predicting the transformation kinetics giving lath martensitic microstructure in Fe-base alloys. Both the martensite start temperature (Ms) and the overall transformation kinetics will be addressed. A multicomponent database (Fe-Al-C-Co-Cr-Cu-Mn-Mo-N-Nb-Ni-Pd-Re-Si-Ti-W-V) has been developed to predict martensitic transformation kinetics. The salient features of this database will be presented. Utilizing multicomponent thermodynamics, the model for composition and temperature dependence of shear modulus and a set of unique kinetic parameters, we will demonstrate that it is possible to predict the martensitic transformation kinetics with good accuracy in multicomponent alloys by incorporating heterogeneous nucleation theory.

2:50 PM
New Developments in Surface Roughness Measurements-Characterization of Chrome 13 Pipes: *Fred F. Farshad*[1]; [1]University of Louisiana, Dept. of Chem. Eng., P.O. Box 44130, Lafayette, LA 70504 USA

In the past, the selection of tubulars (OCTG) has been routine and basic, with most users selecting carbon steel pipe as their choice. Today, a paradigm shift from the use of traditional carbon steel to 13% Cr is taking place. The origin of the use of 13% Cr steel pipe is the existence of oil and gas fields producing appreciable amounts of CO2, most often in the complete absence of H2S. In particular, CO2 corrosion leads to the performance of tubing by localized attack leading to a very short lifetimes before breakthrough. The object of this paper is to furnish the engineer with a simple means of estimating the surface roughness K, and relative roughness, K/D, for chrome 13 pipes. In 1944, Moody prepared a plot of relative roughness K/D versus pipe diameter D, for a number of materials. Moody's relative roughness correlation was based on experiments on the pipes artificially roughened with sand grains. Moody did not provide the relative roughness plot for chrome 13 pipes, nor did he perform a regression analysis of the data to provide functional forms of the equations relating relative roughness, K/D, as a function of pipe diameter, D. Currently, chrome 13 pipes are being utilized world wide. Consequently, absolute surface roughness and relative roughness values of chrome 13 pipes are needed by practicing engineers to properly model the hydrodynamics in pipes. It is important to emphasize that dimensional analysis suggests that the effect of surface roughness is not due to its absolute dimensions, but rather to its dimensions relative to the inside diameter of the pipe, K/D. Thus, it has been the thrust of this research to develop a new set of relative roughness charts along with its corresponding mathematical equations for chrome 13 pipes.

3:15 PM
An Atomistic Study of Solid-Liquid Interfaces: *Michael I. Baskes*[1]; Marius Stan[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Techn. Div., P.O. Box 1663, MS-G755, Los Alamos, NM 87545 USA

A semi-empirical Lennard-Jones/Embedded Atom Method model is used to capture real materials behavior through the introduction of many-body forces. Using MD calculations, the model is used to study the dependence of the solid/liquid interface velocity on temperature and composition. Slowing of interface velocity by solutes is demonstrated. The results of MD calculations are used to get free energies of all phases. Solid-liquid equilibrium calculations illustrate the consequences of the differences in energy and size between the components on the phase diagram.

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Stability of X2AlTi (X: Fe Co Ni and Cu) Heusler Phase in B2-Type Aluminides: Kazuhiro Ishikawa[2]; *Ryosuke Kainuma*[1]; Ikuo Ohnuma[1]; Kiyoshi Aoki[2]; Kiyohito Ishida[1]; [1]Tohoku University, Dept. Mat. Sci., Aobayama 02, Aoba-ku, Sendai, Miyagi 980-8579 Japan; [2]Kitami Institute of Technology, Dept. Mat. Sci., Koencho 165, Kitami, Hokkaido 090-8507 Japan

It has been reported that the precipitation of Ni_2AlTi (H: $L2_1$) Heusler phase in the NiAl (β: B2) phase increases the creep strength at high temperatures in the Ni-Al-Ti system. The information about the phase equilibrium between the H and β phases is important for the microstructure control of the two-phase alloys. Very recently, the present authors reported the phase equilibria and ordering reaction between the H and β phases in the (Ni, Fe)-Al-Ti and (Ni, Co)-Al-Ti quaternary systems. In the present study, the phase equilibria and ordering reaction between those phases in the (Ni, Cu)-Al-Ti and (Co, Fe)-Al-Ti quaternary systems are determined and phase stability of X_2AlTi (X: Fe, Co, Ni and Cu) Heusler phase in the β phase is discussed.

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Thermodynamics of Systems with Multiple Simultaneous Internal Processes and Its Application to the CVD Diamond Growth: *Zi-Kui Liu*[1]; Ji-Tao Wang[2]; [1]The Pennsylvania State University, Dept. of Mats. Sci. & Eng., 209 Steidle Bldg., University Park, PA 16802 USA; [2]Fudan University, Dept. of Elect. Eng., 220 Handan Rd., 200433 Shanghai, China

Phase equilibrium in a system is usually defined when all internal processes in the system are ceased to take place and a characteristic state function of the system, typically a free energy, is at its minimum. However, there are systems that several internal processes occur simultaneously in the system and the system free energy is kept at minimum and remains unchanged with a constrained condition. In the present paper, fundamental thermodynamics of this type of systems is discussed. It is suggested that a combined driving force can be used to determine the phase equilibrium of this type of systems. This combined driving force is a weighted average of the driving forces of individual internal processes and the weights depend on the relative rates of the individual internal processes. Consequently, one may argue that these internal processes are coupled. As an example, the phase equilibrium in the C-H binary system is discussed in connection with the activated CVD diamond growth in which the relative stability of graphite and diamond is altered through the internal process coupling.

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Thermodynamic Assessment of the Nb-Ti System: *Yuelan Zhang*[1]; Huashan Liu[1]; Zhanpeng Jin[1]; [1]Central South University of Technology, Dept. of Mat. Sci. & Eng., Changsha, Hunan 410083 China

The thermodynamic properties of the Nb-Ti system have been evaluated by using regular model to describe the Gibbs energies of various phases inclusive equilibrium as well as metastable equilibrium phases. A set of thermodynamic parameters more consistent with most of the selected experimental data than previous assessments has been obtained. Stable and metastable phase equilibria, T_O loci, metastable chemical spinodal curve of beta phase and thermodynamic properties are calculated with optimized parameters.

Cyanide: Social, Industrial, and Economic Aspects: Cyanide Management III and Fundamentals

Sponsored by: Extraction & Processing Division, Waste Treatment & Minimization Committee, Precious Metals Committee, International Precious Metals Institute, Society of Mining, Metallurgy and Exploration, Inc., Northwest Mining Association

Program Organizers: Courtney Young, Montana Tech, Metallurgical and Materials Engineering, Butte, MT 59701 USA; Corby Anderson, Montana Tech., CAMP and Metallurgical and Materials Engineering, Butte, MT 59701 USA; Larry Twidwell, Montana Tech, Metallurgical and Materials Engineering, Butte, MT 59701 USA

Wednesday PM — Room: 225
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Larry Twidwell, Montana Tech, Metall. and Mats. Eng., Butte, MT 59701-8997 USA; Kwadwo Osseo-Asare, Penn State University, Dept. of Mats. Sci. and Eng., University Park, PA USA

2:00 PM Invited

Cyanide Management at Telfer Gold Mine Using SART: Janene Barter[1]; Robert Dunne[2]; [1]GRD Minproc, Ltd., Level 8 140 St George's Terrace, Perth, Western Australia 6000, Australia; [2]Newcrest Mining, Ltd., P.O. Box 6380, East Perth, Western Australia 6892, Australia

Telfer Gold Mine treats both copper gold oxide and sulfide ores in a remote location in the Great Sandy Desert, Western Australia. The oxide ore is treated in a conventional CIL circuit by blending the low and high copper grade ores to minimise cyanide consumption and allow smooth operation. As the depth of the pit has increased the copper head grade has also increased, to the point it was becoming uneconomical to treat. In addition, it was desirable to reduce the amount of cyanide reporting to the tails dam both from an environ mental and economical viewpoint. To overcome this problem Newcrest Mining Ltd engaged GRD Minproc Ltd to assist with the evaluation of processes to reduce the amount of cyanide entering the tails dam with consideration given to copper recovery. From this investigation the Sulfidisation, Acidification, Recycle and Thickening (SART) method was selected. A detailed testwork program for SART was then undertaken with the objective to design and construct a SART plant at Telfer. This paper outlines how the SART process was chosen followed by details of the testwork that lead to the design of the SART plant. It also touches on some of the important issues for designing this type of process.

2:25 PM Invited

Cyanide Recovery/Destruction Using Air Sparged Hydrocyclone Technology: *Jose R. Parga Torres*[1]; Jan D. Miller[2]; [1]Institute Technology of Saltillo, Dept. of Metall. and Mats. Sci., V. Carranza 2400, C.P. 25000, Saltillo, Coahuila, Mexico; [2]University of Utah, Dept. of Metall. Eng., 412 William C. Browning, Salt Lake City, UT 84112 USA

In mining operations, cyanidation is the predominant method by which gold and silver are recovered from their ores and it is recognized that cyanide consumption can be a major factor which contributes to operating cost for cyanidation. Also after extraction and recovery of precious metals substantial amounts of cyanide are delivered to tailings ponds which creates environmental problems due to the toxicity of cyanides. In this regard, the air-sparged hydrocyclone (ASH) has been used as a reactor for the treatment of cyanide solutions in two ways: first for cyanide recovery by acidulation using the Mexican modification of the Mills-Crowe process and second for cyanide destruction by oxidation with the use of chlorine dioxide (ClO2). In both cases excellent performance can be achieved using the high capacity ASH technology.

2:50 PM Invited

Innovative Cyanide Solution Treatment by Thermal Plasma: *Gervais Soucy*[1]; Luc Fortin[2]; Vijaya Kasireddy[2]; Jean-Luc Bernier[3]; Frank M. Kimmerle[2]; [1]University of Sherbrooke, Dept of Chem. Eng., Fac. of Eng., Sherbrooke, Quebec J1K 2R1 Canada; [2]Alcan International, Ltd., Arvida Res. and Dev. Centre, P.O. Box 1250, Jonquiere, Quebec G7S 4K8 Canada; [3]Alcan Aluminium, Ltd., Usine Grande Baie, P.O. Box 900, Ville de la Baie, Quebec G7B 4G9 Canada

During mineral and industrial processing, contaminated wastewater can be generated such as cyanide (free species or in complex form).This paper presents the treatment of cyanide solution being derived from the aluminum industry by direct contact with thermal plasma in a novel reactor. The energy is provided by a plasma submerged in the solution which allows direct contact between the plasma and the solution. The scope of this study was to determine the feasibility of treating free and complex cyanides in solution through bench scale operations. An innovative reactor was designed and fabricated to provide stable operating conditions. Many parameters were studied, such as the NaOH concentration (30 and 60 g/L), initial SPL (Spent PotLining) leachate concentration (150 and 350 mg/L), plasma power (10 and 19 kW) and relative reactor gauge pressure (0 and 1.34 MPa). These experiments allowed us to evaluate the kinetics of complex cyanide decomposition under thermal plasma conditions. At atmospheric pressure (about 100ºC), the rate of cyanide decomposition was 12 times greater than that of thermal hydrolysis occurring in a plug flow reactor at the same temperature. These improvements are attributed to the presence of both steep thermal gradients and reaction photocatalysis by the plasma UV radiation.

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Oxidation of Cyanide in an Electrochemical Porous-Electrode Flow-Reactor: Peter C. Sanford[2]; *Gerard P. Martins*[1]; [1]Colorado School of Mines, Dept. of Metallur. and Mats. Eng., 920 15th St., Golden, CO 80401 USA; [2]Science Applications International Corporation, 405 Urban St., Suite 400, Lakewood, CO 80228 USA

Cyanides are present as a dilute constituent of streams from a variety of metallurgical and mineral-processing operations. In addition, a cyanide concentration in excess of approximately 100 ppb is toxic to vertebrates, these operations require a waste-treatment process to reduce the already dilute cyanide species by several orders of magnitude. Furthermore, the waste streams may have flowrates in excess of a million gallons per day. The paper to be presented reports on research conducted with a laboratory-scale electrochemical porous (graphite felt) electrode (anode) flow-reactor, in conjunction with a computer-aided simulation of a mathematical model describing the intrinsic rate-phenomena associate with the electrode processes. The laboratory-scale cell, of rectilinear geometry was found to be capable of reducing a feed-stream with cyanide (KCN-KOH) concentration of 10ppm to less than 100ppb, at current efficiencies in the neighborhood of 50%. The computer simulation of the cell performance based on the mathematical model (and constraints imposed for tractability) was found to provide only for qualitative agreement.

3:50 PM Invited

Electrochemical Destruction of Free Cyanide on a Cobalt Oxide Doped Electrode: *Fockedey Etienne*[1]; Stavart Arnaud[2]; Van Lierde A.[1]; [1]Université Catholique de Louvain, Unité de Physico Chimie Et D'Ingénierie Des Matérieaux, Batiment Réaumur, 2°Etage, Place Sainte Barbe, Louvain la Neuve 1348 Belgium; [2]Meura Technologies, Voie Minckelers, 1, Louvain la Neuve 1348 Belgium

We studied the destruction of free cyanure on a Co3O4 doped carbon felt. The experiments were done with an electrochemical filtre press cell on dilute alkaline solutions containing 250 mg/l NaCN. No chloride were added to the solutions. The cobalt oxyde protect the felt against corrosion and has a catalytic effect on the degradation of the cyanide. This kind of electrode allows to work with current density as high as 1200 A/m² without any seenable degradation of the felt. The most interesting results were obtained with a current density of 400 A/m². In this case, we decrease the cyanure concentration to 5 mg/l with a current efficency greater than 70% and a specific energic consumption of about 10 kWh/ kg CN.

4:10 PM Invited

Cobalt/Nickel Separation by Cyanide Complexation: Scott A. Shuey[1]; Larry G. Twidwell[2]; [1]MineDepot.com, 535 W Cornelia, #301, Chicago, IL 60657 USA; [2]Montana Tech of the University of Montana, Met. Eng. Dept., 1300 W. Park St., Butte, MT 59701 USA

The present industrial practice used to separate aqueous cobalt from nickel is multistage solvent extraction processing. This paper describes a different but simpler treatment technology. The proposed technology has been applied to cobalt/nickel hydroxide intermediate solids produced from the treatment of Electrochemical Machining (ECM) waste sludge. The process consists of dissolving the cobalt/nickel hydroxides in cyanide solutions. The cobalt and nickel form aqueous solution cyanide complexes. Nickel can be selectively precipitated from the cobalt as a nickellic hydroxide solid. Cobalt can be recovered from the leach solution by precipitation of a cobaltous/cobaltic cyanide double salt. The double salt product can then be subsequently treated to produce an appropriate cobalt product. The process chemistry, proposed flowsheets, and preliminary economics will be presented and discussed.

4:35 PM Invited

Anodic Behavior of Gold-Silver Alloys in Aqueous Cyanide Solutions: Tao Xue[1]; *Kwadwo Osseo-Asare*[1]; [1]Dynatec Canada, Metall. Techn. Div., 8301-113 St., Fort Saskatchewan, Alberta Canada; [1]Penn State University, Dept. of Mats. Sci. and Eng., Steidle Bldg., University Park, PA 16802 USA

Gold frequently occurs in nature in the form of a gold-silver alloy. Thus, investigation of the electrochemical behavior of gold-silver alloys in cyanide solution has practical significance. In this paper, the potentiodynamic method is used to determine the anodic polarization curves of gold-silver alloys in cyanide solutions. To facilitate the interpretation of the results, complementary experiments conducted in NaOH solution in the absence of cyanide are also reported. In combination with results on the solution equilibria of the Au- and Ag-CN-H2O systems and the polarization behavior of pure gold and pure silver in cyanide solutions, the difference between the electrochemical behavior of the pure metals and that of the alloys is clarified. On the basis of electrochemical kinetics concepts, the dissolution mechanism of gold-silver alloys, the effect of the presence of silver on the cyanidation of gold, as well as the factors which control the anodic reaction rate are clarified.

Defect Properties and Mechanical Behavior of HCP Metals and Alloys: Generalized Deformation and Texture

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Chemistry & Physics of Materials Committee, Jt. Nuclear Materials Committee, Titanium Committee

Program Organizers: Man H. Yoo, Oak Ridge National Laboratory, Metals & Ceramics Division, Oak Ridge, TN 37831-6115 USA; James R. Morris, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Carlos N. Tome, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

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February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Thomas R. Bieler, Michigan State University, Mats. Sci. and Mech., East Lansing, MI 48824-1226 USA; Anthony D. Rollett, Carnegie Mellon University, Mats. Sci. and Eng., Pittsburgh, PA 15213-3177 USA

2:00 PM Invited

Texture, Deformation Mechanisms and Forming Properties of Hexagonal Alloys: *Marie Jeanne Philippe*[1]; [1]Université de Metz,

LETAM, Ile du saulcy BP 80794, Metz, Cedex 1 57012 France

The mechanical properties of hexagonal alloys are strongly anisotropic because of the associated sharp texture and the anisotropy of the HCP monocrystal. The present paper reviews the following aspects associated with forming of Zinc, Titanium and Zirconium Alloys: (1) initial texture and texture evolution during deformation; (2) active deformation mechanisms; (3) mechanical properties associated with (1) and (2), and (4) modeling and prediction of mechanical properties. Deformation can be accommodated by both glide and twinning, and their relative contribution depends on the alloying elements, the grain size, the grain orientation, and temperature and strain rate conditions. Yield stress and ductility can be directly related to the deformation mechanisms activated. The present work demonstrates the qualitative correlation between items (1) to (3) above, and the forming properties for all the alloys studied. A correlation is made between deformation texture and the composition of the alloys. We show that it is possible to model the texture evolution and to predict the mechanical properties of the alloys studied taking into account the initial texture and the active deformation mechanisms. Predictions and experimental results are compared and the most important deformation parameters are identified and discussed. As a conclusion, we identify the next steps required to make progress in this type of work.

2:30 PM

Identification of Material Parameters in Yield Functions for Textured Titanium Sheets: *Chi-Sing Man*[1]; Ying Zhang[2]; Yu Xiang[3]; Mojia Huang[1]; [1]University of Kentucky, Dept. of Math., 715 Patterson Office Tower, Lexington, KY 40506-0027 USA; [2]Xiamen University, Dept. of Mats. Sci., Xiamen, Fujian 361005 China; [3]University of Kentucky, Dept. of Chem. and Mats. Eng., 177 Anderson Hall, Lexington, KY 40506-0046 USA

Man has recently proposed a hierarchy of yield functions which, in increasing sophistication, would take account of the effects of crystallographic texture on the plastic flow behavior of weakly-textured orthorhombic sheets of hexagonal metals. In this paper we examine: (i) whether the two yield functions lowest in the hierarchy would be adequate for describing the angular dependence of the yield stress and of the r-value for commercially pure titanium sheet in uniaxial tension tests; (ii) whether the experimental data on the angular dependence of the uniaxial yield stress and of the r-value would suffice for the identification of material parameters in the two yield functions.

2:50 PM

Role of Twinning in the Constitutive Response of Zirconium: *Carlos N. Tomé*[1]; George C. Kaschner[1]; Thomas A. Mason[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Techn., MST-8, Mail Stop G755, Los Alamos, NM 87545 USA

Polycrystal models have been traditionally used for understanding and describing texture evolution during plastic forming. More recently, they have been used for understanding the hardening of the aggregate, through the correlation between the macroscopic stress and the internal hardening mechanisms at the grain level. In hcp materials, twinning is an important deformation mode and its contribution to texture and hardening has to be accounted for. While the former has been incorporated in polycrystal models, very little has been done to account for the hardening contribution. In a recent study[1] we use tension and compression results of rolled Zr tested along different directions to adjust a constitutive law. In doing so we account for the twinning contribution to texture using a Predominant Twin Reorientation scheme, and for its contribution to hardening by treating the twinning activity in a similar way as we treat the slip activity. Although the resulting constitutive description is successful in describing the material response at moderate strains (~20%), two conclusions are extracted from this study concerning the hardening contribution from twinning: the latter is more relevant to the constitutive law than the associated texture contribution, and an approach different than the one for slip is required to describe it. In the present work we extend a model originally proposed by Karaman et al.[2] for Hadfield steel, and reformulate the constitutive response of Zr using a twinning model that accounts for directional barriers to dislocation motion posed by the twin lamellae in the grains, and also for the evolution of twin fraction with deformation in the grains. In addition to the tensile and compressive tests, we use Orientation Image Microscopy (OIM) of bent Zr beams to elucidate the evolution of twinning with deformation and as a way of checking the consistency of the model. [1]C.N. Tomé, P.J. Maudlin, R.A. Lebensohn and G.C. Kaschner, Acta materialia, submitted. [2] I. Karaman, H. Sehitoglu, A.J. Beaudoin, Y.I. Chumliakov, H.J. Maier and C.N. Tomé, Acta materialia 48 (2000) 2031.

3:10 PM

Texture Effects on Dynamic Shear Response in Ti-6Al-4V plates: *Bimal Kad*[1]; Scott Schoenfeld[2]; [1]University of California-San Diego, Struct. Eng., 409 University Ctr., La Jolla, CA 92093-0085 USA; [2]US Army Research Laboratory, Impact Phys., Terminal Effects Div, Bldg. 4600, AMSRL-WM-TD, Aberdeen Proving Ground, MD 21005-5069 USA

Due to the asymmetric and anisotropic nature of crystallographic slip and twinning in HCP materials. the bulk mechanical properties are strongly affected by the orientation distribution of the crystals in the polycrystal aggregate. In this presentation we examine the in-plane shear response of Ti-6Al-4V alloys that are nominally textured via routine deformation and thermal processing schedules. For rolled plates, a 2-dimensional constitutive model for slip and twinning (treated here as pseudo-slip) is derived for the HCP single crystal, and embedded into a finite element model representing a spatial distribution of single crystals. The polycrystal mechanical response is examined with respect to macroscopic shear loading as may take place during a dynamic punch through process.

3:30 PM Break

3:50 PM Invited

Creep Mechanisms and Anisotropic Creep in Zr Alloys: *K. L. Murty*[1]; [1]North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA

Zirconium alloys are commonly used for cladding radioactive UO_2 in water reactors and possess hcp structure at the reactor operating temperatures. Zr alloyed mainly with Sn and Fe (referred to as Zircaloys) is widely used while recently Nb-additions are considered for long-term application with improved corrosion resistance. Creep characteristics of these thin-walled tubing materials are evaluated for predicting the dimensional changes of these structures in-service. Zircaloy-4 that finds application in the pressurized water reactors exhibit deformation behavior characteristic of Class-M (also known as Class-II) alloys with climb of edge dislocations as the dominant mechanism while Nb additions are shown to lead to alloy class (Class-A or Class-I) behavior with the stress exponent (n) of around 3 (identified as the viscous glide due to solute locking). At low stresses, both materials exhibit Newtonian viscous creep behavior with n=1 identified to be Coble creep. Crystallographic textures developed in these ma terials during the thermomechanical processing result in highly anisotropic deformation, and the effects of stress level and alloying on creep under equibiaxial loading will be described. The significance of transitions in creep mechanisms (as lower stresses are encountered) on the predictability of dimensional changes in-service will be highlighted.

4:20 PM

Diffraction Studies of Twinning in Pure Zirconium: *Partha Rangaswamy*[1]; Donald W. Brown[1]; George C. Kaschner[2]; John F. Bingert[3]; Luc L. Daemen[4]; [1]Los Alamos National Laboratory, Mats. Sci. Techn., MST-8, MS H805, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Mats. Sci. Techn., MST-8, M/S G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Mats. Sci. Techn., MST-6, M/S G770, Los Alamos, NM 87545 USA; [4]Los Alamos National Laboratory, LANCE-12, Lujan Center, M/S H805, Los Alamos, NM 87545 USA

An experimental study using x-ray and neutron diffraction was performed to quantify the twinning volume fraction in pure zirconium deformed under compression. Strongly textured clock-rolled pure zirconium was deformed to a maximum strain of 25% with the loading direction nearly parallel to the c-axis of the hexagonal close packed unit cell. The deformation was performed at applied strain rates between 0.001 and 3500/s, and test temperatures between 77

and 298K. Using both x-rays and neutrons, diffraction peak intensities were recorded as a function of strain in the various samples. The objective of this study was to demonstrate the effectiveness of using diffraction as a method to correlate the diffraction intensity changes with the plastic contribution of the macroscopic strains. The overall goal is to quantify the volume fraction of twinning and use it as a calibration for verifying volume fractions determined through orientation imaging microscopy.

4:40 PM

The Role of the HCP Phase on the Tensile Mechanical Properties of Two-Phase Co-27Cr-5Mo-0.05C Alloy: *Armando Salinas-Rodriguez*[1]; Antonio Mani-Medrano[1]; [1]Centro de Investigacion y de Estudios Avanzados del IPN , Carr. Saltillo-Monterrey km 13, P.O. Box 663, Saltillo, Coahuila 25000 Mexico

Isothermal aging of solution treated Co-27Cr-5Mo-0.05C alloy produces two-phase, FCC-HCP, microstructures. The amount of the HCP phase produced by aging at a given temperature exhibits a sigmoidal dependence on time. Alloys with varying amounts of HCP phase were used to investigate the effects of the phase distribution on their room temperature tensile properties and hardness. The results showed that the yield strength and hardness increase linearly with the increase in the amount of HCP phase. In contrast, the ductility remains constant for 15<%HCP<55. At larger or smaller amounts of HCP phase the ductility decreases linearly. It was also found that the amount of HCP phase, as measured by X-ray diffraction, increases significantly as a result of plastic deformation within the region of nearly constant ductility. Finally, the tensile fracture strength was found to be independent on the amount of HCP present at the start of the deformation. It is proposed that the tensile ductility of this material depe nds on the ability of the metastable FCC phase to transform to HCP dynamically during plastic straining. The region of slow decrease of the ductility with increasing %HCP was attributed to the dynamic softening associated with the strain induced transformation.

5:00 PM

The Basal Slip in Alpha-Zirconium: Experimental Evidences and the Role in Texture Formation: *Margarita Isaenkova*[1]; Yuriy Perlovich[1]; [1]Moscow State Engineering Physics Institute (Technical University), Met. Sci. Dept., Kashirskoe Shosse 31, Moscow 115409 Russia

Up to now the real participation of the basal slip in the plastic deformation of alpha-Zr is significantly underestimated. Several X-ray diffractometric methods were elaborated to follow reorientation of alpha-Zr grains by the plastic deformation of commercial Zr-based alloys. For grains with different initial orientations the trajectories of basal axes were constructed for the cases of gradual rolling of an originally textureless sample and transverse rolling of a sample with the previously developed texture. The obtained trajectories by intermediate grain orientations correspond to the preferential action of the basal slip, as it follows from the known models, connecting modes of texture formation with concrete mechanisms of plastic deformation. The basal slip results in formation of the intermediate quasi-stable rolling texture with basal axes, declined from ND to RD by 15-25°. Its stability up to deformation degrees of 50-60% is conditioned by the mutual equilibrium of opposite reorientation effects, accompanying action of basal and pyramidal slip systems. Only by the further increase of the deformation degree, this intermediate texture transforms into the final stable rolling texture with basal axes, declined from ND to TD by 30-50°.

General Abstract Sessions: Extraction and Processing

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Program Organizers: Thomas P. Battle, E. I. DuPont de Nemours & Company, Inc., White Pigments and Mineral Products, Edgemoor, DE 19809 USA; TMS, Warrendale, PA 15086 USA

Wednesday PM — Room: 212
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Mark E. Schlesinger, University of Missouri-Rolla, Dept. Metallur. Eng., Rolla, MO 65409-0001; Ibrahim Gaballah, INPL-CNRS, Vandoeuvre, France

2:00 PM

Chlorination: An Emerging Technology: *I. Gaballah*[1]; N. Kanari[1]; M. Djona[1]; E. Allain[2]; [1]Mineral Processing and Environmental Engineering Team, Rue du Doyen Marcel Roubault, B.P. 40, Vandoeuvre, Cedex 54501 France; [2]University of Missouri-Rolla, Sch. of Mines and Metall., Ctr. for Pyrometall., 215 Fulton Hall, Rolla, MO 65401 USA

Sodium hydroxide and chlorine are generated by the electrolysis of NaCl. Periodical imbalance of the consumption of these two products poses problems of storage for the operators. Chlorine surplus could be used for the extraction of valuable metals and for upgrading of raw materials. Extraction of tantalum and niobium from tantalo-columbite concentrates and tin slag, upgrading of chromite concentrate, recovery of valuable metals from spent catalysts by using chlorine technique are summarized. Thermodynamic and kinetics aspects of several chlorination reactions are outlined.

2:25 PM

Technology Selection for La Caridad Precious Metals Plant: *Irshad A. Rana*[1]; [1]Fluor Daniel, Mining, 43686 Skye Rd., Fremont, CA 94539 USA

Available and proven technologies were reviewed for the La caridad Precious Metals Plant to produce approximately 450 mt/year silver, 3 mt/year gold and assorted other metals from the anode slimes originating from the La Caridad Copper Refinery and some purchased materials. For the conventional process the technologies considered were autoclave leaching, roasting, Moebius parting plant, chemical precipitation of fine gold from slolution and oxygen assisted smelting and refining. The Phelps Dodge process was considered which consists of slimes leaching, sulfatization roast, silver leaching/electrowinning, gold leaching and solvent extraction/ precipitaion. Also evaluated was the complete hydrometallurgical wet chlorination process in which a series of leaching and precipitation steps are used to produce refined precious metals. A flowsheet was finally selected, plant built during 1999 and commissioned in year 2000.

2:50 PM

Production of Pure Metals and Semiconductive Materials: *A. V. Tarasov*[1]; Yu O. Mamayev[1]; [1]State Research Center of Russian Federation, State Rsch. Instit. on Non-Ferrous Mets., 13 Acad. Korolyov St., Moscow 129515 Russia

Issues relating to production of bismuth, antimony and tellurium of high purity for application relating to synthesis of low-temperature thermoelectric alloys based on bismuth and antimony chalcogenides have been discussed. Experimental investigations of physical and chemical aspects of the behavior of individual impurities of their combinations have been carried out using different crystallization methods. Process parameters for production of high-purity materials have been optimized. The necessity for integrated use of distillation and zone re-crystallization techniques for production of high-purity materials has been confirmed.

3:15 PM Break

3:30 PM

Hydrometallurgical Technologies of a Complex Silver-Gold Concentrate and its Calcine by Chlorine and Thiosulfate: *Shengming Xu*[1]; Jingming Xu[1]; Ru'an Chi[1]; Guocai Zhu[1]; [1]Tsinghua University, Inst. of Nuclear Energy Techn., Beijing, 102201 China

Leaching of a complex silver-gold concentrate and its calcine was investigated using chlorine and thiosulfate for extraction of gold and silver. The results of elementary experiments showed that the concentrate needed to be pre-treated prior to leaching. In order to eliminate the carbonaceous materials and decompose the sulfides, a series of roasting tests was firstly carried out; the roasting conditions of the concentrate were also chosen. The results showed that the additive had a favorable effect on extractive yield of silver in the temperature range of 400º-650º. Silver minerals in the concentrate will be dissociated, then oxidized into sulfate or chloride with a different additive. Following chlorine leaching of gold, the calcine was leached out by thiosulfate again. The extractive yields of gold and silver would excess 95% and 97%, respectively. This new route seems to be more environmental and economical than the traditional cyanide process.Keywords: Silver concentrate; Roasting; Leaching; Chlorine; Thiosulfate

3:55 PM

Thiourea Leaching of Gold and Silver from A Carbonaceous Silver Concentrate: *Shengming Xu*[1]; [1]Tsinghua University, Insti. of Nuclear Eng. Techn., Beijing 102201 China

An innovative process for thiourea leaching of gold and silver from a carbonaceous flotation concentrate assaying 17.4 g/t Au, 4301 g/t Ag and 6.71 % C, and containing different sulfide minerals, was investigated. According to the results of chemical phase analysis of gold and silver, the concentrate can be classified as refractory ore because silver is difficult to extract by traditional cyaniding or thiourea process. A thiourea carbon-in-pulp process, however, is suitable for the extraction of gold prior to silver preferentially. The influence of leaching temperature, leaching time, thiourea concentration, pH value and activated carbon added amount on extractive yield of gold was firstly investigated, and extractive yield of gold can reach 92% in this stage. In order to recover the residual silver, the leached gold residue further ground in a vibration miller for 10 min. was subsequently oxidized by a dilute ferric chloride solution, and most of silver minerals were dissolved into the solution. Thus, total extractive yield of silver will increase from 60% to 95%. Keywords: carbonaceous silver concentrate; thiourea carbon-in-pulp process; ferric chloride leaching

General Abstract Sessions: Composites/New Products and Processes

Sponsored by: TMS

Program Organizers: Thomas P. Battle, E. I. DuPont de Nemours & Company, Inc., White Pigments and Mineral Products, Edgemoor, DE 19809 USA; TMS, Warrendale, PA 15086 USA

Wednesday PM Room: 229
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: James C. Foley, Ames Laboratory, Metall. and Ceram. Prog., Ames, IA 50011 USA; Funsho Ojebuoboh, ASARCO, Denver, CO 80216 USA

2:00 PM

Metal/Polymer Interface and Solder Joint Reliability of a Wafer Level CSP: *Hun Han*[1]; Jin Yu[1]; [1]KAIST, Mat. Sci. and Eng., 373-1 Kusong-dong, Yusong-gu, Taejon 305-701 Korea

Wafer level chip scale package (WLCSP) has the highest potential compared with flip chip package (FCP), because they provide the benefits of real chip size package with low cost and high reliability. We use low modulus polymers (stress buffer layer; SBL) which were designed to relax the thermal strain generated at the solder joint. However, reliability of metal/polymer interface and solder joint are still critical problems to the WLCSP. We studied the reliabilities of metal/SBL interfaface and solder joint according to variation of the polymer surface treatments and the metal structure. To measurement of adhesion property on metal/SBL, peel strength was evaluated. In addition, the effects of under bump metallurgy (UBM) on the reliability solder joint at the WLCSP will be discussed.

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HVOF Application of Nickel and Nickel Alloy Jackets to Tungsten Heavy Alloy Penetrators: *John Vincent Kelley*[1]; [1]Army Research Laboratory, Weapons Mats, Div,, AMSRL-WM-MC, Bldg. 4600, Aberdeen Proving Ground, MD 21005 USA

There has been an increased desire to replace depleted uranium (DU) anti-armor penetrators with tungsten heavy alloy (WHA). Unfortunately, WHA does not perform ballistically nearly as well as DU. One effort to improve ballistic performance of WHA examines high velocity oxy-fuel (HVOF) coatings as a jacket material. The differences in the coefficients of thermal expansion (CTE) make it difficult to achieve adequate adhesion of most coatings to tungsten. Further compounding the problem are residual stresses inherent in thermally sprayed coatings. Early attempts to apply 0.010-0.020 inches of Ni to tungsten substrates yielded immediate disbonding and/or cracking of the coatings. This work is an investigation of modifications to parameters and coating thicknesses to minimize heat transfer to achieve better adhesion. The use of materials with a CTE nearer to that of tungsten as a bond coat was also examined as a method for increasing the overall adhesion of the coating system.

2:50 PM

Fatigue Behavior of Ceramic Matrix Composites with Nondestructive Evaluation (NDE) Techniques: *Jeongguk Kim*[1]; Peter K. Liaw[1]; Hsin Wang[2]; You-Tae Lee[3]; [1]University of Tennessee, Mats. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Mets. and Ceram. Div., Oak Ridge, TN 37831 USA; [3]Kyongdo College, Dept. of Auto., Kyungbook 757-800 Korea

High-cycle fatigue behavior of continuous fiber reinforced ceramic matrix composites (CFCCs) was investigated with the aid of nondestructive evaluation (NDE) techniques. The NDE methods used for this investigation include ultrasonic testing (UT), infrared (IR) thermography, and acoustic emission (AE) techniques. Prior to fatigue testing, UT and IR thermography were used to characterize the initial defect distribution of CFCC samples, i.e., developing ultrasonic C-scans and thermal diffusivity maps, respectively. A qualitative correlation between the C-scan and thermal diffusivity map has also been obtained. During fatigue testing, AE sensors and an IR camera were used for in-situ monitoring of progressive damages of CFCC samples. A stress versus cycles to failure (S-N) curve has been provided to predict the lifetime of CFCC samples as functions of initial defects and progressive damages. UT and IR thermography were conducted on fractured samples after testing to compare progressive damages with the init ial defects. Microstructural characterization using scanning electron microscopy (SEM) was performed to investigate fracture mechanisms of CFCC samples. In this study, NDE techniques were used to facilitate a better understanding of fracture mechanisms of CFCCs during high-cycle fatigue testing.

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Oxide Dispersion Hardened Platinum Materials for High Temperature Applications: *Bernd Fischer*[1]; Dietmar Freund[2]; Rainer Volkl[2]; David F. Lupton[2]; [1]FH Jena-University of Applied Sciences, Dept. of Mats. Tech., Carl-Zeiss-Promenade 2, Jena D-07745 Germany; [2]FH Jena-University of Applied Science, Dept. of Mats. Sci., Carl-Zeiss-Promenade 2, Jena D-07745 Germany

In spite of their high prices platinum materials are well suited to being used as high temperature materials for varied technical applications. This paper reports on the fabrication, structure, properties and use of a new class of oxide dispersion hardened platinum materials (Pt DPH materials) with improved essential properties. The production follows a new route. By the addition of oxide-forming

elements (for example zirconium and yttrium) during the melting process it is possible to precipitate dispersoids in the course of the working process by means of internal oxidation. The materials Pt DPH, Pt-10%Rh DPH and Pt-5%Au DPH are available. The new oxide dispersion hardened platinum materials are excellent to process (forming, welding). Because of the dispersoids they have excellent grain stability in long-term use at the highest temperatures. Mechanical high temperature properties (stress-rupture strength, creep data) are given in the unwelded and welded state for the temperature range 1200°-1600°C and for the material Pt-10%Rh DPH up to 1700°C. In accordance with the practical use of the materials, the results of stress-rupture tests with periodical change of temperature and the effects of notches with special geometry are discussed. Under all test conditions the new oxide dispersion hardened platinum materials have not only an increased stress-rupture strength but also a good ductility even at the highest temperatures. These facts apply in particular to the welded state. Finally the paper reports on first tests of the new platinum materials for components in the glass industry. The excellent chemical stability of the materials in aggressive glass melts has been investigated. Compared with the previously known platinum materials, the Pt DPH materials show improved properties which lead to a considerable increase in the time to failure for components in the glass industry.

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New Process for the Synthesis of Potassium Ferrate ($K_2Fe^{VI}O_4$): *N. Kanari*[1]; I. Gaballah[1]; C. Mathieu[2]; N. Neveux[2]; O. Evrard[2]; [1]Mineral Processing and Environmental Engineering Team, CNRS UMR 7569, ENSG INPL, BP 40, Vandoeuvre, Cedex 54501 France; [2]Universite Henri Poincare Nancy I, Lab. de Chimie du Solide Minerale, BP 239, CNRS UMR 7555, Vandoeuvre, Cedex 54506 France

Potassium ferrate is a superoxidant material containing iron in hexavalent state (Fe^{VI}). The conventional synthesis's methods are based on wet processing leading to ferrate decomposition and consequently low synthesis efficiency. A new process for the dry synthesis of potassium ferrate is developed and already patented. It consists to the reaction of iron sulfate with an oxidant in a postassium hydroxide medium. Synthesis is achieved in a rotary reactor at room temperature and the overall reaction was exothermic. The potassium ferrate (Fe^{VI}) synthesis' efficiency is about 60%. Kinetics aspects of the potassium ferrate synthesis are detailed.

4:15 PM

The Challenge for Lead-free Brass in Casting Alloys: *Funsho Ojebuoboh*[1]; [1]ASARCO, Inc, 495 E. 51st Ave., Denver, CO 80216 USA

Copper-based alloys, i.e., brasses and bronzes, are the preferred and traditionally alloys for sand-cast plumbing components, and more recently, permanent mold castings for finer water fixtures. As it is for other engineering alloys, lead is a particularly important additive to copper alloys where it imparts the key attributes of machinability and pressure tightness. To truly gain the benefits of these attributes, manufacturers use as much as 8% lead in sand castings. However, after the US Safe Drinking Water Act of 1986, the industry (ingot manufacturers through end-users) has endeavored to reduce the level of lead in the value chain. This paper examines EnviroBrass, a family of lead-free brasses, with particular attention to the development of the alloys, alloy properties, and obstacles to their adoption for drinking-water applications.

4:40 PM

Synthesis of Open Cell Metal Foams by Plasma Enhanced, Electron Beam Directed Vapor Deposition: *Douglas Ted Queheillalt*[1]; Yasushi Katsumi[1]; Haydn N.G. Wadley[1]; [1]University of Virginia, Mats. Sci. & Eng., 116 Engineers Way, P.O. Box 400745, Charlottesville, VA 22904-4745 USA

Open cell, reticulated metal foams have been synthesized by a plasma enhanced, electron beam-directed vapor deposition (EB-DVD) process and their physical and mechanical properties evaluated. The deposition process uses an open cell, reticulated polymer or vitreous carbon foam template upon which is deposited various metal and alloy coatings. The electron beam evaporated flux was entrained in a rarefied transonic gas jet and propagated along the flow stream lines through the template structure. During vapor transport, the atoms pass through a plasma created by a hollow cathode arc discharge system. These combined technologies lead to high deposition rates of atoms with high energies, promoting enhanced coating quality. After vapor deposition, the template may be removed by thermal decomposition resulting in ultra-lightweight metal foams composed of a three-dimensional open cell, reticulated structure possessing triangular ligaments with either hollow or carbon cores.

High Temperature Coatings - IV: Ceramic Coatings

Sponsored by: Materials Processing and Manufacturing Division, ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee, Surface Engineering Committee

Program Organizers: Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Janet Hampikian, GA Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Wednesday PM, February 14, 2001 — Room: 219, Location: Ernest N. Morial Convention Center

Session Chairs: Narendra B. Dahotre, University of Tennessee Space Institute, Dept. of Mats. Sci. and Eng., Tullahoma, TN 37388; Jeff Th De Hosson, University of Groningen, Dept. of Appl. Phys., Groningen 9747 AG The Netherlands

2:00 PM Invited

Properties of WC/C Coatings Investigated with Cross-Sectional TEM and Nanoindentation: *Jeff Th De Hosson*[1]; Nuno J. Carvalho[1]; [1]Un. of Groningen, Dept. Appl. Phys., Nijenborgh 4, Groningen 9747 AG The Netherlands

This paper concentrates on the interplay between microstructure and properties of coatings studied with cross-sectional transmission electron microscopy and nano-indentation techniques. Coatings of multilayer tungsten carbide/carbon (WC/C) were deposited by physical vapor deposition (PVD) onto steel substrates. The coatings are composed by a chromium columnar interlayer to improve the adhesion to the substrate, an intermultilayer of WC and carbon, and an interlaminar amorphous WC/C multilayer structure. The mechanical properties were evaluated with respect to the hardness and the effective Young's modulus. The hysteresis loops are analyzed and discussed in detail. Moreover a new technique for cross-sectional transmission electron microscopy of the nano-indentations was developed to gain a better view of the interplay between the mechanical properties, the microstructure and the chemical composition of the system. The information obtained by this technique is also correlated with the load-displacement data from a nano-indentation cycle to reveal detailed deformation mechanisms

2:25 PM Invited

Electrophoretic and Electrolytic Deposition of Ceramic Coatings: *Igor Zhitomirsky*[1]; [1]McMaster University, Dept. of Mats. Sci. and Eng., 1280 Main St. West, Hamilton, Ontario L8S 4L7 Canada

Two processes were used to prepare oxide coatings by electrodeposition: the electrophoretic process (EPD) that is based on the use of suspensions of ceramic particles and the electrolytic process (ELD) that starts from solutions of metal salts. ELD enables formation of nanostructured thin films. EPD is an important tool for preparation of thick ceramic films and body shaping. The feasibility of electrolytic deposition of various ceramic materials has been demonstrated. It was shown that chemical problems related to ELD of some important ceramic materials could be solved by use of specific precursors. An important finding was the possibility of electrochemical intercalation of cationic polyelectrolytes with inherent binding prop-

erties into ELD deposits. Advanced solvent-binder-dispersant systems were developed for deposition of consecutive ceramic layers of controlled thickness in multilayer EPD processing. Thin and thick films were deposited as monolayers or laminates on various substrates, including metals, conducting ceramics, carbon fibers and mats, platinized silicon wafers. The results presented involve the examination of the microstructure, composition and crystallization behavior of the deposits. Deposition yield was evaluated under various experimental conditions. Mechanisms of EPD and ELD, new developments and applications of the two methods are discussed.

2:50 PM

Yttria Stabilized Zirconia/Alumina Coatings Deposited by Combustion Chemical Vapor Deposition: David W. Stollberg[1]; Monique McIntosh[1]; W. Brent Carter[1]; *Janet M. Hampikian*[1]; [1]Georgia Institute of Technology, Mats. Sci. and Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Yttria stabilized zirconia/alumina (YSZ/alumina) thin films were grown on sapphire substrates by combustion chemical vapor deposition, using yttrium and zirconium 2-ethylhexanoate and aluminum acetylacetonate as precursors. These compounds were dissolved in toluene and burned in a flame to produce five different compositions of YSZ/alumina: 15, 30, 45, 62.8 (the eutectic composition) and 80 mol% alumina. The mechanical properties of these films were investigated as a function of alumina content using nanoindentation. The highest fracture toughness of the films was found to occur at alumina compositions between 30 and 45%. The thermal stability of some films deposited at the eutectic composition was investigated as a function of temperature and annealing time. The as deposited microstructure exhibits particle sizes at the nanometer level, characterized by transmission electron microscopy. Particle sizes increased on average to approximately 0.4 microns after annealing for 5 hours at 1500ºC.

3:10 PM

High Temperature Oxidation of VC Coated H13 Steel: *Swapnil V. Shah*[1]; Narendra B. Dahotre[1]; [1]University of Tennessee Space Institute, Dept. Mats. Sci. & Eng., Cen. for Laser Appl., 411 B.H. Goethert Parkway, Tullahoma, TN 37388 USA

ASISI H13 is the most widely used material for die-casting dies. High temperature oxidation of the die material during service affects die lifetime. Hard ceramic coating like VC can significantly increase the die life. In the present study attempts are made to coat H13 by VC using laser surface engineering technique. Oxidation behavior of coated H13 steel at high temperature (up to 1000ºC) is studied along with detailed microstructural and mechanical properties evaluation.

3:30 PM

Near Net Shape Forming of Hafnium-Based Ceramic Components: Synthesis and Characterization: *Arvind Agarwal*[1]; Tim McKechnie[1]; Samuel J. Causey[2]; Mark M. Opeka[3]; [1]Plasma Processes, Inc., 4914 D Moores Mill Rd., Huntsville, AL 35811 USA; [2]Southern Research Institute, 757 Tom Martin Dr., Birmingham, AL 35211 USA; [3]Naval Surface Warfare Center, Carderock Div., Code 681, 9500 McArthur Blvd., West Bethesda, MD 20187-5700 USA

Ultrahigh temperature applications such as combustion chamber liners, rocket thrusters, thermal protection systems for carbon/carbon composites and leading edges of the spacecraft require materials, which are protective and oxidation resistant at temperatures higher than 20000C. Refractory ceramics such as hafnium diboride (HfB2), hafnium carbide (HfC) and hafnium nitride (HfN) are candidate materials because of their high melting points, low coefficient of thermal expansion, high erosion and oxidation resistance. Conventionally, processing of these ceramics has been difficult and very expensive due to their intrinsic brittle nature. Near net shape forming of these ceramics using vacuum plasma spray technique offers a cost effective method to fabricate structural components. In the present investigation, it has been demonstrated that HfB2, HfC and HfN can be spray formed to near net shapes. Microstructural characterization for density and grain size distribution has been performed using optical and scannin g electron microscope (SEM). Phase identification and chemical characterization have been carried out using x-ray diffraction (XRD) and energy dispersive spectroscopy (EDS).

3:50 PM Break

4:05 PM

Environmental Barrier Coatings For Ceramic Matrix Composites: Evaluation After Exposure To Simulated Combustor Environments: *Karren L. More*[1]; Peter F. Tortorelli[1]; Larry R. Walker[1]; James R. Keiser[1]; Harry E. Eaton[2]; Gary D. Linsey[2]; Joshua B. Kimmel[3]; Jeffrey R. Price[3]; [1]Oak Ridge National Laboratory, Met. and Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6064 USA; [2]United Technologies Research Center, East Hartford, CT; [3]Solar Turbines, Inc., San Diego, CA USA

Recent gas turbine engine tests utilizing SiC/SiC composite combustor liners as well as laboratory exposures of similar materials to elevated water-vapor pressures and temperatures have demonstrated rapid oxidation/degradation of these materials under typical engine operating conditions. In order to achieve the lifetime goals for SiC-based composite materials in engine applications such as combustor liners, protective coatings for the composite materials will be required. Oxide-based environmental barrier coatings (EBCs) have been developed specifically for these SiC/SiC composite materials. The EBCs have been exposed for >6000 h in the laboratory under conditions chosen to best simulate the key degradation mode in combustion environments and are also currently in an engine test which has been running for >10,000 h. This work will focus on the microstructural evaluation of several EBC and composite systems and their stabilities after exposures in both laboratory facilities and gas-turbine engines. Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Power Technologies, Continuous Fiber Reinforced Ceramic Composite Program, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:25 PM

Oxidation-Resistant CVD Mullite Coatings with Controlled Composition and Microstructure: *Svetlana M Zemskova*[1]; J. Allen Haynes[1]; Kevin M. Cooley[1]; [1]Oak Ridge National Laboratory, Met. & Ceram. Div., P.O. Box 2008, MS 6063, Oak Ridge, TN 37831-6063 USA

Application of silicon-based ceramics as high-temperature structural materials requires the development of environmentally-resistant coatings due to rapid degradation in high temperature water vapor. The CVD method of fabricating dense, high-purity, crystalline mullite (3Al2O3x2SiO2)coatings on silicon nitride was utilized in this study. The textured coatings were generally comprised of crystalline grains (0.3-0.5μm) with a columnar structure. Coatings with thickness of less than 2 μm, provided excellent corrosion protection for silicon nitride exposed to low-velocity, high pressure (10 atm) steam at 1200ºC.

4:45 PM

The Spectral Emittance and Stability of Coatings and Textured Surfaces for Thermophotovoltaic (TPV) Radiator Applications: *Brian Vern Cockeram*[1]; Dorothy P. Measures[1]; Jim L. Hollenbeck[1]; [1]Bechtel Bettis, ZAP 08D/MT, P.O. Box 79, West Mifflin, PA 15122 USA

Coatings and surface modifications are needed to improve the surface emissivity of materials under consideration for TPV radiator applications. The wavelengths of photons emitted from the surface of the TPV radiator should ideally match the range of wavelengths that correspond to the bandgap of the TPV cells for optimum efficiency. Vacuum plasma spray coatings (ZrO2+18% TiO2+10% Y2O3, ZrC, Fe2TiO5, ZrTiO4, ZrO2+8% Y2O3+2% HfO2, and Al2O3+TiO2) and a chemical vapor deposited coating of rhenium whiskers have been shown to be thermally stable and have produced a desired increase in the surface emissivity of refractory metals and nickel-base materials [1]. These coatings are further evaluated by measuring the spectral emissivity before and after long-term vacuum anneals. The microstructure of the coatings is evaluated to characterize the thermal stability of the coatings after long-term vacuum annealing. 1B.V. Cockeram, D.P. Measures, and A.J. Mueller, Thin Solid Films, 355/356 (1999), pp. 17-25.

5:05 PM

Friction and Wear Propertiesof CVD-Coated Diamond at Room Temperature: *Ali Soleman Al-Watban*[1]; [1]Riyadh Technical College, P.O. Box 53699, Riyadh 11593 Saudi Arabia

In this paper, the room temperature friction and deformation properties of a CVD coated polycrystalline diamond were studied when softer metallic and ceramic sliders were used. The experiments were conducted at relatively low sliding speeds (10 mm/min), in a vacuum of 0.0001 mbr after having cleaned the specimen surfaces by out-gassing them for about 30 min at 800ºC. As a result of increasing the number of traversals significant wear of the coated diamond by softer metallic sliders (aluminum and mild steel) was observed. With ceramic sliders, it is shown that the multiple traversals result in the formation of wear groove. For reasons of comparison, the same technique has been applied to type I single crystal diamond.

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Laser Processing of Ultrahard Coatings: *Ashok Kumar*[1]; [1]University of South Florida, Dept. of Mech. Eng., Center for Microelec. Res., Tampa, FL 33620 USA

The development of superhard coatings with high level of hardness, wear resistance and toughness is an important area of research with numerous applications. Our research is focused on the development of carbide and nitride coatings by the Pulsed Laser Deposition (PLD) method. The PLD is a vapor deposition process that combine near atomic-level control of the surface composition with the high kinetic energies of the condensing vapor produced by the laser ablation. Thin film coatings of carbides (titanium carbide, silicon carbide and boron carbide) and nitrides (titanium nitride, silicon nitride, and aluminum nitride and carbon nitride) were deposited on Si (100) substrates using PLD method. The structural and microstructural properties of these films have been characterized using x-ray diffraction, scanning and transmission electron microscope techniques. The mechanical properties of the films were evaluated to measure the hardness and modulus values. Microlaminate made of alternate layers of TiN and TiB2 films is likely to offer promises of exceptionally high hardness and modulii and, therefore, can be employed for future protective coatings. Single layer of TiN, TiB2 and TiB2/TiN microlaminate coatings with varying thickness were initially deposited on Si(100) and oxidized Si(111) substrates by pulsed laser deposition techniques and then characterized by x-ray diffraction, transmission electron microscopy and nano-indentation methods. Analysis of the resulting data revealed that the elastic modulus and hardness of multilayer coatings are superior to monolithic coatings of either of the two constituent films. It is suggested that the smooth nature of the interface between TiN and TiB2 is responsible for the improved hardness. This work was supported by NASA EPSCoR program.

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Effect of Enamel Coating on The Corrosion of Titanium Alloys: *Yuming Xiong*[1]; *Fuhui Wang*[1]; *Weitao Wu*[1]; *Yan Niu*[1]; [1]Institute of Corrosion and Protection of Metals, State Key Lab. for Corr. and Prot., Wencui Rd. 62, Shenyang, Liao Ning 110016 China

The air oxidation and corrosion induced by the synergistic effect of solid NaCl deposits and water vapor of the titanium alloys with an enamel coating have been investigated. The results revealed that the enamel coating markedly improved the oxidation resistance of the alloy at 600-800ºC in air, since the coating possesses a good thermal chemical stability and a thermal expansion coefficient matching well with titanium alloys. The coating showed good corrosion resistance to solid NaCl deposits in mixtures of oxygen with water vapor at 600ºC. The enamel coating can also improve long-term oxidation of the alloy in air at 600ºC, and acts as the barrier for suppressing the migration of oxygen into the substrate, thus prevents the alloys from oxygen embrittlement.

International Symposium on Deformation and Microstructure in Intermetallics: Microstructure II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Physical Metallurgy Committee, Jt. Mechanical Behavior of Materials

Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Peter M. Hazzledine, UES Inc., Dayton, OH 45432 USA

Wednesday PM — Room: 220
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Robert A. Varin, University of Waterloo, Canada; Sung H. Whang, Polytechnic University, Dept. of Mech. Eng., Brooklyn, NY 11201 USA

2:00 PM Invited

Complex Faults in Boron-Containing B2 Iron Aluminides: *Sharvan Kumar*[1]; Lixin Pang[1]; [1]Brown University, Div. of Eng., Box D, Providence, RI 02912 USA

Complex faults have been observed on the {001} planes in an Fe-40Al-0.7C-0.5B alloy in the as-extruded condition. Six variants of such faults can occur; these faults are characterized by a missing aluminum plane and an in-plane shift of 1/2<001>. Boron is claimed to segregate at such faults. These faults exhibit APB and stacking fault contrast when examined using a superlattice and fundamental reflections respectively. A variety of heat treatments were conducted to understand the origin and evolution of these faults as well as conditions under which they occur. These results will be presented and possible formation mechanisms will be discussed.

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Relating Dislocation Core Observations with Yielding Behavior in Ni_3Ge-Fe_3Ge $L1_2$ Alloys: *Kevin J. Hemker*[1]; T. John Balk[1]; [1]Johns Hopkins University, Mats. Sci. and Eng. and Mech. Eng., 200 Latrobe Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

Ni_3Ge-Fe_3Ge has been found to be a model system for relating the macroscopic mechanical behavior of $L1_2$ intermetallic alloys with atomic level dislocation core structures. Increases in iron content cause a gradual transition from anomalous to normal behavior, and remarkable low temperature strengthening. Transmission electron microscopy (TEM) and image simulations have been combined to determine the operative deformation mechanisms and to perform detailed measurements of superdislocation dissociations. The transition in yield strength behavior has been correlated with a transition from octahedral glide and cross-slip locking to cube glide, while low temperature strengthening coincides with enhanced cross-slip. The propensity for cross-slip has been related to a significant drop in cube plane antiphase boundary energy. It has also been noted that low temperature strengthening and the transition to cube glide are both consistent with an increase in complex stacking fault energy.

3:00 PM

Dipolar and Multipolar Defects in Al-Rich Gamma-TiAl and the Motion of Ordinary Dislocations: *Patrick Veyssiere*[1]; Fabienne Gregori[2]; [1]LEM, Cnrs-onera, Bp 72, Chatillon Cedex 92322 France; [2]LPMTM, Institut Galilee, 99 Av. J. B. Clement, Villetaneuse 93430 France

The generation and the organisation of single and multipolar prismatic loops is studied in Al-rich γ-TiAl single crystal strained to 2% at room temperature. In the near-[153] load orientation slip takes place on one single slip plane and mostly by ordinary dislocations. It is shown that the partial annihilation of mixed dipoles by local cross-slip contributes to the population of prismatic loops on which mobile dislocations are pinned. These are in turn annihilated by impacting mobile dislocations forming new loops. As the pinning/annihilation process repeats itself, prismatic loops gradually aggregate in certain locations of the sample forming loose walls. On the other hand, elastic interactions between ordinary dislocations and the longest prismatic loops may give rise to multipoles comprising a limited number of branches the extremities of which are aligned

with the screw direction. Elongated prismatic loops and multipoles may interact with impacting dislocations forming trailing-like configurations on both mixed and screw segments. This process is at the origin of a significant fraction of the dipole-decorated ordinary dislocations that constitute one of the essential characteristics of the deformation microstructure of γ-TiAl deformed in the low temperature domain of the flow stress anomaly. These processes are at variance from trailing mechanisms considered so far in order to explain dipole-decorated dislocations and to design models of the flow stress anomaly.

3:20 PM

Slip and Twin Interactions with Polytwin Interfaces in L10-ordered Intermetallics: *Jörg M.K. Wiezorek*[1]; William A. Soffa[1]; [1]University of Pittsburgh, Dept. Mat. Sci. & Eng, 848 Benedum Hall, Pittsburgh, PA 15261 USA

Polytwinned (PT), lamellar microstructures readily form via solid-state transformations in alloys based on intermetallic phases with the ordered tetragonal L1o-structure, e.g. TiAl and FePd. The PT-composition planes are common {111} in TiAl and common {101} in FePd. The mechanical properties of PT-grains are anisotropic with "hard" and "soft" orientations. For "hard" oriented grains shear transfer across PT-interfaces must occur and appears to be of particular importance to the development of quantitative descriptions of the behavior of this type of compounds. The deformation modes active in these L1o-phases involve slip of super- and ordinary dislocations, as well as typically significant amounts of ordered twinning. Methods of scanning and transmission electron microscopy have been used to investigate details of these transfer processes. Using crystallographic analyses based on the Thompson-tetrahedron adapted to the L1o-structure possible mechanisms for shear transfer across the distinct configurations at {111}- and {101}-conjugated PT-interfaces have been identified. These theoretically derived transfer mechanisms are compared to experimental observations in TiAl and FePd. Results of this study are related to the physical and mechanical behavior of PT-L1o-intermetallics.

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Twinning Processes in Creep-Deformed Lamellar TiAl: *Luke L. Hsiung*[1]; T. G. Nieh[1]; [1]Lawrence Livermore National Laboratory, Chem. and Mats. Sci., L-369, P.O. Box 808, Livermore, CA 94511 USA

Deformation twinning in creep-deformed lamellar TiAl has been found to be intimately related to the motion, pile-up and dissociation of interfacial (Shockley partial) dislocations. Since the interfacial (Shockley partial) dislocations are energetically less favorable to undergo cross-slip or climb, under normal conditions they can only move conservatively along interfaces. Consequently, the pile-up configuration once generated cannot be easily dissipated and thus remain in place even at elevated temperatures. The dislocation pile-up eventually leads to the emission of deformation twins from the interfaces into γ lamellae. In addition to the {111}<112]-type twinning, {112}<111]-type twinning has also been identified. The critical shear stresses for different twinning processes are evaluated based upon the pile-up and core dissociations of interfacial dislocations. This work was performed under the auspices of the U.S. Department of Energy through contract #W-7405-Eng-48 with Lawrence Livermore National Laboratory.

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Deformation Structure during Creep Deformation in Soft Orientation PST Crystals: *Hee Young Kim*[1]; Kouichi Maruyama[1]; [1]Tohoku University, Grad. Sch. of Eng., Dept. of Mat. Sci., Aobayama 02, Sendai 980-8579 Japan

The creep deformation behavior and microstructural evolution of polysynthetically twinned (PST) crystals Ti-48Al were investigated. The soft orientation with the lamellar plates oriented 35° to compression axis were deformed at 1150K under the applied stress of 100-400 MPa. Deformations took place in the maximum shear stress direction in the soft orientation. The dislocation structures in γ domains of six different orientation variants were examined by transmission electron microscopy, and operative slip and twinning systems were analysed. The macroscopic plastic strain and strain compatibility at domain and lamellar boundaries were discussed with associated the slip systems in each domain. The refinement of lamellae occurred by mechanical twinning during creep deformation. The effect of applied stress and strain on activity of twining was investigated quantitatively during creep deformation.

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Fracture Initiation in Gamma-TiAl Related to Crystallographic and Microstructural Features Studied in 4-point Bending: Benjamin Andrew Simkin[1]; Martin A. Crimp[1]; Thomas R. Bieler[1]; [1]Michigan State University, Dept. of Matls. Sci. and Mech., 3536 Engineering, East Lansing, MI 48824-1226 USA

Sub-critical surface fractures in 4-point bend samples of a duplex gamma-TiAl based alloy (Ti-48%Al-2%Nb-2%Cr) are characterized according to their relation to microstructural and crystallographic features of the material. Some fractures appear to initiate in the vicinity of Ti3Al particles due to apparent stress concentrations. Other fracture initiation sites occur at grain boundaries, and often are associated with the impingement of deformation twins. These initiation sites are characterized via electron channeling contrast imaging (ECCI) and electron backscatter patterns (EBSP). Using these methods, the local grain orientation, crack orientation relative to the principal stress axies, and the deformation defects in the vicinity of the crack are determined. An attempt is made to correlate macroscopic texture to the eventual plastic strain to failure.

International Symposium on Shape Casting of Aluminum: Science and Technology: Microstructural Evolution During Solidification and Heat Treatment

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Aluminum Committee, Non-Ferrous Metals Committee, Solidification Committee, Jt. Mechanical Behavior of Materials

Program Organizers: John E. Allison, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48124-2053 USA; Dan Bryant, Chester, VA 23836-3122 USA; Jon Dantzig, University of Illinois, Department of Mechanical & Industrial Engineering, Urbana, IL 61801-2906 USA; Ray D. Peterson, IMCO Recycling, Inc., Rockwood, TN 37854 USA

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February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Murat Tiryakioglu, Western Kentucky University, Dept. of Manuf. Sci., Bowling Green, KY 42101-3576 USA; Arne K. Dahle, The University of Queensland, Dept. of Min., Brisbane, Australia

2:00 PM Keynote

Formation of Shear Defects in Pressurised Casting Processes: *D. H. StJohn*[1]; A. K. Dahle[1]; [1]University of Queensland, CRC for Cast. Mets. Manuf. (CAST), Dept. of Min., Mins. and Mats. Eng., Queensland, Australia

Shear defects are formed in a casting when partially solidified material has to flow over extended distances. They are therefore often apparent in casting processes such as semi-solid casting and in those where pressure is being applied (e.g. in pressurised casting processes such as squeeze and high-pressure die-casting). On complete solidification the shear defects often take the form of bands of segregation, porosity and tears or a combination of these defects. The properties of the casting are thus adversely affected. This paper outlines the cause of shear defects in terms of the development of mechanical strength during solidification. It is shown that the development of shear strength during solidification can be sub-divided into four regimes that have distinct rheological properties. Identification of these regimes provides a basis for development of a simulation program for predicting the location and severity of shear defects. Strategies to control the formation of these defects

can be suggested based on the new concept of rationalising the origin and causes of these defects.

2:45 PM Invited

Understanding Eutectic Formation in Hypoeutectic Al-Si Alloys: *Arne K. Dahle*[1]; Kazuhiro Nogita[1]; Stuart D. McDonald[1]; Jacob W. Zindel[2]; [1]The University of Queensland, Dept. of Min., Mins. and Mats. Eng., Brisbane, Qld 4072 Australia; [2]Ford Motor Company, Ford Res. Lab., Mats. Sci. Dept., Dearborn, MI 48121 USA

Commercial hypoeutectic Al-Si foundry alloys contain between 50 and 90 vol% eutectic. Solidification of the eutectic is the last major solidification event and therefore the critical stage where casting defects, such as porosity, form in these alloys. Thermal analysis, microstructural inspection, quenching, and electron back-scattering diffraction (EBSD) mapping have been used to study the effects of a range of different modifying elements and levels on eutectic nucleation and growth. The results show that eutectic solidification can occur by three distinctly different nucleation and growth modes, in isolation or sometimes together, but controlled by the modifier element used and the concentration added. Furthermore, it has been found that eutectic nucleation, and the resulting macroscopic growth mode, is independent of the plate-like to fibrous transition in silicon morphology upon modification. The eutectic nucleation mechanism and resulting macroscopic growth pattern dramatically affect the mushy zone permeability and pressure drop and therefore controls both porosity content and distribution.

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Evolution of the Eutectic Microstructure during Solidification of Hypoeutectic Al-Si Alloys: *Hema Vardhan Guthy*[1]; Makhlouf Makhlouf[1]; [1]Metals Processing Institute-Worcester Polytechnic Institute, 100 Institute Rd., Worcester, MA 01609 USA

Commonly used Al-Si casting alloys are typically hypoeutectic with silicon ranging from 7 to 11wt%. Although these alloys generally possess a relatively short freezing range, good castability and desirable overall properties, their mechanical properties are influenced by porosity, shrinkage and segregation, which in turn are determined by the last stage of solidification, namely eutectic formation. In this investigation, the evolution of the eutectic microstructure in hypoeutectic Al-Si alloys was studied using thermal analysis and metallographic techniques. Quench experiments of various Al-Si alloys of commercial purity and of high purity were performed. The resulting microstructure was characterized using optical and scanning electron microscopy. In unmodified alloys, both the eutectic silicon and the eutectic aluminum originate from the primary aluminum. Eutectic aluminum grows continuously out of the primary aluminum without any re-nucleation while the eutectic silicon nucleates on the primary aluminum. On the other hand, in Sr-modified alloys, the eutectic silicon nucleates on the primary aluminum while the eutectic aluminum nucleates on the eutectic silicon. Moreover, equiaxed eutectic grains were observed in Sr modified alloys with impurity elements segregated along the grain boundaries but no specific morphology was observed in eutectic grains of unmodified alloys.

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4:00 PM Invited

Modelling of Microsegregation in Shape Cast Aluminum Alloys: *N. Saunders*[1]; B. Boutwell[2]; [1]Thermotech, Ltd., Surrey Technology Centre, The Surrey Research Park, Guilford, Surrey GU2 7YG UK; [2]AEAT Technology, Inc., 241 Curry Hollow Rd., Pittsburgh, PA 15236-4696 USA

In recent years, thermodynamic modelling has reached the stage where phase equilibria can be modelled with a high degree of accuracy for many of the major types of Al-alloys. Such modelling is also readily extendable to predicting non-equilibrium solidification behaviour under so-called "Scheil-Gulliver" conditions and highly accurate predictions for fraction solid, heat of solidification and the phases formed during solidification can be obtained. It is now of interest to see what effect a more explicit treatment of diffusion in the solid state would make and, to this end, the software package DICTRA will be used to model the solidification of an Al-Si-Cu based alloy. A potential advantage of using DICTRA is that annealing schedules after solidification can be taken into account and the effect of subsequent heat treatment on the reduction in micro-segregation of the cast product can be modelled. The presentation will (i) provide a review of previous solidification modelling and (ii) present results from a DICTRA simulation applied to an Al-Si-Cu based casting alloy.

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The Effect of Ti Additions on the Grain Size and Ageing Properties of Aluminium Alloy A356: *Mark Alan Easton*[1]; Joseph Barresi[2]; David H. StJohn[3]; [1]Monash University, Dept. of Mats. Eng., Wellington Rd., P.O. Box 69M, Clayton, Victoria 3800 Australia; [2]Comalco Research and Technical Services, Edgars Rd., Thomastown, Victoria 3074 Australia; [3]University of Queensland, Dept. of Min., Mins. and Mats. Eng., Brisbane, Queensland 4072 Australia

Al-Si foundry alloys usually contain up to 0.2wt% titanium, added for grain refinement. It was found, using results published in the literature and from experiments, that additions of titanium up to 0.2wt% cause only a minor or negligible decrease in grain size in an A356 alloy for a wide variety of casting conditions. Boron additions in the form of stoichiometric TiB2 master alloys, are much more effective at reducing the grain size and therefore should be considered as the preferred grain refining addition. As well as grain size it is important to consider whether Ti in solution plays a role in the age hardening response of Al-Si alloys and these results are presented. Considerations for determining the optimum titanium concentration are discussed.

5:00 PM

Modeling Thermal Growth During Heat Treatment in Aluminum Castings: *Chris Wolverton*[1]; John Allison[1]; [1]Ford Motor Company, MD3028/SRL, P.O. Box 2053, Dearborn, MI 48176 USA

Macroscopic, irreversible dimensional changes are notorious for occuring during heat treatment of aluminum alloys. These dimensional changes (often called "thermal growth" since they typically involve an expansion of the material rather than a contraction) can deleteriously affect the performance of a given alloy. We have investigated the thermal growth in cast 319 Al, determining that the precipitate phase transformation to the Al2Cu (Θ') phase is responsible for the growth. In conjunction with experimental data, we have used a variety of computational tools (first-principles quantum-mechanical calculations, computational thermodynamics methods, and microstructural evolution models) to construct a model of thermal growth. The model is capable of quantitative prediction of the temperature-, time-, and composition-dependence of thermal growth in 319, and accounts for multiple Cu-containing precipitate phases. Although we have constructed the model for 319 Al, this type of model should be generally applicable for thermal growth in other alloys as well.

Lead-Free Solder Materials and Soldering Technologies VI: Reliability, Electromigration, Applications

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Sung Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598 USA; Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Hareesh Mavoori, Lucent Technologies, Bell Laboratories, Murray Hill, NJ 07974 USA; Ronald W. Smith, Materials Resources International, North Wales, PA 19454 USA

Wednesday PM Room: 227
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Srinivas Chada, Motorola, APTC Dept., Plantation, FL 33322 USA; Ray Fournelle, Marquette University, Mats, Sci, & Eng., Milwaukee, WI USA

2:00 PM Invited

Pb-free Solders for Power Die Attach: *J.N. Lalena*[1]; Martin W. Weiser[1]; Nancy F. Dean[1]; [1]Honeywell Electronic Materials, 15128 E. Euclid Ave., Spokane, WA 99216

Power semiconductor devices such as IGBTs, MOSFETS, Bipolar Transistors, Rectifiers, and Thyristors are normally fabricated by soldering a Si die to a Cu leadframe that serves as a heat spreader. These die are often quite large (0.8 mm square is common) and have high heat output (>100 W is not uncommon). The solders used for these devices must have both good thermal conductivity to dissipate the heat and very good resistance to thermomechanical fatigue due to the large CTE difference between Si and Cu. Several high-Pb and a couple of high-Sn solder alloys have dominated this application for many years. The electronics industry is in the process of adopting Pb-free solders for board level assembly. Most of the proposed solders are based upon the Sn-Ag, Sn-Cu, and Sn-Ag-Cu eutectics which melt between 210 and 230ºC. As a result these solders are reflowed at 235 to 270ºC depending upon the solder, process, and thermal mass of the system that is being assembled. Of the current high volume die attach solder alloys only the high-Pb solder alloys will survive these reflow temperatures-too much liquid phase forms in the high-Sn alloys. We have looked at a variety of systems to find Pb-free alloys that meet the stringent requirements of a die attach solder for power semiconductor devices. We will discuss the requirements in detail and how some of the most promising systems stack up against these requirements. Some alloys such as Zn4Al3Mg3Ga discussed in a previous year by Shimuzu et. al. have promising melting and wetting behavior, but have inadequate mechanical properties.

2:20 PM

Board Level Reliability of Pb-free and Pb-bearing Polymer-Core Solder Balls for CSP Application: *Seung Wook Yoon*[1]; Jong Heon Kim[1]; Shin Choi[1]; Ik Seong Park[1]; Heung Sup Chun[1]; [1]Hyundai Electronics Industries Company, Ltd, Semiconductor Group, Ichon, Kyunggi-do 467-701 Korea

Chip scale packages (CSP) have essential solder joint quality problems, and a board level reliability is a key issue in design and development of the CSP type packages. And many countries such as Japan and Europe, have proposed laws reducing or eliminating the use of Pb and other toxic substance in an effort to decrease landfill pollution and ground water contamination. This paper is focused on the results of investigations on polymer-core solder ball with Pb-bearing as well as Pb-free solder for CSP applications. The solder-joint reliability of a LFCSP(Lead-Frame CSP) and WLCSP(Wafer Level CSP) on printed circuit board (PCB) under thermal fatigue is studied. The solder joints are subjected to thermal cycling and their lifetime, crack initiation site, crack propagation mode are observed. Their solder joint life times were compared with conventional Sn-Pb eutectic solder and microstructures were also examined by OM, SEM and EDX. In order to find out the role of polymer core in solder joint, the stress/strain intensity factors at the solder joint region are investigated by fracture mechanics with FEM (finite element method)computer simulation.

2:40 PM

Ball Shear Strength and Microstructure of Eutectic Tin Lead and Lead Free Solder Ball After Reliability Test: *P. J. Zheng*[1]; S. C. Lee[1]; J. Z. Lee[1]; J. G. Hwang[1]; Jim C. L. Wu[1]; [1]Advanced Semiconductor Engineering, Inc., 26, Chin 3rd Rd., Nantz Export Processing Zone, Kaohsiung City, Taiwan

Currently, the alloy materials used as BGA solder ball are mostly eutectic tin lead materials such as 63wt%Sn/37wt%Pb and 62wt%Sn/36wt%Pb/2wt%Ag. Due to environmental pollution concern, e.g., landfill pollution and ground water contamination, many countries have legislated to reduce or eliminate the use of lead. This comes out the massive need of lead free alloy to be used in electronic components. However, because the mechanic property of lead free alloy is quite different from eutectic tin lead, the process parameters used in PBGA assembly flow need to be rebuilt. In addition, the reliability of the corresponding PBGA products also needs to be evaluated. Since the cost deviation between lead free and eutectic solder ball is still a key concern, how to select a suitable and cost-effective lead free material becomes an important issue. In the study, the shear strength and microstructure of lead free solder ball versus eutectic solder ball after reliability test is investigated. Two kinds of eutectic tin lead materials 63wt%Sn/37wt%Pb and 62wt%Sn/36wt%Pb/2wt%Ag and two kinds of lead free alloys 96.5wt%Sn/3.5wt%Ag and 95.5wt%Sn/4.0wt%Ag/0.5wt%Cu are selected to be solder balls in 618 PBGA (with 0.76 mm ball diameter). After the assembly, the samples are first preconditioned under JEDEC level 3 standard. Temperature cyclic test (TCT), thermal shock test (TST), temperature and humidity test (THT), pressure cook test (PCT) and high temperature storage test (HTST) are followed after the precondition test. The ball shear strengths of solder balls are respectively measured right after assembly, pre-conditioned and the end of each reliability test. Our preliminary result shows that 96.5wt%Sn/3.5wt%Ag possess highest shear strength. For example, at the end of 1000 cycles of TC test, the shear strength is 1500 g for 96.5wt%Sn/3.5wt%Ag, which is 1.5 times higher than other three materials. Optical microscope and SEM are used to observe the failure mode of shear test after each reliability test. SEM and EDX are applied to analyze the different intermetallic compounds (IMC) that formed at solder joint of the four solder ball alloys. The analysis results will be presented and discussed in the final paper.

3:00 PM

Chip-on-glass(COG) Mounting Using a Laser Beam Transmitting Glass Substrates: *Jong-Hyun Lee*[1]; Won-Yong Kim[2]; Dong-Hoon Ahn[2]; Yong-Seog Kim[1]; [1]Hong Ik University, Mats. Sci. and Eng., Mapo-Cu Sangsu-Dong 72-1, Seoul 121-791 Korea; [2]Yeonwoo Engineeing Company, Ltd., Choong-Ri #425, Koosung-Myun, Yongin-Si, Kyungki-Do 449-910 Korea

For Chip-on-glass(GOG) electronic packaging in liquid crystal display, anisotropic conductive film(ACF) is being used extensively. The reliability and reworkability of the joints need to be improved further for extended applications of the COG mounting method. In this study, COG mounting process was attempted by local heating of solder bumps using a laser beam transmitted through glass substrate. The laser beam from diode transmitted the glass was absorbed by a metal pad and consequently heated Sn-Ag-Cu solder or solder paste in contact with the pad. The pad materials consisted of adhesion layer (i.e. Cr or Ti) and solderable layer (i.e. Ni or Cu). The interfacial microstructure and mechanical properties of the joint were investigated at different energy input rates, radiation times and pad materials.

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Electroless Ni/Immersion Au Finish: Impact on Interconnection: *Polina Snugovsky*[1]; Peter Arrowsmith[1]; Marianne Romansky[1]; [1]Celestica, Proc. Eng. Dev., 844 Don Mills Rd., 33/178, Toronto,

Ontario M3C 1V7 Canada; [1]Celestica, Inc., Techn. Assurance Labs., 844 Don Mills Rd., 20/149, Toronto, Ontatio M3C 1V7 Canada

Electroless Nickel/Immersion Gold finish is widely used in the electronic industry. Weak lifted joints, which exhibit a dark nickel-like surface on the conductive pads were detected and named "black pad". Many reports in the literature in recent years described cases of poor solder joints on electroless Ni/immersion Au. Celestica also face the black pad issue, and accumulated some knowledge in recognizing and reworking assemblies showing this phenomena. The black pad case reported here has some unique features. The investigation and finding of black pad in SMT processes is described. The presentation and the manuscript will include: Black pad occurrence and failure mechanism in wire bonding; Effect of board construction processes on black pad formation; Thick Au and thin Ni appearance on defective pads; Au and Ni layer composition; Mechanism of defective electroless Ni/immersion Au deposition; Weak solder joint on thick Au-black pad; Comparison of intermetallic formation during SMT on good and bad pads: microstructure, kinetics; SMT parameters influence on solder joint formation on bad pads; Rework reliability

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Electromigration in SnPb Solder Alloys: *Cheng-Yi Liu*[1]; Q. T. Huynh[2]; King-Ning Tu[2]; [1]Intel, Component Research, 5000 W. Chandler Blvd., Mail Stop Ch5-166, Chandler, AZ 85226 USA; [2]UCLA, Dept. of Mats. Sci. & Eng., Los Angeles, CA USA

Both thin film and thick film of SnPb solder samples were prepared for electromigration. The thin film samples were of 1 mm in thickness with Cu films as electrodes. They were tested at current density of 1 x 105 amp/cm2 and at room temperature, resulting in the formation of a large number of hillocks of Sn at the anode and voids at the cathode. The dominant interfacial diffusing species was found to be Sn. The thick film samples were SnPb solder lines reflowed in V-grooves on (001) Si wafer surfaces. The width and depth of the V-groove were 100 mm and 69 mm, respectively. The electrodes were Cu wires inserted in the two ends of the V-groove before solder reflow. These thick solder lines were also tested at current density of 1 x 105 amp/cm2 but at 150°C, resulting in the formation of a large lump of accumulation of both Pb and Sn at the anode and a large void at the cathode. The dominant bulk diffusing species was observed to be Pb. We shall discuss the different electromigration behaviors in these two kinds of samples. Besides those changes occurring at the anode and cathode, there was a substantial amount of microstructure evolution in the films and lines, owing to grain growth and phase separation. Also we will present the electromigration behavior as a function of SnPb alloy composition, as in the 95Pb5Sn and pure Sn samples.

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Whisker Formation Study of Lead-free Sn-Cu Plated Packages by Pressure Cook Test: *Jim C.L. Wu*[1]; Jeffrey C. B. Lee[1]; Y. S. Chou[1]; C. H. Chang[1]; [1]Advanced Semiconductor Engineering, Inc, 26, Chin 3rd Rd., Nantz Export Processing Zone, Kaohsiung City, Taiwan

Tin-lead (85%/15%) plating system has been widely used in the package production line for more than ten years. It offers the best in classes from the production efficiency and cost. Due to the environmental and human being health concerns, some of the area, for example, Europe and Japan, has legislated that lead is going to be reduced or totally eliminated before next decade. Therefore, to develop another plating technology and system toward lead-free package is of great interested not only from novel new technology availability but also from market share business-wised consideration. There are several popular alloy system candidates in the market, such as pure Sn, Sn-Ag, Sn-Bi and Sn-Cu. However, tin rich alloy has been reported that they have whisker growth latent risk in the lead finishing. Therefore, how to develop a time effective acceleration test and setup a reject criteria to screen the material, process and reliability evaluation are of significant importance. In this report, pressure cook test(PCT) was applied to study the whisker formation of Sn-Cu lead finishing during the reliability test. The test vehicles include PLCC-44L, QFP-208L and TSOP-48L. The impact of percentage of copper on lead formation and reliability tests is also investigated. The influences with various leadframe materials, chemicals, stress/forming and copper contents on the microstructure were also scrutinized by SEM, EDX, and X-ray. The preliminary results showed that the chemical and leadframe materials play important roles on whisker growth latent risk. Copper diffusion during the PCT test was also reported.

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Chemistry, Microstructure and Mechanical Properties of Sn-Bi/Cu Interface: *Jian-Ku Shang*[1]; Pi Lin Liu[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mats. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA

Bi-containing alloys are being explored as potential replacements to the Sn-Pb eutectic alloy in the solder interconnect. While most of the attention has been focused on the bulk solder alloys, the metallurgical compatibility of these alloys with the common metallizations such as copper and nickel has not been adequately investigated. In this study, the compatibility of the eutectic Sn-Bi alloy with copper metallization was investigated by examining the chemistry, microstructure, and mechanical properties of the Sn-Bi/Cu interface in the as-reflowed and aged conditions. While the mechanical properties of the Sn-Bi/Cu interface were slightly inferior to those of the eutectic Sn-Pb/Cu interface in the as-reflowed condition, drastic weakening of the Sn-Bi/Cu interface was found after the interface was artifically aged. Such a weakening effect was shown to result from the unique interfacial chemistry and microstructure developed by the aging treatment.

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The Characteristics of Vibration Fracture of Pb-Sn and Lead-Free Sn-Zn Eutectic Solders: *Chiang-Ming Chuang*[1]; *Truan-Sheng Lui*[1]; Li-Hui Chen[1]; [1]National Cheng Kung University, Mats. Sci. and Eng., Tainan, Taiwan 701

This work investigates the characteristics of vibration fracture of Sn-Zn lead-free solder and Pb-Sn solder. The frequency of all most fatigue studies about solders were below 1 Hz, in the past. However, when these electronic components include solder joints were assembled in the vehicles, the frequency of vibrant situation is very different. The experimental materials of rapid solidification were tested as tension and vibration during unstable state to stable state in room temperature. The experimental results show that the tensile strength of Pb-Sn solder and Sn-Zn lead-free solder after rapidly solidified decrease with aging time increasing, then tend to stable after a period days. The results of vibration test show that, in the same vibration force, Sn-Zn lead-free solder has smaller specimen's end deflection and higher crack propagation resistance. Oppositely, when the initial specimen's end deflection of two experimental materials was similar, the strain in the notch area was equal, the traditional Pb-Sn solder has batter crack propagation resistance.

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Electromigration Effect upon the Sn-0.7wt%Cu/Ni and Sn-3.5wt%Ag/Ni Interfacial Reactions: *Chih-ming Chen*[1]; Sinn-wen Chen[1]; Mei-yau Du[1]; [1]National Tsing-Hua University, Dept. of Chem. Eng., #101 Kuang-Fu Rd., Sec. 2, Hsin-Chu, Taiwan 300 Taiwan

Electromigration refers to the phenomenon that the passage of electric currents induces the movement of metallic atoms in metals. This study investigates the effect of electromigration upon the interfacial reactions between the promising lead-free solders, Sn-Cu and Sn-Ag, with Ni substrate. Sn-0.7wt%Cu/Ni and Sn-3.5wt%Ag/Ni couples reacted at various temperatures with and without the passage of electric currents were examined. Only one intermetallic compound Ni_3Sn_4 was found at the interfaces of all the couples reacted at 160°C, 180°C, and 200°C. The growth rates of the Ni_3Sn_4 phase were either enhanced or retarded by the passage of a 500 A/cm^2 electric current depending on its flow direction. The effectiveness of the passage of electric currents decreased with increasing temperatures.

Lightweight Alloys for Aerospace Applications: Deformation, Fatigue and Environmental Fracture-I

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee

Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Nack J. Kim, Center for Adv. Aerospace Materials, Pohang 790-330 Korea; Eui W. Lee, Naval Air Warfare Center, Code 4342, MS5, Patuxent River, MD 20670 USA; William Frazier, Naval Air Warfare Center, Aircraft Division, Patuxent River, MD 20670-1908 USA

Wednesday PM — Room: 213
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: O. S. Es-Said, Loyola Marymount University, Los Angeles, CA USA

2:00 PM Invited

The Effect of Multi-Axiality on Creep/Fatigue Failure in Gamma Titanium Alloys at Elevated Temperatures: *Kamran Nikbin*[1]; [1]Imperial College, MED, Exhibition Rd., London SW13 9NA UK

The thermal efficiency of a gas turbine is only around 38 % with a gas entry temperature of 1200K, whilst at 1800K the efficiency rises to over 50 % [1], hence there is a strong demand for materials capable of operating at ever increasing temperatures. In response to this considerable research has and indeed is continuing to be performed on intermetallic materials such as g-TiAl. The low-pressure turbine section, where blade temperatures up to 750ºC and blade root temperatures of 500ºC are commonplace, is seen as a typical environment for this material. g-TiAl possesses some particularly attractive properties such as low density (approximately half that of nickel-based superalloys), and good creep resistance, however it presents the engineer with problems not associated with conventional materials. The paper will deal with and analyse the differences in behaviour, in creep and creep/fatigue under multi-axial stress state, of different batches of Gamma- Titanimum with similar composition and heat treatment.

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Dwell-Fatigue Behavior of Ti-6242 Alloy: *V. Sinha*[1]; M. Savage[1]; J. Tatalovich[1]; M. J. Mills[1]; J. C. Williams[1]; [1]The Ohio State University, Dept. of Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

The current understanding of the dwell-fatigue behavior of a/b titanium alloys will be reviewed. The results of this review will be used to motivate a discussion of a recent research results obtained for a typical high temperature a/b titanium alloy, Ti-6242. This alloy is very extensively used for rotors in the compressor section as multi-stage spools of the aeroengines. A significant deficit in the fatigue life of this class of alloys has been reported in the literature under dwell-fatigue conditions at room temperature and high stress levels when compared with that under continuous cycling conditions. In this study, the effects of microstructure (i.e. lamellar/ Widmanstätten vs. globular) on the dwell-fatigue response of Ti-6242 are being investigated. Furthermore, the influence of microtexture (in the equiaxed a/b forged microstructure) on the dwell-fatigue life is also being examined. The globular microstructures of two kinds are investigated: (i) having randomly oriented a grains, and (ii) having maximum microtexture. The effects of hydrogen content are also examined in some detail. In the end, some possible crack initiation and propagation mechanisms are discussed and supported by scanning electron microscopy and orientation imaging microscopy results. (This work is being supported by The Federal Aviation Administration).

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Understanding Foreign Object Damage through Spatially Resolved Residual Stress Measurements: *B. L. Boyce*[1]; J. O. Peters[1]; J. M. McNaney[1]; R. O. Ritchie[1]; [1]University of California at Berkeley/LBNL, Mats. Sci. Div., MS 62-203, 1 Cyclotron Rd., Berkeley, CA 94720 USA

Foreign Object Damage, caused by the ingestion of debris into turbine engines, can significantly degrade the usable lifetime of structural components (most specifically fan blades). The current study uses synchrotron x-ray diffraction to examine the formation of residual stresses by the impacting process and the implication of these residual stresses on crack formation and propagation under cyclic loading. Moreover, the observed residual stress gradients will be used to asses the validity and limitations of numerical models.

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Deformation, Fracture and Fatigue in a High Strength Aluminum Alloy: *A. P. Reynolds*[1]; Kumar Jata[2]; [1]University of South Carolina, Dept. of Mech. Eng., 300 Main St., Columbia, SC 29208 USA; [2]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., WPAFB, OH 45433 USA

Tensile, fracture, and fatigue studies have been conducted on a dispersion strengthened aluminum alloy with sub-micron grains. Full-field, surface strain distributions were measured by digital image correlation (DIC) in selected cases. DIC was used to understand the strain distribution/partitioning in uniaxial tension and in the plastic zone at a crack tip.

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The Compressive Behavior of Ti-6Al-4V/TiC Layered Composites Examined Through Experiments and Modeling: C. L. Briant[1]; C. W. Bull[1]; K. S. Kumar[1]; *A. J. Wagoner Johnson*[1]; [1]Brown University, Div. of Eng., Box D, Providence, RI 02906 USA

The Ti-6Al-4V/TiC composite system is being studied in compression as a function of strain rate. Symmetric, three-layered structures have been successfully fabricated with equal layer thickness by diffusion bonding individual layers. The layers are made of either the monolithic material or a 10%TiC particulate reinforced composite. The layer-interfaces are void-free and the interfacial bond is excellent. Preliminary results show that the engineering stress of the layered structures is bounded by the engineering stress of the individual layers, despite the yield of only the monolithic layer. While the structures show similar hardening behavior as the individual layers, the strength and ductility are much greater than the individual monolithic and reinforced layers, respectively. The interface between layers constrains the softer material from flowing radially, causing the diameter of the cylindrical specimen to vary. Structure failure is postponed by crack deflection near the soft/hard layer interface. The contribution of the interface to the compressive strength of the structures will be modeled by altering the interfacial strength, or interface boundary conditions. Layer thickness and number of layers will also be varied to further improve properties.

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Influence of Heat Treatment on the High Rate Deformation Behavior of a Metal Matrix Composite: *Don Lesuer*[1]; Chol Syn[1]; Mary LeBlanc[1]; [1]Lawrence Livermore National Laboratory, L-342, Livermore, CA 94551 USA

Many potential applications of metal matrix composites involve use of the material under dynamic loads. In this paper we report on the influence of heat treatment on the high rate deformation of the 6090 aluminum alloy reinforced with 25% SiC particulate. Six heat treatments that changed the strength of the matrix alloy were considered a peak age temper, two underage tempers and three overage tempers. High rate testing was done in compression using the split Hopkinson pressure bar technique, and data was obtained at strain rates of 103 s-1 to 104 s-1. The underage and overage tempers produced lower flow stresses than the peak age treatment but all heat treatments produced comparably work hardening rates. The influence of the stress-strain behavior of the matrix on the stress-strain behavior of the composite has been analyzed in terms of

composite-strengthening, continuum-plasticity models. Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48

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Intergranular and Stress Corrosion Cracking Behavior of Al-Li-Cu Alloy AF/C458 After Artificial Aging: D. Mathur[1]; P. I. Gouma[1]; R. G. Buchheit[1]; [1]Ohio State University, Dept. of Mat. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The stress corrosion cracking (SCC) behavior of AF/C458 (Al-2.05Li-2.70Cu-0.6Mg-0.3Zn-0.08Zr) in 1.8 inch plate form was studied as a function of artificial aging at either 150°C or 190°C for times ranging from 2.4 to 36 hours. SCC resistance of the alloy in the short-transverse and long-transverse orientations was evaluated by alternate immersion and constant extension rate testing (CERT) in 3.5% NaCl solutions. The alloy was also evaluated for susceptibility to a pre-exposure embrittlement phenomenon unique to Al-Li alloys. By all measures, the SCC behavior of this alloy is superior to AA 2090 and AA 8090. Resistance is likely derived from a variety of factors including controlled texture, grain size, high toughness, and controlled grain boundary precipitation. The experimental observations of SCC combined with a microstructural characterization by transmission electron microscopy supports the notion that when the alloy is SCC-susceptible, an anodic dissolution-based cracking mechanism involving selective dissolution of Zn-modified T_1 (Al_2(Cu,Zn)Li) precipitated at low and high angle boundaries is operative.

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Localized Corrosion Mechanisms and TEM Characterization of Al-Li-Cu Alloy AF/C458 After Interrupted Quenching from Solutionizing Temperatures: P. I. Gouma[1]; J. E. Kertz[1]; R. G. Buchheit[1]; [1]Ohio State University, Dept. of Mat. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Isothermal time-temperature-localized corrosion mechanism curves were determined for the Al-2.05Li-2.70Cu-0.6Mg-0.3Zn-0.08Zr alloy AF/C458 to understand the effect of slow or delayed quenching on localized corrosion mechanisms. Alloy samples were subject to a series of systematic interrupted quenching experiments conducted at temperatures ranging from 480° to 230°C for times ranging from 10 to 1000 seconds. Samples were then exposed to a oxidizing aqueous chloride environment to induce localized attack. The alloy exhibited pitting, intersubgranular attack (ISGA), or intergranular attack (IGA) depending on the time at temperature. Corrosion behavior was interpreted on the basis of a detailed microstructural evaluation by transmission electron microscopy, which clearly showed that IGA and ISGA were related to the precipitation of Zn-modified T_1 (Al_2(Cu,Zn)Li) at high and low angle boundaries resepctively. In situations where the T_B (Al_7Cu_4Li) phase was present on boundaries instead of T_1, IGA and ISGA susceptibility was comparatively diminished.

Magnesium Technology 2001: Forming

Sponsored by: TMS: Light Metals Division, Magnesium Committee and Reactive Metals Committee; International Magnesium Association; and ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee

Program Organizers: John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA; David Creber, Alcan International, Ltd., Kingston R&D Center, Kingston, Ontario K7L 5L9 Canada; Russell H. Jones, Battelle Pacific Northwest National Laboratory, Richland, WA 99352 USA; Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Ramaswami Neelameggham, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Eric A. Nyberg, Pacific Northwest National Laboratory, Materials Processing Group, Richland, WA 99352 USA; Mihriban O. Pekguleryz, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Kevin Watson, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H7R 1G5 Canada

Wednesday PM — Room: 203-205
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA

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Alloy Design and Microstructure Evolution of Thixoformable Mg-Ni Alloys: *Shaekwang Kim*[1]; Won Ha[1]; Chulhong Bae[1]; Youngjig Kim[1]; [1]Sung Kyun Kwan University, Sch. of Metall. and Mat. Eng., 300 Chunchun-dong Jangan-gu, Suwon, Gyunggi-do 440-746 Korea

The importance of processing of magnesium alloys in semisolid state is increasing rapidly. The understanding of the process is concerned primarily with the microstructure of semisolid alloys generated during either partial solidification or partial remelting, with their Rheological behaviors and with the modeling of this behavior for the purpose of numerical simulation of thixoforming processing. However, very little work has been done in the development of alloy composition tailored to thixoforming processing. The aim of this research is to develop thixoformable Mg-Ni alloys and to evaluate the microstructure evolution of them during partial remelting as functions of Ni content and isothermal holding temperatures. The characteristics of thixoformable Mg-Ni alloys are based on the fact that fraction and composition of liquid and fine solid globules without the network among solid globules can be simply controlled by normal solidification and reheating procedures.

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Microstructure Studies after Different Solution Treatments of a Thixo-Cast AZ91: *Enrico Evangelista*[1]; Marcello Cabibbo[1]; Emanuela Cerri[2]; [1]University of Ancona, Dept. of Mechanics, Via Brecce Bianche, Ancona I-60131 Italy; [2]University of Lecce, Dept. of "Ingegneria dell'Innovazione", Via Arnesano, Lecce I-73100 Italy

The microstructure of a thixoformed AZ91 Mg-Al-Zn consists of large alpha globules separated by quasi-eutectic phase (alpha+betha). Solution heat treatments at 395°C, 415°C and 435°C for different exposition times were carried out. Light Microscopy (LM) investigations showed that the alpha-Mg based areas are developed into individual grains, while the betha phase (Mg17Al12) particles are present only in the eutectic area. Transmission Electron Microscopy (TEM) revealed small Mg-rich particles inside the eutectic (divorced eutectic). The solution treatment induced the almost complete transformation of the original microstructure, produced by thixo-forming, in a more conventional structure of equiaxed grains respectively in 1hr, 3hrs, 24hrs for 435°C, 415°C and 395°C. From this time the eutectic is localised in the grain boundaries and the alpha-Mg based grains enriches in aluminium and zinc content

due, basically, to the simultaneous dissolution of the alpha-phase and reduction in eutectic quantity.

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Superplasticity in Coarse Grained Class I Solid Solution of HCP Mg-Al: *Tsutomu Ito*[1]; Junya Saeki[1]; Masahisa Otsuka[1]; [1]Shibaura Institute of Technology, Dept. of Mats. Sci. and Eng., 3-9-14 Shibaura, Minato-ku, Tokyo 1088548 Japan

Hexagonal close packed magnesium alloys have recently been expected as structural materials for transportation systems due to their excellent specific stiffness and specific strength. However, a limited number of their slip systems due to an anisotropy in hcp structure make them less ductile at room temperature than face centered cubic materials such as aluminum or copper alloys. On the other hand, in the case of elevated temperature, magnesium alloy should potentially exhibit high ductility because both pyramidal slip and prism one are activated in addition to basal glide. We have been investigated high temperature deformation behavior on three kinds of coarse-grained Mg-Al class I solid solution. The stress exponent was 3 and the activation energy for deformation was close to that for chemical interdiffusion. The characteristics are similar to those of class I solid solutions. The alloys showed enhanced ductility over 250% at 723K and initial strain rate around 1 x 10-4s-1. The result suggest that the alloys could have exhibited class I superplasticity. The mechanism of deformation and fracture will be discussed on the basis of microstructural observation.

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Production of a Superplastic Microstructure in Magnesium Alloys and their Application: *Claus Christian Kedenburg*[1]; Antonia Schram[1]; Ulrich Draugelates[1]; [1]Institute of Welding ISAF, Magnesium Dept., Agricolastrasse 2, 38678 Clausthal, Germany

Despite the increasing interest of the industry in lightweight materials during the last years an intensive industrial use of magnesium alloys due to the restricted cold-work-ability caused by its hexagonal lattice is still very limited. Considering this limitation a solution is provided by the process of superplastic forming of magnesium based alloys which, in contrast to other types of materials, is neither metalurgically developed nor process optimized. Presuppositions for the superplastic behavior of magnesium alloys are discussed in the introductory part of this presentation. Methods to quantify the superplastic behavior and the importance of the m-values derived thereof are explained in the following. Since a major precondition for superplastic forming is a very fine grain structure, various possibilities of grain-fining-procedures of magnesium-alloys are described with the help of parameter-lists and pictures of grain-structures. Finally, the results of the investigations (max. fracture elongation of 1050% at a constant strain rate of 1,6×10-4 s-1) are graphically displayed and possibilities of utilizing the superplastic characteristics, e.g. easy production of AM20, ZRE1 and QE22 magnesium alloy sheets with superplastic characteristics, are shown.

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Creep and Hot Working of Mg Alloys: *Hugh J. McQueen*[1]; W. Blum[2]; [1]Concordia University, Mech. Eng. Dept., 1455 Maisonneuve W., Montreal, Quebec H3G 1M8 Canada; [2]University of Erlangen-Nurenberg, Matls. Sci. Dept., Erlangen D91058 Germany

Die cast Mg alloy AZ91 was subjected to creep (<10-4s-1) in both compression and tension and to hot working (10-2-10 s-1) in torsion. Constitutive analysis showed that the dependencies of maximum deformation resistance (steady state creep rate at constant stress or steady state flow stress at constant strain rate) on temperature T were consistent across the entire range. The creep tests were concerned with the total strain within the operating life time and were compared to behavior of AS21. The torsion tests were concerned with the dependence of fracture strain on T and and were compared to the behavior of AZ31 and ZK60. Twinning took place at low strains to reorient grains not suited for slip and occurred much more profusely in the hot working. As a result of dynamic recovery, subgrains developed primarily near the grain boundaries, being much larger in creep than those in hot working consistent with the stresses. In the creep specimens, the particles underwent considerable change over the long periods. In contrast, during hot working the specimens underwent dynamic recrystallization that gave rise to considerably improved ductility and reduced strength at high T.

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Forging of Magnesium Using a Squeeze Cast Pre-Form: Gabriela Tausig[1]; *Nigel Jeffrie Ricketts*[2]; Stephen Ronald Peck[2]; [1]National Forge Operations, 465 Somerville Rd., West Footscray, Victoria 3012 Australia; [2]CSIRO, Manufacturing Science and Technology, Technology Court, Pullenvale, Queensland, Australia

The supply of magnesium billet for forging stock is limited to only a few suppliers. It is difficult to find and is expensive. It should ideally be DC cast and grain refined, but a number of suppliers provide billet in a cast form with a large grain size. An alternative to DC cast billet is a cast pre-form. In order to minimise shrinkage porosity, squeeze casting was used to provide porosity-free preforms in AZ31 magnesium alloy for forging trials. These trials were conducted at National Forge Operations and showed that the preforms via the squeeze casting route can produce good quality forgings in a single forging step. Forging of an automotive clutch hub at National Forge was able to be conducted more easily than the literature would suggest. A comparison of the modelling work and the results obtained is presented. Forging via a cast pre-form would appear to offer promise in the provision of forging stock for single-stage forging.

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Assessment of Equal Channel Angular Extrusion Processing of Magnesium Alloys: *S. R. Agnew*[1]; L. Chen[2]; Y. Lu[2]; M. Stoica[2]; D. Fielden[2]; P. K. Liaw[2]; [1]Oak Ridge National Lab, Oak Ridge, TN 37831 USA; [2]University of Tennessee, Knoxville, TN 37996 USA

Equal channel angular extrusion (ECAE) offers the potential to introduce very large strains into a metal workpiece without changing its cross-section. Hence, it could be an attractive technique for developing a fine-grained forging stock with good forming characteristics. In general, the mechanical properties of hexagonal close packed (hcp) metals respond even more positively to grain refinement than cubic metals. Furthermore, because ECAE is significantly slower than straight extrusion, die chilling is a strong concern for materials that must be processed hot. Magnesium alloys are ideal candidates to benefit from the ECAE technique, because they are hcp and their low melting point allows them to be processed isothermally at temperatures of 300ºC and below. An assessment of the technique has been made with the alloys AZ31B and ZK60. The mechanical properties of AZ31B appear largely unaffected by the process, however, the ZK60 alloy responds very favorably. Following ECAE, the ductility of ZK60 has been shown to increase by 2 to 3 times over the entire temperature range investigated (24-450deg C) with no significant change in strength. Possible explanations for the property enhancements and the development of crystallographic textures will be discussed.

5:05 PM

AM70-Magnesium Processed by Semi-Solid Casting: *Dierk Hartmann*[1]; Wolfram Wagener[1]; [1]EFU Gesellschaft für Ur-/Umformtechnik mbH, Simmerath (D)

The process of semi-solid casting (SSC) is defined by forming a metal with thixotropic behavior in a temperature range between solidus and liquidus. The thixotropic behavior is dependent on the type of alloy and on the value of shear stress that acts during the forming process. The viscosity of the material is reduced and the forces to fill the tool are relatively low and comparable to forces during a casting process. Combining the positive characteristic of forging (advanced microstructure) with the characteristics of casting (complexity), this alternative manufacturing process allows the production of parts with low porosity, for example, in a near-net-shape design. The reason semi-solid casting of magnesium is relatively unknown is the lack of a "standard" feedstock material and sufficient material property data. In this paper the use of hot extruded magnesium for SSC is described for the production of demonstrator components. Tensile test specimens were machined to achieve more information about mechanical properties of SSC-magnesium.

Materials Issues in Microelectronics - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Michael R. Notis, Lehigh University, Department of Materials Science, Bethlehem, PA 18015 USA; Sailesh Merchant, Lucent Technologies, Orlando, FL 32819 USA; Martin W. Weiser, Honeywell Electronic Materials, Spokane, WA 99216 USA; Jin Yu, KAIST, Department of Materials Science, Seoul, Korea

Wednesday PM
February 14, 2001

Room: 226
Location: Ernest N. Morial Convention Center

Session Chairs: Michael R. Notis, Lehigh University, Dept. of Mats. Sci. & Eng., Bethlehem, PA 18015 USA; Martin W. Weiser, Honeywell Electronic Materials, Spokane, WA 99216 USA

2:00 PM

Reactive Formation of a Solid Phase: *Francois M. D'heurle*[1]; [1]IBM Corporation, P.O. Box 218, Yorktown Heights, NY 10598-0218 USA

Fundamental aspects of the reaction of a solid with another solid, a liquid, or a gas in order to form a new solid layer containing one or several separate phases will be considered. For solid-solid reactions the usual linear or parabolic kinetics will be reviewed. The specific character of linear kinetics most prominent in the reaction of a solid with molecular gases will be described. In solid-solid reactions, e.g., in the formation of thin film silicides, one can often neglect the formation of terminal solid solutions in the two sources; this is not possible in solid-liquid reactions because (a) the solubility limits tend to be high, and (b) rapid diffusion in the liquid dominates the interaction. Althought the approach will be formal, practical illustrations pertinent to silicide formation, oxidation, and soldering will be highlighted.

2:30 PM Invited

Reactions at Materials Interfaces in Seimconductors: *Sailesh M. Merchant*[1]; [1]Lucent Technologies, Bell Laboratories, 9333 South John Young Parkway, Orlando, FL 32819 USA

Materials interfaces of semiconductor devices play an important role in determining device performance. An understanding of metallurgical and materials interactions at these interfaces is key to determining their reliability. This paper reviews the role of the metallurgist/materials scientist, who invokes simple principles of phase constitution, interdiffusion, phase transformations, mechanical and chemical interface stability, to understand and minimize these reactions. Examples are provided from various steps of semiconductor device processing where these materials phenomena are commonly observed. Reactions and instabilities at materials interfaces, such as during silicidation, interconnect formation, packaging and assembly of semiconductor devices, are reviewed.

3:00 PM

Silicidation Reactions of Ti/Ni Bilayer on Chemically Oxidized Si Surface: *Tan Wee Leng*[1]; Pey Kin Leong[2]; Chooi Simon[2]; Ye Jian-Hui[3]; Mangelinck Domanique[3]; Osipowicz Thomas[4]; [1]National University of Singapore, Dept. of Comp. and Elect. Eng., 10 Kent Ridge 117576 Singapore; [2]Chartered Semiconductor Manufacturing Ltd., 60 Woodlands Industrial Park D, St. 2, Singapore 738406 Singapore; [3]Institute of Material Research and Engineering, 4 Engineering Dr. 3, Singapore 117576 Singapore; [4]National University of Singapore, Dept. of Phys., 4 Engineering Dr. 3, Singapore 117576 Singapore

Interfacial reaction in a Ti/Ni/SiOx/Si system was studied in detail. The chemical oxide was prepared by treating a (100)Si surface chemically with NH4OH:H2O2:H2O solution which forms ~ 12Å of SiOx. A 300Å of Ni layer was then sputtered on the chemically prepared Si substrate, followed by a 50Å or 100Å sputter Ti deposition. Subsequently, the samples were annealed in RTP for temperature ranging from 500°C to 800°C. Material and chemical characterization using XRD, RBS and XPS depth profiling were carried out to identify the various phases of Ni silicide and to study the inter-diffusion of the different elements before and after annealing. It was found that with a 50Å Ti cap, no reaction occurs between Ni and Si up to a temperature of 750°C. In this temperature range, inter-diffusion between Ni and Si takes place with increasing annealing temperature but no reaction occurs. However, when a 100Å Ti cap was used, reaction between Ni and Si starts at 600°C, forming NiSi. At 750°C, our XRD results show the presence of both NiSi and NiSi2 phases. At 800°C and above, full conversion to NiSi2 took place. The possible mechanism responsible for the Ni-salicidation is proposed.

3:20 PM

Piezoelectric Actuation of Crack Growth Along Polymer-Metal Interface: Tianbao Du[1]; M. Zhang[2]; S. Seghi[1]; K. J. Hsia[2]; J. Economy[1]; *J. K. Shang*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mats. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA; [2]University of Illinois at Urbana-Champaign, Dept. of Theor. and Appl. Mech., Urbana, IL 61801 USA

A new experimental technique for determining mechanical properties of the polymer-metal interface was developed by replacing the conventional mechanical testing machine with a piezoelectric actuator. Crack growth along an adhesive bond was found to depend on the magnitude of the applied electric field. The driving force for the crack growth was computed from the finite element analysis as a function of crack length, applied field, material properties and specimen geometry. Kinetics of the crack growth was correlated with the piezoelectric driving force. The resulting crack-growth behavior was compared with the results from the conventional mechanical testing technique. Work supported by the National Science Foundation under grant NSF CMS 98-72306.

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Estimations of the Interfacial Fracture Energy of a Cu/Cr/PI System by the T-Peel Test: *J. Y. Song*[1]; Jin Yu[1]; [1]KAIST, Center for Electronic Packaging Materials, 373-1 Kusong-dong Yusong-gu, Taejon, Korea

T-peel tests were conducted to determine the metal/polymer(m/p) adhesion strength of a Cu/Cr/PI structure with interface precracks between Cr and PI used in electronic packaging. Effects of the biased RF plasma pretreatment and the metal layer thickness on the peel strength (P) were investigated, and the energy dissipated by plastic bending (Ψ) and the interfacial fracture energy (Γ) are estimated. During the steady state peeling, the peel angle (ϕ) and the maximum curvature at peeled film bases were directly measured by using an optical camera, from which Ψ can be deduced from the elastic/plastic analysis of Kim and Aravas. P, Ψ and ϕ values vary with the plasma density (ρ) and the metal layer thickness. The interfacial fracture energy between Cr and PI which increases with ρ but is independent of m/p layer thickness were deduced. Later, Γ values were compared with the theoretical analysis by Moidu et. al. and Wei and Hutchinson.

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Shear Strength of a Cu-Ta Interface by Molecular Dynamics: *Pekka Heino*[1]; [1]Tampere University of Technology, Electronics, P.O. Box 692, Tampere FIN 33101 Finland

During last few years, the electrical and mechanical properties of copper have received a lot of interest in the electronics community, mainly because of its low electrical resistance. Recently we have studied mechanical properties of nanoscale pure copper connections \1\. However, a barrier layer between copper and the rest of the system is needed to prevent diffusion. In such disordered systems the interfaces are often the weakest spots. Thus, in this work we study the interface of copper and tantalum, which is often used as a barrier metal, because of positive heat of formation. We use molecular dynamics with embedded-atom potentials as means. We study the energy and shear strength of several low-index interfaces. A strong positive correlation is found indicating that low-energy interfaces are weak. In addition, some interfaces are formed by de-

positing Cu on different Ta surfaces and their microstructure is analyzed. 1. P. Heino and E. Ristolainen: Mechanical properties of nanoscale copper under shear, Microelectronics Reliability 40 (2000) 435-441

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Void Formation of Wire-Bond During High Temperature Aging: *Ker-Chang Hsieh*[1]; Hen-So Chang[1]; Theo Martens[2]; Albert Yang[2]; [1]National Sun Yat-sen University, Instit. of Matls. Sci. and Eng., Kaohsiung, Taiwan; [2]Philips Electronic Bldg. Elements Industries, Ltd., Tech. Dev. Div., 10, Chin 5th Rd. N.E.P.Z., P.O. Box 35-48, Kaohsiung, Taiwan

Voids are formed along with the growth of gold/aluminum intermetallic phases at wire-bond interfaces during high temperature aging. This phenomenon is known to degrade the bond.The purpose of this study is to clarify the factors that influence bondability and bond degradation, and to understand the mechanism of void formation. Samples were prepared under various wire-bonding conditions, Al-pad thickness, aging temperatures and aging times. The treated samples were examined, and the ball bond cross section microstructure is reported. Microstructure analysis was perfomed on a JEOL Superprobe JXA-8900R.

5:00 PM Invited

Materials and Process Challenges for Packaging Higher Density/Higher Frequency Microelectronics: *Mark Thomas McCormack*[1]; [1]Fujitsu Computer Packaging Technologies, 3811 Zanker Rd., San Jose, CA 95134 USA

Many issues are apparent upon review of the requirements put forth by the electronics industry's microelectronics packaging roadmaps. Among the most challenging are those that will require new or modified fabrication processes and materials sets. After discussing many of these issues in broad terms, selected examples that concentrate on solving higher density/higher frequency in package substrates will be discussed.

Materials Processing Fundamentals VI

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Process Fundamentals Committee, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: P. N. Anyalebechi, ALCOA, Ingot & Solidification Platform, Alcoa Center, PA 15069-0001 USA; A. Powell, MIT

Wednesday PM Room: 218
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Ray Y. Lin, University of Cincinnati, Dept. of Mat. Sci. & Met. Eng., Cincinnati, OH 45221-0012 USA

2:00 PM

Optimization of Oxide Powder Extrusion Paste for Forming Alloy Honeycomb Precursors: *Joe K. Cochran*[1]; Kevin M. Hurysz[1]; Raymond H. Oh[1]; Wesley D. Seay[1]; [1]Georgia Institute of Technology, Mats. Sci. and Eng., 771 Ferst Dr., Atlanta, GA 30332-1320 USA

The extrusion of oxide powders pastes through a thin-wall honeycomb die results in a low density metallic structure following reduction. These pastes are a combination of two phases: a solid phase composed of the particular oxide or oxide mixture carried by a fluid solution of water, binder, and lubricant. The key to forming high quality, defect free extrudate lies in the optimization of paste properties and is contingent on solids loading and fluid-phase rheology. To extrude efficiently, the fluid must yield to the paste enough compliance to flow through the die, yet provide a high enough viscosity or yield stress at low shear rates to avoid deformation following extrusion. Measurements of viscosity, yield stress, and wall shear stress are used to characterize water-lubricant and water-binder solutions. These data are used to optimize the water-binder-lubricant solution and facilitate the extrusion of articles having complex geometry. This investigation will consider the paste compositions necessary to form high thermal conductivity (copper), structural (maraging steel), and elevated temperature (Inconel 617) alloys.

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Sulfidation of Chalcopyrite with Gaseous Sulfur: *Rafael Padilla*[1]; Marcial Torres[1]; Maria Cristina Ruiz[1]; [1]University of Concepcion, Dept. of Metall. Eng., Edmundo Larenas 270, Concepcion, Chile

The reaction of chalcopyrite with gaseous sulfur has been investigated in the range 350-450°C in order to transform this mineral into a more acid soluble copper sulfide and insoluble iron sulfide. The results of thermodynamic analysis of this system as well as the X-ray diffraction analysis of the reaction products showed that the in the temperature range 350-450°C the sulfidation reaction proceeded without formation of intermediate copper-iron sulfides according to: CuFeS2+1/2S2(g) = CuS+FeS2 The effects of temperature and time on the conversion of chalcopyrite were studied. Conversion of chalcopyrite reached about 80% very rapidly with increase in temperature up to about 400°C. The recovery of copper from the sulfidized samples was determined by leaching in oxygenated 1.2M H2SO4, 2M NaCl solution. Copper recoveries over 90 % were obtained from samples sulfidized for 30 min at temperatures lower than 400°C.

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Metallic Iron Reactions in Aqueous Systems: *Batric Pesic*[1]; Victor C. Storhok[1]; [1]University of Idaho, Coll. of Mines, McClure Hall, Moscow, ID 83844-3024 USA

Despite its ubiquitous nature and its role as of the most important metals to the mankind the dissolutions reactions of iron in aqueous solutions, surprisingly, have not been studied. There is an enormous body of literature on corrosion of iron in aqueous systems but all the reported studies were electrochemical in nature, i.e. corrosion studies. This paper will discuss the reactions of iron with water as a function of pH, temperature, surface geometry, size, application of external potentials, and finally the possible role bacterium, Thiobacillus ferrooxidans. Fundamental knowledge of chemical reactions of iron with water and water constituents is of importance to understanding the processes of corrosion of iron and its alloys, and also of importance to the development of hydrometallurgical processes involving iron in either solid or soluble form.

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Magnetite Stratification During Slag Reduction: *Gabriel Riveros*[1]; *Andrzej Warczok*[1]; [1]Universidad de Chile, Departamento de Minas, Av. Tupper 2069, Santiago, Casilla 2777 Chile

Smelting of copper concentrate into white metal produces highly oxidized slag containing from 15 to 25% of magnetite. Copper recovery from the slag requires effective magnetite reduction and phase separation. Slag reduction and cleaning in an electric furnace is commonly used. Results of slag reduction with graphite electrodes in a crucible simulated electric furnace showed strong tendency for magnetite stratification and formation of related gradient of copper content along the slag height. Microscopic and analytical examination of slag samples allowed for determination of various parameters, such as electrode immersion, current intensity and reverts addition, on distribution of magnetite content. Analysis of mechanisms of magnetite stratification in an electric furnace pointed out the role of the immersion of electrodes, current density, reverts and coke addition. Possibilities of formation of dead zones and built-up on the furnace hearth have been discussed.

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In-Situ Monitoring of the Sintering Process with Non-Contact Electromagnetic Acoustic Transducers: *James C. Foley*[1]; David K. Rehbein[1]; Daniel J. Barnard[1]; [1]Ames Laboratory, Metallurgy and Ceramics Program, 122 Metals Development, Ames, IA 50011 USA

In-situ characterizations of green state part density and sintering state have long been desired in the powder metal community. Recent advances in non-contact electromagnetic acoustic transducer (EMAT) technology have enabled in-situ monitoring of acoustic amplitude

and velocity as sintering proceeds. Samples were made from elemental powders of Al (99.99%), Al (99.7%), Ag, (99.99%), Cu (99.99%) and Fe (99.9%). The powders were pressed in a uniaxial die and examined with acoustic waves for changes in velocity and amplitude during sintering. The changes in acoustic properties were correlated with sample microstructures and mechanical properties. Evolution of a series of reverberating echoes during sintering is shown to provide information on the state of sintering, changes in sintering kinetics as well as having the potential for detection of interior flaws. This work is funded by a laboratory directed research and development grant and by DOE-BES under contract no.W-7405-Eng-82.

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A Model for Coupled Laminar Fluid Flow and Electrochemical Reactions in Electric Field Smelting and Refining of Steel: *David Michael Dussault*[1]; Adam Powell[2]; [1]MIT, Mats. Sci., 77 Massachusetts Ave., Rm 4-033, Cambridge, MA 02139 USA; [2]MIT, Mats. Sci. and Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139 USA

A model for coupled laminar fluid flow and electrochemical reactions is developed using the Navier-Stokes equations and the phase field method. Application to the electric field enhanced refining of steel is discussed. Because the process is limited by ferrous ion transport to the cathodic slag-metal interface, the Mullins-Sekerka instability gives rise to liquid iron fingers protruding into the slag. Two-dimensional numerical results are given which show that the model captures this instability.

Modeling of High Temperature Alloys: Alloy Modelling

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Shailesh Patel, Special Metals, Huntington, WV 25705-1771 USA; Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA

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February 14, 2001 Location: Ernest N. Morial Convention Center

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

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Materials Properties Modelling for Ni-based Superalloys: *Nigel Saunders*[1]; Xiuqing Li[1]; [1]Thermotech, Ltd., Surrey Technology Centre, The Surrey Research Park, Guildford, Surrey GU2 7YG UK

Thermodynamic modelling has reached a status where predictions for the phases present in Ni-based superalloys can be made to a high degree of accuracy [1]. Although pertaining to equilibrium such calculations have already had significant application in industrial practice [2]. However, it is clear that the long term goal of such modelling should be to provide a tool that will predict more general materials properties, for example long term stability with regard to TCP phase (sigma, mu...) formation, more general TTT diagrams for NiFe-based superalloys, mechanical properties, thermo-physical and physical properties, etc.. The present paper will report on new work to achieve this goal, in particular with respect to work on TTT diagrams and preliminary results on mechanical properties. References [1] N. Saunders, Superalloys 1996 eds. R. D. Kissinger et al. (Warrendale, PA: TMS, 1996), 101 [2] N. Saunders, M. Fahrmann and C. J. Small, to be presented at Superalloys 2000, Seven Springs, PA, Sept.21-25 2000

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Modeling of the Partitioning and Phase Transformation Temperatures of a As-Cast a Third Generation Single Crystal Ni-base Superalloy: *Gerhard E. Fuchs*[1]; Brett A. Boutwell[2]; [1]University of Florida, Mats. Sci. & Eng. Dept., P.O. Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA; [2]AEA Technology, Mats. & Chem. Proc. Assess., 241 Curry Hollow Rd., Pittsburgh, PA 15236-4696 USA

Cast single crystal Ni-base superalloys exhibit severe solidification segregation that must be subsequently removed by solution heat treatment. In order to understand how some of the elements in these alloys effect the solidification partitioning, the as-cast microstructure of the third generation single crystal Ni-base superalloy, CMSX-10 was examined. In addition, the solidification partitioning was calculated using Thermo-Calc. The solidus, liquidus and gamma-prime solvus temperatures were also calculated for the compositions of the base alloy, and the determined compositions of the the dendrite cores and the interdendritic regions. All of the calculated values were compared to the experimentally determined values. The results of this study and areas for future work will be discussed.

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Examining the Effects of Elastic Stress in Ostwald Ripening Through Numerical Simulation: *Katsuyo Thornton*[1]; Norio Akaiwa[2]; P. W. Voorhees[3]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., 77 Massachusetts Ave., 13-5130, Cambridge, MA 02139 USA; [2]National Research Institute for Metals, Tsukuba, Japan; [3]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

We examine the effects of elastic stress on the kinetics of coarsening in an elastically homogeneous, anisotropic solid. Our computational model simulates Ostwald ripening in a solid-solid system efficiently and accurately by taking advantage of powerful numerical methods such as the boundary integral method with the fast multipole method. We study large systems consisting of many thousands of particles to produce quantitative, statistically meaningful measures of the temporal evolution of the microstructure. Our numerical simulation indicates that the power-law exponent for the average particle size remains 1/3, while the rate constant depends on the elastic stress. We provide insight into the fundamental phenomena underlying the processes that govern the evolution of the microstructures in elastically stressed solids through both theory and the simulation.

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Nucleation of Gamma' and Gamma" in INCONEL 706 Utilizing Computational Thermodynamics and Diffusion Kinetics: *Brett A. Boutwell*[1]; Raymond G. Thompson[2]; [1]UES Software, Inc., 241 Curry Hollow Rd., Pittsburgh, PA 15025 USA; [2]University of Alabama at Birmingham, Dept. of Mats. & Mech. Eng., 1150 10th Ave. South, Birmingham, AL 35294-4461 USA

The nucleation and subsequent growth of gamma' and gamma" precipitates in INCONEL alloy 706 has a significant impact on the mechanical properties, and therefore the performance, of the alloy. The growth of such precipitates can be modeled using various models and numerical methods, such as those incorporated in the software package DICTRA. Nucleation, however, is a bit more difficult process to model given the large number of factors that can influence the alloy behavior. A model for predicting nucleation kinetics of coherent, homogeneous precipitates using data from computational thermodynamic and diffusion kinetic data was developed. The thermodynamic and kinetic values needed for the nucleation model were obtained from Thermo-Calc and DICTRA. A database of atomic mobilities was created to allow DICTRA to model the multicomponent diffusivities of alloy 706. The nucleation model was then used to predict the incubation time of gamma' and gamma" precipitation in INCONEL alloy 706 at several temperatures. The results of the model calculations were compared to T-T-T and T-T-H data for the alloy.

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Application of Computational Thermodynamics to Model Stray Grain Formation: *John M. Vitek*[1]; Stan A. David[1]; S. S. Babu[1]; [1]Oak Ridge National Laboratories, Oak Ridge, TN 37831-6096 USA

The paper will examine stray crystal formation in single crystal material. Extensive use of computational thermodynamics will be made to quantitatively identify some of the factors that influence the extent of stray crystals.

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Capitalizing on Computational Tools in the Development of a New Low-Cost Diesel Exhaust Valve Alloy: *Michael Gustav Fahrmann*[1]; Gaylord D. Smith[1]; [1]Special Metals Corporation, Huntington Alloys/Technology, 3200 Riverside Dr., Huntington, WV 25705 USA

Recently, computational tools such as Thermo-Calc have been made available to industry to predict phase equilibria in multi-component alloy systems. These computer codes, in conjunction with validated thermo-chemical databases, account to a large degree for the complexity of commercial alloys. An example is presented as to how this tool has actually been used to expedite alloy development for new low-cost diesel exhaust valve material. The current predictive capabilities of Thermo-Calc in conjunction with a commercial Ni-database are also demonstrated in this context.

4:50 PM

Calculation and Verification of Solidification Diagrams in Superalloys: *Wanhong Yang*[1]; Keh-Minn Chang[1]; Wei Chen[2]; Sarwan K. Mannan[3]; Shailesh J Patel[3]; [1]West Virginia University, Morgantown, WV 26506-6106 USA; [2]General Electric, Power Systems, Schnectady, NY USA; [3]Special Metals, 3200 Riverside Dr., Huntington, WV 25705 USA

Solidification is the most important process that determines the segregation and structure of a material. Niobium containing nickel base superalloys were studied using differential thermal analysis, interrupted quenching of solidifying metals and scanning electron microscopy/electron dispersive spectrometry. Liquidus temperature, elemental partitioning coefficient and solid fraction formation in the mushy zone was measured. The solidification was also modeled using the commercial software package Thermo-Calc and a Ni-database, which has been successfully used for solid state phase calculation of superalloys. Comparison of the experimental results with calculation generally yielded good agreement. The versatility of the thermodynamic calculation was also proved. However, large scattering in the liquidus temperature prediction and deviation from measured partitioning coefficient were observed.

Properties of Nanocrystalline Materials: Magnetic Properties

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Jt. Mechanical Behavior of Materials, Chemistry & Physics of Materials Committee

Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Horst W. Hahn, Techische Hchschule Damstadt, Darmstadt D-64287 Germany; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552 USA

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February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Bhakta B. Rath, NRL, Washington, DC USA; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552 USA

2:00 PM Invited

Observation of Hybrid Domain Walls in Exchange-Coupled Ferromagnet/Antiferromagnet Bilayers Using the Magneto-Optic Indicator Film (MOIF) Technique: *R. D. Shull*[1]; A. J. Shapiro[1]; V. S. Gornakov[2]; V. I. Nikitenko[2]; N. Goekemeijer[3]; C. L. Chien[3]; [1]National Institute of Standards and Technology, Mats. Sci. and Eng. Laboratory, 100 Bureau Dr., MS8552, Gaithersburg, MD 20899-8552; [2]Institute of Solid State Physics, RAS, Chernogolovka 142432 Russia; [3]The Johns Hopkins University, Dept. of Phys. and Astron., Baltimore, MD 21218 USA

Microscopic domain processes have been observed at 300K in an exchange-coupled ferromagnet FM)/antiferromagnet (AF) bilayer of $Ni_{81}Fe_{19}$ (160 Å)/$Fe_{50}Mn_{50}$ (300 Å). The bilayer had been ac demagnetized above the Néel temperature of FeMn and cooled in zero field to 300K. Consequently, hysteresis loop measurements showed two loops shifted to opposite sides of the origin. Domain imaging (via the MOIF technique) revealed alternately directed domains separated by hybrid domain walls, consisting of both FM and AF "parts" extending through the bilayer. The AF domain walls remained intact even after the reversal of the FM magnetization (M); only the FM portion of the hybrid domain wall moved. Upon decrease of the field (H), the magnetization decrease was initiated in the FM adjacent to the AF interface. Also, a new type of asymmetry was observed in the remagnetization process for an off-axis H.

2:30 PM Invited

Synthesis, Processing and Magnetic Properties of Nanostructured Gamma Ni-Fe Alloys: *Jai-Sung Lee*[1]; Yun-Sung Kang[1]; [1]Hanyang University, Dept. of Metall. and Mats. Sci., 1271 Sa-1-dong, Ansan, Kyunggi-do 425-791 Korea

The nano-processing has been known to be a potential and promising way to improve the magnetic properties of the Ni-Fe alloy system, such as higher permeability and lower coercivity. In this paper we report on a new processing route for fabricating nanostructured (ns)gamma-Ni-Fe alloy and on its related magnetic properties. The processing of ns gamma-Ni-Fe alloy in this study, which consists of nano powder synthesis and its consolidation process, has been conducted using a mechano-chemical process (MCP) specially for powder synthesis. Regarding the processing, we focus on two kinetic issues occuring during processing of gamma-Ni-Fe nano alloy; in-situ alloying process and densification process of gamma-Ni-Fe nano powder. Magnetic properties of the ns gamma-Ni-Fe alloy were investigated as a function of grain size and the result was discussed in terms of microstructure and chemical property.

3:00 PM Invited

Magnetic Properties of Co Nanocrystals in CoB Amorphous Matrix: A. González[1]; A. Hernando[1]; [1]Instituto de Magnetismo Aplicado, P.O. Box 155, 28230 Las Rozas (Madrid), Spain

The magnetic properties and crystallization behavior of two amorphous compositions of the Co-B system, $Co_{80}B_{20}$ and $Co_{75}B_{25}$, have been studied. By comparing the results it is found that the excess of Co with respect to the stequiometric Co_3B in the $Co_{80}B_{20}$ sample has a major effect on the properties of the amorphous material and on the crystallization process. Transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM) and differential scanning calorimetry (DSC) have been used to characterize the different crystallization states of $Co_{80}B_{20}$ alloy. This composition undergoes a primary crystallisation which transforms the material into a two phase system composed of Co nanocrystals embedded in a Co-B ferromagnetic amorphous matrix. The role of the cobalt grains on the magnetisation processes and the coercive field is studied for temperature ranging from 5 to 300K. It is found that the hardening effect of the grains diminishes on increasing the temperature. This behavior has been analyzed in terms of the magnetic coupling between grain

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Magnetic Microstructure and Anisotropy in Nanocrystalline Feromagnets Measured by Small-Angle Neutron Scattering: *Joerg Weissmueller*[1]; [1]Institut fuer Nanotechnologie, Forschungszentrum Kalrsruhe, Gabh,Herman-von-Helmholtz-Platz 1, D-76334 Germany and Technische Physik, Universitet des Saarlandes, Saarbrfacken, Germany

Magnetic small-angle neutron scattering (SANS) studies provide the unique opportunity to probe the magnetic microstructure in the bulk of nanocrystalline ferromagnets with a spatial resolution of few nm to a few 100nm, complementing imaging techniques which are sensitive to the materials surface and to larger structures. Recent progress in the SANS data analysis has made it possible to obtain quantitative information on (i) the magnetic microstructure and its variation as a function of the applied magnetic field, H, (ii) the ferromagnetic exchange-stiffness constant, and (iii) the magnitude

and microstructure of the magnetic anisotropy. SANS data for the magnetically soft nanocrystalline transition metals Ni and Co show that the dominant structure of the magnetization are continuous fluctuations of the spin directions about the direction of the applied field, with correlation lengths that vary, as a function of H, from few nm to (at least) tens of nm. By contrast, for the magnetically hard rare earth Tb the magnetization is found to be 91 locked into the basal plane of each crystallite up to applied fields of several Tesla.

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Nanocrystalline Hard Magnetic Materials: *Oliver Gutfleisch*[1]; A. Bollero[1]; A. Kirchner[1]; K. H. Muller[1]; L. Schultz[1]; [1]Institute of Solid State and Materials Research, P.O. Box 270016, Dresden 01171 Germany

Recent developments that occurred in nanocrystalline rare earth-transition metal hard magnets are reviewed and particular emphasis is placed on ongoing research work at IFW Dresden. Principal synthesis methods used include mechanical alloying, melt spinning and hydrogen assisted methods such as reactive milling and the hydrogenation-disproportionation-desorption-recombination (HDDR) process. These processing techniques are applied to NdFeB-, NdFeC-, PrFeB-, SmFe- and SmCo-type systems with the aim to produce high remanence magnets with high coercivity. Concepts of maximizing the energy product in nanostructured magnets by either inducing a texture via anisotropic HDDR or hot deformation or enhancing the remanence via exchange coupling are evaluated. The latter phenomenon is observed in nanocomposite magnets consisting of soft and hard magnetic phases where the magnetic interaction leads to magnetically single demagnetization curves despite a multi-phase microstructure provided grain sizes are below a certain threshold and paramagnetic intergranular phases are absent. This has been realized for example in thermodynamically highly stable Sm_2Co_{17} compounds which have been grain refined via a mechanically induced reversible gas-solid reaction using severe hydrogenation conditions. Finally, highly textured, radially oriented $Nd_2(Fe,Co,Ga)_{14}B$-based ring magnets produced by backward extrusion with improved temperature coefficients of coercivity and an energy density $(BH)_{max}$ of 325kJ/m^3 are described.

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Nanocomposite Magnetic Material: *Pekka Ruuskanen*[1]; M. Karttunen[1]; J. Enqvist[1]; [1]VTT Technical Research Center of Finland, Dept. of Chem. Tech., P.O. Box 1402, Tampere 33101 Finland

A nanocomposite magnetic material was developed using iron nanoparticles with a diameter of less than 80 nm. The iron nanoparticles were mixed with a styrenic block copolymer using a Brabender Plasticorder batch mixer. After mixing, the resulting material was compacted in a hot press at a temperature of 180°C. Because it proved difficult to obtain an adequate packing density with the iron nanopowders in their original form, a procedure for modifying the surface of the nanoparticles was developed to improve wetting by the plastic melt. This procedure made it possible to achieve a packing density of about 40 vol%. The quality factor (Q) and the permeability (i) of the compacted samples were measured at different frequencies using commercially-available ferrite plate and iron particles with an average diameter of approximately 30 ìm as the reference material. It was found that the quality factor Q at higher fre quencies was much higher in the nanocomposite magnetic material than in the reference material. At a frequency of 8.2 MHz the quality factor Q was 175 in the polymer nanocomposite and 400 in the commercial ferrite. When the frequency was increased to 200 MHz, the quality factor Q was 70 in the polymer nanocomposite and 3 in the commercial ferrite. The permeability of the nanocomposite material remained at an almost constant value of 11 as the frequency was increased from 8.2 MHz to 200 MHz while the permeability of the commercial ferrite material fell from 400 to 33 over the same frequency range. The results obtained show that nanotechnology has potential as a way of developing magnetic materials for use in high-frequency applications.

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Structure Peculiarities and Magnetic Properties of Nanocrystalline Thin Films: *Vladimir Grigor'evich Shadrow*[1]; Anatolyi Vasilyevich Boltushkin[1]; Lyudmila Vasil'evna Nemtsevich[1]; [1]Inst. Solid State Physics, Acad. Sci. of Belarus, P. Brovki,17, Minsk, Belarus 220072 Belarus

Growth processes,structure peculiarities and properties of hard and soft magnetic Co and Fe based films have been investigated by means of EM, XRD, AFM and AGFM as well as post deposition treatment effect on the films structure and properties. A mechanism of nanocrystalline structure formation and its influence on the films properties is discussed.Intergranular magnetic interaction and magnetization reversal processes in the above films are investigated through remanence and delta M curves measurements and time dependence measurements.

Sampling, Sensors & Control for High Temperature Metallurgical Processes: Aluminum Reduction Technology - Advanced Control

Sponsored by: Light Metals Division, Extraction & Processing Division, Materials Processing and Manufacturing Division, Aluminum Committee, Pyrometallurgy Committee, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: Adrian Deneys, Praxair, Inc., Tarrytown, NY 10591 USA; Derek Fray, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3Q2 UK; Matt Krane, Purdue University, Department of Materials Engineering, West Lafayette, IN 47907; Markus Reuter, Delft University of Technology, Applied Earth Science, Delft 2628 RX The Netherlands; Fiona Stevens McFadden, University of Auckland, Chemistry and Materials Engineering, Auckland, New Zealand

Wednesday PM — Room: 230
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Fiona Stevens McFadden, University of Auckland, Chem. and Mats. Eng., Auckland, New Zealand; Matthew John M. Krane, Purdue University, Dept. of Mats. Eng., West Lafayette, IN 47907 USA

2:00 PM

Control Electrochemical Cell Dynamics with Electrode Current Measurements: *James R. Barclay*[1]; [1]Universal Dynamics, #100-13700 International Place, Richmond, BC V6V 2X8 Canada

Knowing the electrical current distribution in a multiple-electrode, electrochemical cell, for example, a prebake aluminum reduction pot, provides significant insights into the cell dynamics. Metal pad movements, bath chemistry variations, anode spikes, bubble phenomena all affect the current distribution and efficiency of the cell. This paper describes a robust current sensing system that graphically shows cell dynamics. The current sensors are suitable for harsh reduction cell environments and are easily installed and maintained. The system can display real time data, analyze historical data and graphically replay cell events.

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Digital Processing of Anode Current Signals: An Opportunity for Improved Cell Diagnosis and Control: Graeme C. Barber[2]; *Jeffrey T. Keniry*[4]; Mark P. Taylor[3]; Barry J. Welch[4]; [1]Alumination Consulting Pty, Ltd., 2, Governors Dr., Mt Macedon, Vic 3441 Australia; [2]Consultant, 5, Fullwood Pde, Doncaster East, Vic 3109 Australia; [3]Comalco Aluminium Ltd., 12, Creek St., Brisbane, Qld 4000 Australia; [4]University of Auckland, Dept. of Chem. and Mats. Eng., Auckland, New Zealand

While digital signal processing (DSP) is now commonplace in many industrial applications, it has received surprisingly little attention or application in aluminium smelting. Despite advances in data acquisition and storage, line current and cell voltage remain the only signals that are continuously monitored for control of vital cell functions such as alumina feeding, thermal regulation and magnetic stability. But are we using these signals to their maximum potential for diagnosis and control of the process? A full complement of

anode signals has been studied from industrial cells using high frequency (50Hz) sampling, with subsequent processing in time and frequency domains using the Fast Fourier (FFT) technique. While the imprinting of the metal surface motion is a well-known observation under low frequency sampling, this work shows that the higher frequency signals associated with bubble formation and release from the anodes also provide an imprint for specific process events and cell behaviour. The potential applications of DSP of individual anode and composite cell signals in diagnosis and control are discussed.

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Application of Advanced Process Control to Aluminium Reduction Cells-A Review: *Fiona J. Stevens McFadden*[1]; Geoffrey P Bearne[2]; Paul C. Austin[3]; Barry J. Welch[1]; [1]Auckland University, Chem. and Mats. Eng., Private Bag 92019, Auckland, New Zealand; [2]Comalco Research, P.O. Box 315, Thomastown, Victoria 3074 Australia; [3]University of Auckland, Elect. & Electr. Eng., Private Bag 92019, Auckland, New Zealand

The aluminium electrolysis process is fundamentally unchanged since its advent in the late 1880's. The control of the process has however, developed since then, with the trend being to increased mechanisation and automation. Process computers were implemented in the mid-1960's and currently the process is controlled with a mixture of automated and manual systems. In terms of control algorithms, although there have been refinements in use, the principles of the control strategies have not changed substantially in the last 30 years and control of the process outputs is in general achieved using single-input/single-output control loops. Through the application of advanced process control, which draws on elements from disciplines ranging from control engineering, signal processing, statistics, decision theory and artificial intelligence, performance improvements have been gained in other process industries. For aluminium reduction cells optimal control has been investigated along with artificial intelligence techniques such as fuzzy logic control, expert systems and neural networks for identification, prediction and control. Process simulators (physio-chemically derived dynamic models) have also been developed for the development and tuning of control strategies.

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A Multivariable Control of Aluminum Reduction Cells: *Kevin L. Moore*[1]; Nobuo Urata[2]; [1]Utah State University, CSOIS/ECE, 4160 Old Main Hill, Logan, UT 84322 USA; [2]Kaiser Aluminum and Chemical Corporation, Ctr. for Tech., Prim. Al Bus. Unit, 6177 Sunol Blvd., Pleasanton, CA 94566 USA

This paper considers control of the aluminum reduction process, using a dynamic model developed from the literature and Virtpot, a model developed at Kaiser Aluminum. Analysis shows the process is controllable and observable, but not easily stabilizable using one input, and that short-term changes in measured voltage result primarily from changes in alumina concentration rather than anode-to-cathode distance (ACD). Next, a multivariable control strategy is developed to regulate cell voltage by adjusting feed rate rather than beam movement. We introduce the idea of a feed voltage, obtained by subtracting expected voltage deviations due to ACD changes and beam moves from the filtered voltage. Feed rate is adjusted to compensate for deviations of feed voltage from its target. Simultaneously, beam movements are made to compensate for the difference in expected anode consumption and metal pad rise, based on changes in feed period. Simulations show the effectiveness of the proposed control strategy.

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From Cell Models to a Virtual Potroom: *Laszlo G. Tikasz*[1]; Rung T. Bui[1]; Vincent Villeneuve[1]; Sylvain Doyon[1]; [1]University of Quebec at Chicoutimi, Dept. of Appl. Sci., 555 Univ. Blvd., Chicoutimi, Quebec G7H 2B1 Canada

This paper analyzes the 'virtual cell' concept in which dynamic models of aluminium electrolytic cells are used as training, operation support and research tools in aluminium reduction plants. A systematic approach is proposed to transform a chosen cell model into a virtual cell that exchanges data with its environment like a real cell does. Then, a method is given to clone a virtual cell to form a group of cells or even populate an entire 'virtual potroom'. The virtual cells-like the real ones-are identical by design but individual in performance. Every one of these virtual cells is under automatic control. The controllers, organized into a hierarchical scheme, can be real or virtual. Examples are given for various cell and controller arrangements simulating typical cell operations in a plant.

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Virtual Aluminum Reduction Cell: *V. V. Yurkov*; V. Ch. Mann; T. V. Piskazhova; K. F. Nikandrov

At the TMS Annual Meeting of 2000 the "Model of Process of Electrolysis" was presented. Applying this dynamic mathematical model it was possible to imitate the operation of the industrial cell as the first approximation. After the conference this work was continued. A number of active and passive experiments were conducted. Specially designed diagnostic equipment was used for measuring and recording into a database temperature regimes of the cell different units. At the same time in order to identify the model some calculations on the model were carried out (equations selected and coefficients adjusted). This paper presents the results of the performed work, describes a "virtual cell", created on the basis of the dynamic model and a "virtual control system", which is the mathematical twin of an electrolysis control system operated at KRAZ.

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Development of Fuzzy Expert Control Technique for Aluminum Electrolysis: *Jie Li*[1]; *Fengqi Ding*[1]; Zhong Zou[1]; Minjun Li[1]; *Yexiang Liu*[1]; *Youkang Bian*[2]; Zhiming Wu[2]; *Gang Liu*[2]; [1]Central South University of Technology, Dept. of Metall. Sci. and Eng., Changsha, Hunan 410083 China; [2]Qinghai Aluminum Corporation, Xining, Qinghai 810108 China

In order to upgrade the basic control unit called "cell controller" in process control systems of aluminum electrolysis, a new control algorithm called "Fuzzy Expert Controller (FEC)" was developed. Because all the knowledge and experience that can be collected from the field experts as well as all the information which can be sampled for the analysis and control of cell state are not precise, the FEC was designed as a rule-based system working with rules in which imprecise and precise propositions were mixed freely. Its self-regulation mechanism adjusted the universes of discourse of fuzzy variables according to the change of cell state and the transition of control modes, achieving the object of on-line modifying its operating points and dynamic and static performance. Application results on smelters showed that the control accuracy, robustness and stability were satisfactory, and remarkable effects of production increasing and energy saving were achieved.

Second Global Symposium on Innovations in Materials Process & Manufufacturing: Sheet Materials: Composite Processing

Sponsored by: Materials Processing and Manufacturing Division, Powder Materials Committee, Shaping and Forming Committee, Solidification Committee

Program Organizers: Mahmoud Y. Demeri, Ford Motor Company, Manufacturing Systems Dept., Northville, MI 48167 USA; Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; John Papazian, Northrup Grumman, Bethpage, NY 11714 USA; Klaus Siegert, University of Stuttgart, Institute for Metal Forming Technology, Stuttgart D-70174 Germany

Wednesday PM, February 14, 2001 — Room: 228, Location: Ernest N. Morial Convention Center

Session Chairs: Patrick Blanchard, Ford Motor Company, Ford Research Laboratory, P.O. Box 2053, MD3135, SRL, Dearborn, MI 48121-2053 USA; Carl Johnson, Ford Motor Company, Ford Research Laboratory, Dearborn, MI 48121-2053 USA

2:00 PM Invited

The Future of Thermoplastic Composites in Automotive Applications: *Carl Johnson*[1]; [1]Ford Motor Company, Ford Research Laboratory, P.O. Box 2053, MD3135, SRL, Dearborn, MI 48121-2053 USA

In recent years the automotive industry has expressed an interest in stamped thermoplastic composites as these materials can be formed into highly complex cosmetic and semi structural components. The fundamental material compositions take advantage of continuous fibers to improve component performance when compared to conventional injection or compression molded thermoplastics. The improvement in mechanical properties, in turn, provides the opportunity to design a new class of applications utilizing thin wall lightweight structures. Considering the distinct advantages of continuous fiber stamped thermoplastics, implementation of these materials within the auto industry has been limited. This is due, in part, to the absence of robust processing technology and a requirement for engineering design methods. This paper examines the state of progress in implementing this technology by considering a number of current applications. A discussion of the benefits and limitations of material configurations and processing characteristics is presented in the context of competing technologies. Finally, a vision of future research and development activities required for full implementation of this technology is discussed.

2:30 PM

Forming of Engineered Prepregs and Reinforced Thermoplastics: *Michael John Clifford*[1]; Andrew C. Long[1]; Patrick de Luca[2]; [1]The University of Nottingham, Sch. of Mech., Matls. Manufact. Eng. and Mgmt., University Park, Nottingham NG7 2RD UK; [2]ESI International, 20 Rue Saarinnen-SILIC 270, Rungis, Cedex F-94578 France

Composite materials, such as engineered prepregs and reinforced thermoplastics, have found widespread use in the aerospace industry. In addition, the recent development of low-cost materials suitable for medium/high volume production (e.g. commingled glass/polypropylene fabrics) has attracted much interest from the automotive sector. Manufacturing high-performance engineering components from sheet material requires careful design. For sheet metals this problem is usually addressed using numerical simulations. Therefore a pragmatic approach for composite sheet forming is to modify existing packages developed for metal stamping to take account of the complex rheology of composites. In this paper, attention is focused on modelling the various mechanisms that occur during forming of composite sheets. In particular, a model is proposed for the dominant mechanism, intraply shear, based on the matrix rheology and the fibre architecture. The results are used to model composite sheet forming using an explicit non-linear FE code (PAM-FORM).

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Characterization of Textile Composite Behavior: Experiments and Simulations: *Jian Cao*[1]; Xiongqi Peng[1]; *Julie Chen*[2]; Darin Lussier[2]; [1]Northwestern University, Dept. of Mech. Eng., 2145 Sheridan Rd., Evanston, IL 60208 USA; [2]University of Massachusetts-Lowell, Dept. of Mech. Eng., Lowell, MA 01854 USA

Textile composite materials possess superior mechanical properties, such as high specific-strength and high specific-stiffness, and great material architecture flexibility. It is essential to understand the mechanical behavior of textile composites during processing so that optimal design of products and manufacturing processes using these materials can be achieved. This paper presents experimental data using a shear frame test and a numerical procedure for obtaining the effective nonlinear elastic moduli of textile composites during forming. The objective is to develop an efficient material model that considers the microstructure of the textile composite, and can be implemented in the numerical simulation of the stamping process. In this model, the woven fabric is assumed to be nonlinear orthotropic and in a plane stress state. The comparisons between experiments of a forming test and numerical simulations will be presented.

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Consolidation During Non-Isothermal Stamp Forming of Carbon/PA12 Composites: *M. D. Wakeman*[1]; L. Zingraff[1]; P-E. Bourban[1]; J-A. E. Manson[1]; Patrick Blanchard[2]; [1]Swiss Federal Institute of Technology (EPFL), Composites and Polymer Technology Laboratory (LTC), Science Park Bldg. A (PSE-A), CH-1015 Lausanne, Switzerland; [2]Ford Motor Company, Ford Res. Lab., 2101 Village Rd., Mail Drop 3135, Dearborn, MI 48121 USA

Stamp forming of thermoplastic composite materials offers the potential for rapid processing of shell-like components. The highly non-isothermal process is suited to a variety of thermoplastic composite precursor materials, ranging from dry unconsolidated commingled yarns to fully impregnated products. Initial void contents have a strong effect on the subsequent stamping process whereby the consolidation accompanying the deformation and heat transfer processes during stamping is reduced with higher impregnation levels. Reduced void contents in the final product increase mechanical properties and are related to the initial material form. To investigate these phenomena, carbon fibre weave reinforced polyamide 12 sheets, in two material forms, have been studied. A commingled yarn system, with varying degrees of preconsolidation, and fully impregnated sheets produced by the impregnation of carbon weaves with anionically polymerized laurolactam have been examined. The void content during the stamping process has been mapped experimentally and compared to a non-isothermal consolidation simulation to show the importance of the initial material consolidation quality on the properties of the final stamped component.

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Integrated Virtual Prototyping for Composite Design, Analysis and Manufacturing: *Olivier Guillermin*[1]; [1]Composite Design Technologies, 486 Totten Pond Rd., Waltham, MA 02451 USA

Tight interaction between design, analysis and manufacturing is required to engineer a composite part. In most cases, the final part contains details and modifications that create significant differences between the design model, the analysis model, and the manufactured part. For example, dramatic changes in fiber orientation can occur which induce wrinkling and large thickness changes. Or ply drop-offs and staggers may change the laminate stack-up symmetry and balance. All of these issues can have a considerable effect on the performance of the part. This paper describes how the FiberSIM suite of CAD integrated software tools for composites can be used to address these issues. For the first time, the link between CAD software, analysis packages, and manufacturing equipment makes it possible for designers, analysts and manufacturing engineers to access the CAD master model of a composite part in its to-be-manu-

factured state. Engineers can verify at any time during the design and manufacturing process that the part meets the performance specifications. Practical case studies from industry will highlight how composite engineering is improved and risk is reduced through the use of a complete and detailed virtual prototype.

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Evolution and Management of Process-Induced Damage in Polymer Composite Panels: *A. Sherif El-Gizawy*[1]; *Yean-Der Kuan*[1]; [1]University of Missouri-Columbia, Mech. and Aero. Eng., E3412 Eng. Bldg. E., Columbia, MO 65211 USA

A numerical model describing the evolution of process-induced damage in molded composite panels with woven fiber mats was developed. Part warpage (deformation), and residual stresses developed inside the panels are the damage types considered in this work. The effects of thermo-mechanical and thermo-chemical responses of the material on the evolution of damage during resin transfer molding of the panels are quantified. This numerical model in conjunction with an optimization module based on Simulated Annealing (SA) technique are used in conducting parametric design analysis for minimization of process-induced damage in composite panels. Experimental case studies utilizing epoxy resin with eight harness graphite fiber mats are used to verify the numerical model. Damage predictions from the present model are in agreement with the experimental measurements. The parametric analysis conducted on rectangle panels using the developed system indicates that high cure temperature, low heating rate and low rate of cooling after cure would minimize warpage and residual stresses.

Teaching and Learning Solid State Diffusion - Panel Discussion

Sponsored by: ASM International: Materials Science Critical Technology Sector, Atomic Transport Committee
Program Organizers: Richard D. Sisson, Worcester Polytechnic Institute, Materials Science & Engineering, Worcester, MA 01609 USA; Joe I. Goldstein, University of Massachusetts, Department of Engineering, Amherst, MA 01020 USA; John Morral, University of Connecticut, Department of Metallurgy, Storrs, CT 6260 USA

Wednesday PM Room: 202
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Richard D. Sisson, Worcester Polytechnic Institute, Mats. Sci. and Eng., Worcester, Massachusetts 01609 USA; John Morral, University of Connecticut, Dept. of Metall. and Mats. Eng., Storrs, CT USA

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Teaching and Learning Diffusion and Other Rate Processes: *Richard D. Sisson*[1]; M. M. Makhlouf[1]; [1]Worcester Polytechnic Institute, Mats. Sci. and Eng. Dept., 100 Institute Rd., Worcester, MA 01609 USA

Learning the fundamentals of diffusion in solids, conduction heat transfer and other dynamic processes is sometimes difficult for undergraduate and graduate students. The traditional teaching method of lectures emphasizing the solution to the Fick's or Fourier's Laws with a variety of boundary conditions does not address the learning styles of many students. In this presentation alternative teaching methods will be presented that may help students with a variety of learning styles learn the fundamentals of solid state diffusion. The incorporation of more active learning and team projects will also be presented and discussed.

2:25 PM Panel Discussion

Following the Opening Presentation, the AM Presenters will Form a Panel for an Open Ended Discussion on Teaching and Learning Diffusion.

NOTES