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) _____

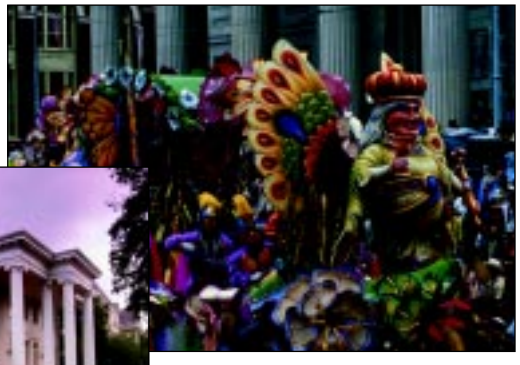
Signature: _____

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.*

TECHNICAL PROGRAM

Ernest N. Morial Convention Center * New Orleans, Louisiana USA * February 11 - 15, 2001

MONDAY AM

2001 EXHIBITION
12:00 PM - 6:00 PM
Ernest N. Morial Convention Center - Hall A

★★★

Grand Opening Reception
5:00 PM - 6:00 PM
Ernest N. Morial Convention Center - Hall A

★★★

Product & Technology Mini-Session
11:45 AM - 2:00 PM
Ernest N. Morial Convention Center - La Louisiane Ballroom A

★★★

TUTORIAL LUNCHEON LECTURE
Extractive Metallurgy Tutorial Luncheon
12:00 PM - 1:30 PM
Hilton Riverside Hotel - Melrose

★★★

Larry Kaufman Honorary Dinner
6:00 PM - 10:00 PM
Hilton Riverside Hotel - Marlborough A&B

2001: An Odyssey of Materials in Space: Advanced Systems and Materials for Space I: High Conductivity Materials

Sponsored by: Extraction & Processing Division, Light Metals Division, Aluminum Committee, Copper, Nickel, Cobalt Committee
Program Organizers: Daniel B. Miracle, Wright Laboratory, Materials Directorate, Bldg 655, WPAFB, OH 45433 USA; Enrique V. Barrera, Rice University, Met. Eng. & Mats. Sci. Dept., Houston, TX 77251 USA

Monday AM Room: 223
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: TBA

8:30 AM Keynote
Advanced Materials for Launch Vehicles and Spacecraft: *Carl Zweben*[1]; [1]Composites Consultant, 62 Arlington Rd., Devon, PA 19333 USA

Space is of vital commercial and military importance, worldwide. Advanced metallic materials and composites are critical, and in many instances enabling, for a large and increasing number of launch vehicle and spacecraft applications. These new materials are gradually replacing the traditional metals used in spacecraft and launch vehicles, such as aluminum, titanium, beryllium, magnesium, steel and superalloys. Composites have great potential in virtually all subsystems, including propulsion, mechanisms, electronics, power, and thermal management. Materials of interest include not only polymer matrix composites (PMCs), currently the most widely used class of structural materials, and carbon-carbon composites (CCCs), which are well established for thermal protection, but also ceramic matrix composites (CMCs), metal matrix composites (MMCs) and other types of carbon matrix composites (CAMCs). In this paper, we consider the unique environments faced by materials used in space and launch vehicles; material requirements; key advanced materials, including advanced monolithic metallic materials and the four classes of composites (PMCs), (CCCs), (CMCs), (MMCs) and (CAMCs); and current applications in structures, thermal management, propulsion, mechanisms, electronic packaging and power subsystems. We also look at barriers to introduction of new materials and likely future directions.

9:00 AM Invited
Synthetic Multifunctional Materials–A New Enabling Technology for Space Missions: *Leo Christodoulou*[1]; Arthur M. Diness[2]; [1]DARPA, DSO, Fairfax Ave., Arlington, VA USA; [2]Institute for Defense Analysis, Alexandria, VA USA

Synthetic Multifunctional Materials (SMFM) are microarchitectured materials explicitly designed, synthesized and/or fabricated to realize multiple performance objectives. For purposes of a new program direction sponsored by DARPA's Defense Sciences Office, a multifunctional material is defined as a structural material with at least one additional performance-linked non-structural function. These additional functions generally impact future space applications, including power generation, self-repair/self-monitoring, sensing, actuation, thermal management, ballistic and/or blast protection, signature management and catalytic/surface chemical activity. This approach is in contrast to the achievement of multifunctional behavior of a structure by means of linked discrete components. The establishment of basic understanding and principles, synthesis and processing routes, rules and tools for the design of and design with SMFM will offer options for achieving benefits such as reduced weight, higher levels of performance, more flexibility in design, reduction of complexity and greater reliability. Challenging directions for SMFM such as design insights, intelligent materials, engineered microstructures, bio-inspiration, mathematical techniques in design and modeling and optimization will be selectively discussed. The efficiencies introduced by development and use of multifunctional materials with structural capabilities will open up new opportunities in space-related materials technologies and space-related missions.

9:30 AM
Mechanical Properties Database For Extruded GRCop-84: *David L. Ellis*[1]; Dennis J. Keller[2]; Bradley A. Lerch[3]; [1]Case Western Reserve University, Matls. Sci. and Eng. Dept., White Bldg., 10900 Euclid Ave., Cleveland, OH 44106 USA; [2]RealWorld Quality Systems, Inc., 20388 Bonnie Bank Blvd., Cleveland, OH 44116 USA; [3]NASA Glenn Research Center, Life Predict. Brnch., M.S. 49-7, 21000 Brookpark Rd., Cleveland, OH 44135 USA

GRCop-84 (Cu-8 at. % Cr-4 at. % Nb) has demonstrated high thermal conductivity and low thermal expansion. This makes it an excellent candidate for combustion chamber liners in regeneratively cooled rocket engines and other heat exchangers. For designing these structures, a mechanical properties database including the tensile, creep and low cycle fatigue properties is required. To generate this database, a design of experiments that included heat treatment was used. Statistical analysis was conducted to generate not only a regression line for the data but also confidence intervals on the values. Results show that the average yield strength of GRCop-84 is nearly twice that of NARloy-Z (Cu-3 wt. % Ag-0. 5 wt. % Zr) between room temperature and 800°C. Creep lives between 500°C and 800°C are considerably longer than NARloy-Z as well. Despite having slightly lower ductility, GRCop-84 has approximately twice the

LCF life of NARloy-Z as well. The simulated braze cycle reduced the strength and creep lives of GRCop-84 but did not appreciably affect the low cycle fatigue lives.

9:50 AM Break

10:10 AM

Improved Properties of Cu-Cr-Nb Alloys Through Mechanical Milling: *Joanna Groza*[1]; Ken R. Anderson[3]; David L. Ellis[2]; [1]University of California-Davis, Dept. of Chem. Eng. and Matls. Sci., Davis, CA 95616 USA; [2]Case Western Reserve University, Matls. Sci. and Eng. Dept., White Bldg., 10900 Euclid Ave., Cleveland, OH 44106 USA; [3]Bechtel Bettis, Inc., Bettis Atomic Power Lab., P.O. Box 79, West Mifflin, PA 15122-0079 USA

Ternary Cu-Cr-Nb alloys, particularly with a composition of 8 at. % Cr and 4 at. % Nb, have demonstrated high strength and high conductivity coupled with good thermal stability. Microstructural refinement to further improve the strength and stability of these alloys was attained by mechanical milling (MM). Mechanically milled Cu-4 Cr-2 Nb and Cu-8 Cr-4 Nb exhibited an increase in hot pressed Vickers hardness of 122% and 96%, respectively. MM produced a corresponding decrease in electrical conductivity of ~33% for both alloys. The increase in hardness was more due to Cu grain-size refinement than to second-phase particle-size refinement. The drop in conductivity was due to second-phase particle-size refinement, which increased both particle/matrix interfacial area and solute solubility. Mechanically processed Cu-4 Cr-2 Nb displayed an enhanced thermal stability. Hot pressed 4 hr-milled Cu-4 Cr-2 Nb experienced a 30% increase in conductivity with only a 22% drop in hardness when annealed at 1273K for 50 hr. Such changes were largely due to an increase in dispersed-particle size (decrease in solute and interfacial electron scattering) and Cu grain size (reduced Hall-Petch effect), respectively. The high strength and stability are essentially due to a combination of small and large, stable Cr2Nb particles, which effectively impede the grain growth of copper matrix. (A figure-of-merit (FOM) coupling hardness and thermal conductivity was developed. This FOM was found to be maximum for the case of 4-hr milled and hot pressed Cu-4 Cr-2 Nb material.)

10:30 AM

Thermo-Mechanical Behaviors of HIPed GRCop-84: *HeeMann Yun*[1]; [1]NASA Glenn Research Center, Matls. Dept., 21000 Brookpark Rd., Cleveland, OH 44135 USA

NARloy-Z (Cu- 3 wt. % Ag-0. 5 wt. % Zr) has been used for combustion chamber liners in regeneratively cooled rocket engines and other heat exchangers. Recently, NASA has developed Cr and Nb added GRCop-84 alloy (Cu-8 at. % Cr-4 at. % Nb) that has demonstrated comparable thermal conductivity and thermal expansion coefficient. For this study, hot isostatic pressing (HIPing) method was utilized for fabricating GR Cop-84. Tensile, creep, and low cycle fatigue (LCF) experiments were conducted on the as-HIPed and the heat-treated (after simulated braze cycle) in the temperature range of 25 to 800°C in air and flowing argon environment. For the statistical analysis on the measured data and experimental variables, a design of experiments (DOE) was used. The tensile yield strength, 1% creep strength, and 1% LCF strength of the HIPed and the heat-treated GRCop-84 are considerably higher than that of the NARloy-Z. The DOE based data analysis and relationships between mechanical properties and microstructures will be discussed.

10:50 AM

Composite Materials for Radiation Shielding during Deep Space Missions: *Richard N. Grugel*[1]; John Watts[1]; James H. Adams[1]; [1]Marshall Space Flight Center, MS-SD47, Huntsville, AL 35812 USA

Minimizing radiation exposure from the galactic cosmic ray (GCR) environment during deep space missions is essential to human health and sensitive instrument survivability. Given the fabrication constraints of space transportation vehicles protective shielding is, consequently, a complicated materials issue. These concerns are presented and considered in view of some novel composite materials being developed/suggested for GCR shielding applications. Advantages and disadvantages of the composites will be discussed as well as the need for coordinated testing/evaluation and modeling efforts.

11:10 AM

Robust Low Cost Liquid Rocket Combustion Chamber by Advanced Vacuum Plasma Process: *Richard Royce Holmes*[1]; Sandra K. Elam[1]; David L. Ellis[2]; Timothy McKechnie[3]; Robert Hickman[3]; [1]NASA Marshall Space Flight Center, SD42, George C. Marshall Space Flight Ctr., Marshall Space Flight Center, AL 35812 USA; [2]Case Western Reserve University, 10900 Euclid Ave., Cleveland, OH USA; [3]Plasma Processes, 4914 D Moores Mill Rd., Huntsville, AL 35811 USA

Next-generation, regeneratively cooled rocket engines will require materials that can withstand high temperatures while retaining high thermal conductivity. Fabrication techniques must be cost efficient so that engine components can be manufactured within the constraints of shrinking budgets. Three technologies have been combined to produce an advanced liquid rocket engine combustion chamber at NASA-Marshall Space Flight Center (MSFC) using relatively low-cost, vacuum-plasma-spray (VPS) techniques. Copper alloy NARloy-Z was replaced with a new high performance Cu-8Cr-4Nb alloy developed by NASA-Glenn Research Center (GRC), which possesses excellent high-temperature strength, creep resistance, and low cycle fatigue behavior combined with exceptional thermal stability. Functional gradient technology, developed building composite cartridges for space furnaces was incorporated to add oxidation resistant and thermal barrier coatings as an integral part of the hot wall of the liner during the VPS process. NiCrAlY, utilized to produce durable protective coating for the space shuttle high pressure fuel turbopump (HPFTP) turbine blades, was used as the functional gradient material coating (FGM). The FGM not only serves as a protection from oxidaton or blanching, the main cause of engine failure, but also serves as a thermal barrier because of its lower thermal conductivity, reducing the temperature of the combustion liner 200°F. from 1000°F to 800°F producing longer life. The objective of this program was to develop and demonstrat the technology to fabricate high-performance, robust, inexpensive combustion chambers for advanced propulsion systems (such as Lockheed-Martin's VentureStar and NASA's Reusable Launch Vehicle, RLV) using the low-cost VPS process. VPS formed combustion chamber test articles have been formed with the FGM hot wall built in and hot fire tested, demonstrating for the first time a coating that will remain intact through the hot firing test, and with no apparent wear. Material physical properties and the hot firing tests are reviewed.

11:30 AM

Impact Crater Similitude and Related Issues for Metal Targets Impacted Below Hypervelocity: *Lawrence E. Murr*[1]; O. L. Valerio[1]; D. Roberson[1]; S. A. Quinones[1]; V. S. Hernandez[1]; N. E. Martinez[1]; E. A. Trillo[1]; F. Horz[2]; [1]University of Texas at El Paso, Metall. and Matls. Eng. Dept., 500 W. University Ave., El Paso, TX 79968-0520 USA; [2]NASA Johnson Space Center, Solar Sys. Exploration Div., Houston, TX 77058 USA

Impact crater shapes in metal targets are often characterized by a penetration depth (p)-to- crater diameter (D_c) ratio: p/D_c, which often approaches a steady-state value of ~0. 5 af and above hypervelocity (± 5 km/s); especially for low-density projectiles striking aluminum alloys. However this steady-state or threshold value has been observed to vary from about 0. 4 to >1 for a range of projectile densities (ρ_p) striking a range of target densities (ρ_t). Moreover, exaggerated crater shapes are observed at impact velocities below hypervelocity, especially in the range of 1 to 2 km/s where values of p/D_c are observed to be as high as 5. 2 for WC projectiles impacting 1100 Al. There are exaggerated crater shape similarities in different projectile/target systems characterized by a corresponding square-root of density ratio ($\sqrt{\rho_p/\rho_t}$). These features result from projectile fragmentation effects. The implications of these observations are that penetration anomalies are exaggerated for very dense particles striking low-density space structures such as aluminum or aluminum alloys at low velocity. This phenomenon is especially feasible in the space debris environment of geosynchronous, low-Earth orbit where the majority of satellites are placed. Research supported by NASA-MURED Grant NAG-9-1171 and NASA Grant NAG-9-1100.

Aluminum Reduction Technology: Prebake Cell Technology

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

Monday AM Room: 206-207
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Claude Vanvoren, Aluminium Pechiney, LRF Tech. Rsch. Ctr., BP 114, Saint Jean de Maurienne 73300 France

8:30 AM

Reduction Cell with Continuous Prebaked Anode-A New Approach: *Birthe Alexa Scholemann*[1]; Siegfried Wilkening[2]; [1]VAW Aluminium AG, Elbewerk, P.O. Box 2269, Stade 21662 Germany; [2]VAW Technologie GmbH, P.O. Box 2468, Bonn 53014 Germany

In the 1960's VAW developed the socalled Erftwerk cell technology, which features a continuous prebaked anode system, still in operation at the Elbewerk smelter today. As a result of continual improvements to process control and operating procedures, the Elbewerk is still a competitive smelter. This paper accentuates the specific advantages of this cell type. The main benefits of the continuous prebaked anode system are: no need for anode changes, complete consumption of anodes and no recling of butts; smaller anode plant, no conventional rodding shop and bath material treatment; production of low-iron aluminium. Plans are on hand to install point feeders and to mechanize manual potroom operations. Modifications are also possible to attain a minor specific energy consumption.

9:00 AM

Potline Amperage Increase from 160 kA to 175 kA during One Month: *Bjorn P. Moxnes*[1]; Egil Furu[2]; Ola Jacobsen[2]; Halvor Kvande[3]; Arnt O. Solbu[2]; [1]Hydro Aluminium Technology Centre Ardal, P.O. Box 303, Ovre, Ardal N-6882 Norway; [2]Hydro Aluminium a.s. Sunndal, P.O. Box 51, Sunndalsora N-6601 Norway; [3]Hydro Aluminium Metal Products Division, Oslo N-0240 Norway

Higher cell amperage now seems to have become a trend in the operation of Hall-Héroult potlines. Many types of prebake cell technologies have shown that they can experience considerable increase in amperage without serious cell operating problems. One vivid example of this is the end-to-end prebake cells at the Hydro Aluminium Sunndal smelter, originally designed for 150 kA, where the amperage of fourteen booster cells was increased from 162 to 175 kA in two months. This gave no operating problems and the current efficiency was maintained at the same level as before. In the summer of 1999 the amperage of the remaining one hundred and seventy cells in the potline was increased by 15 kA up to 175 kA during one month. This would certainly not have been done without the valuable experience gained from the operation of the fourteen booster cells. The measurement program and the operational experiences of this incredibly fast amperage increase are described and discussed.

9:25 AM

Impact of Current Increase on Specific Energy Consumption: *Pierre Beran*[1]; Rene von Kaenel[1]; HansPetter Lange[2]; Jorn Skaar[2]; [1]Alusuisse Technology and Management Limited, Tech. Ctr. Chippis, Chippis 3965 Switzerland; [2]Soral, Sor-Norge Aluminium A/S, Husnes 4560 Norway

Cells at the Søral smelter, originally designed for 100 kA and operated at 127 kA in 1996, were magnetically compensated to further increase the current to 150 kA. Experience showed that an increase in heat losses through the side wall could not be avoided as the current was steadily increased, although the cells remained magnetically stable. The effect of these increased heat losses on specific energy consumption is discussed. Their causes are analysed based on a series of measurements, including ledge scanning, which were carried out annually as the current increased.

9:50 AM

Twenty Years of Continuous Technical Progress at Alucam Prebaked Smelter: *Bassirou Mohamadou*[1]; Raphael Titi Manyaka[2]; Michel Reverdy[3]; [1]ALUCAM, BP 54, Edea, Republic of Cameroon; [2]ALUCAM, BP 1060, Douala, Republic of Cameroon; [3]Aluminium Pechiney, 7 place du Chancelier Adenauer, Paris, Cedex 16 75218 France

Alucam smelter located at Edéa, Cameroon, started in 1957 with the 100kA Pechiney Söderberg design. The 220 cells were installed in 4 buildings with only one line of cells per building allowing better working and safety conditions. It was rettrofitted in 1980 with side-break prebaked 118 kA cells placed in the same location as the Söderberg cells and with 54 additional cells located in two new buildings. Modifications have been systematically tested on cells equipped with a booster rectifier. Pechiney computer models have been used for anode assembly and cathode design modifications; process control was upgraded. Amperage was increased from 118kA to 132kA and current efficiency from 88 to 92% bringing the annual capacity of the smelter from 83kt to 96kt. Quality of work has played a key role in the improvement of the efficiency and the increase of the output, and in limiting the detrimental effect of lack of energy during the dry season.

10:15 AM

Industrial Tests of Retrofitting Soderberg Cell to Prebaked Cell: Peikai Song[1]; Wangxing Li[1]; *Yujing Jiang*[1]; *Jie Li*[2]; Yexiang Liu[2]; [1]China Great Wall Aluminum Corporation, Zhengzhou, Henan 450041 China; [2]Central South University of Technology, Dept. of Metall. Sci. and Eng., Changsha, Hunan 410083 China

Soderberg cell which contributes 60% aluminum production in China is now waiting for modernization. One of such potlines starts to retrofit at China Great Wall Aluminum Corporation (CGWAC). The commercial scale tests were carried out in 1999. Due to the old potroom, cathode busbar system, lining and shells were utilized to the utmost extent, the project investment was lowered. AS advanced technologies, such as full sealing, point feeding, highly intelligent control and reverse two-stage dry scrubbing, etc. were developed and used, satisfactory technical, economic and ecological targets were achieved: current efficiency 91. 61%, DC consumption 13669kWh/t-Al, efficiency of gas collection 97%, the scrubbing efficiency of fluoride 99% and the dust cleaning efficiency 99. 99%. The results showed the retrofit project could not only make the operation of the retrofitted potline meet the increasingly strict environment protection policies in China, but also bring remarkable economic benefit to CGWAC.

10:40 AM Break

10:50 AM

Application of Lithium Modified Electrolyte in High Current Density Aluminum Reduction Cells: *B. Hullett*[1]; S. Stejer[1]; N. Urata[1]; [1]Kaiser Aluminum Center for Technology

Between 1996 and 2000, Kaiser Aluminum tested varying compositions of lithium modified electrolyte in a potline of high anode current density prebake cells at the Mead, WA smelter. The lithium electrolyte operating parameters and resulting potline performance are compared with non-lithium potline performance. The test was pursued to allow higher amperage operation, reduce specific energy consumption and to measure the impact on cell fluoride emissions. The initial electrolyte composition allowed a 4% operating amperage increase with slightly reduced specific energy consumption. Over time, the electrolyte AlF_3 content was increased to improve current efficiency until anode cathode distance limitation inhibited further increase. Gaseous fluoride emissions from the cells showed a 50% reduction compared to Mead's standard electrolyte chemistry. Lithium operation is feasible for further increasing production in high current density cells and can be used to achieve an optimum economic balance between lower fluoride emissions, specific energy and current efficiency.

11:15 AM

AP35: The Latest High Performance Commercially Available New Cell Technology: *Claude Vanvoren*[1]; Pierre Homsi[2]; Benoit

Feve[1]; Bernard Molinier[1]; Yvon di Giovanni[1]; [1]Aluminium Pechiney, LRF Tech. Rsch. Ctr., BP 114, Saint Jean de Maurienne 73300 France; [2]Aluminium Pechiney, Ctr. de Tech. ALUVAL, Zi de Voreppe Moirans, BP 7, Voreppe 38340 France

Pursuing the goal of designing high performance, high productivity and low capital cost cell technology, Aluminium Pechiney re-engineered its well known AP 30 reduction cell to create an advanced cell operating at about 350 kA and called AP 35. Following the avenue opened with the re-engineered smaller sister cell AP 21, the AP 35 design integrates the latest development in lining design, anode assembly and side wall ventilation. Eight test pots have been operating for four years at the St Jean de Maurienne smelter, allowing for extensive industrial trials of several designs and material variations. Technical performances are presented and discussed. The new cell technology is also available for retrofitting of current AP 30 potlines, providing an extra 9% production output (compared to an average operation at 320 kA) with modifications depending on the technical limits of the existing facilities.

11:40 AM

The Developing of GP-320 Cell Technology in China: *Yin Ensheng*[1]; Liu Yonggang[1]; Xi Canmin[1]; Zhang Jiazhi[1]; [1]Pingguo Aluminium Company, Pingguo, Guangxi, China

Aluminium Smelting technology in China before the 1980s was dominated by Soderberg cells, with cell current lower than 80 kA. In the early 1980s, an out-of-date 160 kA prebaked cell technology was imported from Nippon Light Metals. Cell technology operated at that upper current level although a more up-to-date of 160 kA cell technology was developed and installed at the Guangxi site with successful operation starting in 1995. A 320-kA cell technology was developed in 1998 through joint research between Guiyang Aluminium & Magnesium Design and Research Institute and Pingguo Aluminium Company, and without trial a potline (30 pots) was constructed and started in October 1999. The present paper describes features of the advanced technology, which was incorporated in this GP-320 cell design. The GP-320 pots have stable operation at a current of 325 kA. The early current efficiency of 93. 5 to 94% has subsequently been exceeded. The successful operation without any pilot trial and preceding high current experience in China, represents a great leap forward in the aluminium smelting technology. It will help narrow the gap between China's aluminium technology and that of the developed countries. China will take this high-capacity cell design as the basis to speed up the development of the aluminium industry in the future.

Carbon Technology: Anode Raw Materials

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

Monday AM — Room: 215-216
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Amir A. Mirtchi, Alcan International, Ltd., Reduc. Techn. Svc., Jonquiere, Quebec G7S 4K8 Canada

8:30 AM

Size Exclusion Chromatography in the Analysis of Pitch: *Birgit E. Hansen*[1]; Olof Malmros[2]; Nigel R. Turner[3]; Erling H. Stenby[1]; Simon I. Andersen[1]; [1]Technical Universtiy of Denmark, Dept. of Chemical Eng., 2800 Lyngby, Copenhagen Denmark; [2]Koppers Europe, Avernakke, 5800 Nyborg, Denmark; [3]Koppers Europe, Meridian House, Normanby Rd., Scunthorpe, North Lincolnshire DN15 8QX UK

Size exclusion chromatography (SEC) has been investigated as a tool to analyze pitch. SEC is a high pressure liquid chromatography (HPLC) method where the components are being separated by molecular size. The method is well known from the science of polymers where it is used to obtain molecular weight distributions. Several mobile phases were investigated. It was found that when the column was operated at elevated temperature and N-methyl-2-pyrollidinone (NMP) was used as mobile phase, the chromatographic system could be operated without interference from effects not related to molecular size. Additionally, the chromatographic system allowed analysis of the entire quinoline soluble phase. The method was applied to pitch distilled from feedstock tar that had received mild thermal treatment. It was possible to follow the formation of large molecular species by analyzing the toluene insoluble fraction of pitch in NMP. Chromatographic fractions were collected for further analysis by fluorescence spectroscopy.

8:55 AM

Development of Petroleum Enhanced Coal Tar Pitches in Europe: *Nigel R. Turner*[1]; Stewart H. Alsop[1]; Olof Malmros[1]; David Whittle[1]; Birgit E. Hansen[2]; Simon I. Andersen[2]; Erling H. Stenby[2]; [1]Koppers Europe, Avernakke, Nyborg 5800 Denmark; [1]Koppers UK Limited, Scunthorpe Works, Dawes Lane, Scunthorpe, North Lincolnshire DN15 6UR UK; [2]Technical University of Denmark, Dept. of Chem. Eng., Lyngby 2800 Denmark

Petroleum enhanced coal tar pitches are proven industrial products in the USA. European implementation of similar technology is a preferred response to market analysis predictions for future pitch demand. The paper will discuss laboratory scale product development based on similar technology to Koppers in the USA, but based predominantly around more local materials for economic and strategic reasons. Successful development has called for identification of suitable petroleum raw materials and methodology to combine petroleum and coal tar components to make a homogeneous, performance products. Pitch properties and bench scale anode information will be part of the paper. Traditional pitch test results fail to do full justice to the enhanced binder materials. Bench scale anode results indicate unanticipated, positive synergies between coal tar and petroleum derived molecules.

9:20 AM

Preparation of Binder Pitches by Blending Coal-Tar and Petroleum Pitches: M. Pérez[1]; *Marcos Granda*[1]; R. García[1]; E. Romero[2]; R. Menéndez[1]; [1]Instituto Nacional del Carbón, CSIC, La Corredoria s/n, Apartado 73, 33080-Oviedo Spain; [2]Repsol Petróleo, S. A., Valle de Escombreras, 30350-Cartagena Spain

The use of petroleum pitches as binders for Söderberg anodes has the disadvantage that volatiles are removed over 400°C, due to cracking reactions, leading to cokes with high porosity. Moreover, petroleum pitches generate low viscosity systems on carbonization, making the preparation of paste with an appropriate viscosity more difficult. However, a great advantage of petroleum pitches is the almost total absence of toxic and cancerous compounds. The blend of petroleum and coal-tar pitches can be a successful way to improve the binder properties of petroleum pitches. The inherent binder characteristics of coal-tar pitch regulate the viscosity of the blend, while petroleum pitch drastically reduces the emission of toxic and cancerous compounds. This paper reports on the formulation and characterization of coal-tar/petroleum pitch blends. Special emphasis is placed on the possible interactions between the components of the pitches.

9:45 AM

Development of Binder Pitches from Coal Extract and Coal-Tar Pitch Blends: *Peter G. Stansberry*[1]; John William Zondlo[1]; Robert H. Wombles[2]; [1]West Virginia University, Dept. of Chem. Eng., 314 Eng. Sci. Bldg., P.O. Box 6102, Morgantown, WV 26506-6102 USA; [2]Koppers Industries, Inc., 1005 William Pitt Way, Pittsburgh, PA 15238-1362 USA

There is concern among U.S. industrial pitch producers about domestic sourcing of quality binder pitches. Binder pitches are essential ingredients in the manufacture of carbon anodes. Although considerable work has focused on combining petroleum-derived materials with conventional coar-tar pitches, relatively little effort has been directed toward using solvent extracts from coal as feedstocks for blending. Researchers from West Virginia University and Koppers Industries, Inc. studied the effects of pitches developed by solvent extraction of coal. These coal-derived pitches were mixed with conventional coal-tar pitches in quantities sufficient to form laboratory-scale anodes. Formulations with a standard petroleum and coal-

tar pitch were used as controls. The binder pitches, green and baked anodes were then subjected to a battery of standard tests. The results of using solvent-extracted coal pitch on anode characteristics including thermal conductivity, electrical resistivity, compressive and flexural strength, air and CO_2 reactivity, etc. will be presented.

10:10 AM Break

10:20 AM
A Review of Coke Quality Projections: *M. Franz Vogt*[1]; Les Edwards[1]; [1]CII Carbon, L. L. C., 1615 E. Judge Perez Dr., 4th Fl., P.O. Box 1306, Chalmette, LA 70044 USA

Three-fourths of the worlds calcined coke production is used in aluminum smelting. Growth in the primary aluminum industry has been running at 4% per year for the past several years. As growth continues, demand for calcined coke will grow at nearly the same rate. On the other hand, the supply of high quality green coke which is suitable for use as a raw material in anode grade coke production has remained constant. In some regions of the world, particularly North America, the availability has decreased. This paper reviews the supply/demand balance for anode grade calcined coke and the raw materials on which it is dependent. The trends of key quality measurements such as density, sulphur, and vanadium, and the factors which influence these trends are reviewed. The projections for the future indicate that challenges face the calcining industry in meeting the requirements of aluminum smelters.

10:45 AM
Calcined Coke from Crude Oil to Customer Silo: Howard Childs[1]; Bernard Vitchus[1]; *Frank R. Cannova*[1]; [1]ARCO Products Company, 1990 W. Crescent Ave., Anaheim, CA 92801 USA

The quality of calcined petroleum coke used in primary aluminum production is affected by the refining and calcining processes-including choice of crude,crude processing, coking, and calcining as well as the handling systems between the refinery, calciner, and customer. This paper will discuss howeach of these processes affects the final quality of the resulting calcined coke to provide insight as to how refining economics will be affecting calcined coke quality in the future.

11:10 AM
Desulfurization of Petroleum Coke Beyond 1600°C: *Christopher Alan Paul*[1]; Louis E. Herrington[2]; [1]Great Lakes Carbon Corporation, P.O. Box C, Port Arthur, TX 77641 USA; [2]LEHCO, 112 Wildoak Dr., Daphne, AL 36526 USA

Calcining experiments using a graphite tube-furnace at temperatures of 1300-1650°C were conducted to determine the degree of thermal desulfurization that occurs in petroleum coke at these temperatures. Raw sponge and shot coke with ~4 wt% sulfur were used in the experiments. The samples were calcined at residence times of 30-60 minutes. Over 91% desulfurization was achieved in the experiments resulting in a product with 0.35 wt% sulfur. Nitrogen removal will also be measured and discussed. Although density of the calcined coke decreases when calcining at high temperatures, the objective of this work is to determine if high sulfur (low cost) raw coke can be calcined and desulfurized for use as calcined low sulfur industrial product (IP) grade coke. Density and metals are usually not a problem for IP coke. Uses of IP grade calcined coke include TiO_2 production and recarburizer for steel.

11:35 AM
Characterization of Porosity in Cokes by Image Analysis: *Stein Rørvik*[1]; Harald A. Øye[2]; Morten Sørlie[3]; [1]SINTEF Applied Chemistry, Inorg. Proc. and Ana., Trondheim N-7465 Norway; [2]Norwegian University of Science and Technology, Instit. of Chem., Trondheim N-7491 Norway; [3]Elkem ASA Research, Vågsbygd N-4675 Norway

A fully automatic method for image analysis of coke porosity has been developed. The method outputs a continuous pore size distribution from 1μm to 10 mm, and will therefore cover a much broader range than mercury porosity. The method measures only pores inside the coke grains; voids between coke grains in the sample are ignored. A selection of calcined commercial cokes in different size fractions has been analyzed. There are considerable differences in the pore size distributions of the different cokes.

Cast Shop Technology: Training and Safety

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

Monday AM — Room: 208-210
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Seymour G. Epstein, The Aluminum Association, 900 19th St., NW, Washington, DC 20902 USA, Aluminum Association; John Jacoby, Consultant, 3398 North Hills Rd., Murrysville, PA 15668 USA

8:30 AM
Skill Training-Confidence through Competence: *John Hansen*[1]; [1]Kaiser Aluminum & Chemical Corporation, Met. Prod., 3400 Taylor Way, Tacoma, WA 98421 USA

Industry is forced to continually evaluate training needs to support their business goals. In response to that need our Continuous Cast Aluminum Rod Mill has implemented a competency based program which gives explicit and detailed instruction for task completion. The payoff of this concept comes in a modular, more efficient training program which enables personnel to achieve a consistent high standard of success in the production of Electrical Cable and DeOx products. Each task is analyzed; tasks are divided in to steps and sub-steps. Specific standards of performance for each step are then developed. Training Objectives come directly from task analysis and Competency Criteria come directly from the standards of performance. The progress is measured through a process of task accomplishment verification and re-verification. Extensive use of digital photograph laden manuals and desktop Presentation media ensuress that personnel are given an optimal chance to experience 100% success in their work.

8:50 AM
Cast House Flooring–A Ten Year Case Study–Neuman Aluminum USA, Ltd.: *Michael Polinko*[2]; *Dave Quilter*[1]; [1]Thermal Systems America, Canastota Industrial Park, One Madison Blvd., Canastota, NY 13032 USA; [2]Neuman Aluminum USA, Ltd., Dunsmore Rd., P.O. Box 160, Verona, VA 24482 USA

Many different methods of aluminum cast shop flooring have been used over the years. Our presentation will present a look at one of the very first Thermal Floor Tile applications at Neuman Aluminum. The flooring system has been in service since December 1990. Neuman's application was the charge area an aluminum melter. Plant personnel were always repairing the area with little success. Forktruck loading of the charge was not smooth and space was very limited given that there was only one way in and out for the charging forktruck. The floor tiles were used because of their robust design and non-wetting properties. The paper will incorporate photos from 1990 and now along with drawings of the area and a simple straight forward economic analysis of the project.

9:10 AM
An Update on the Aluminum Association's Molten Metal Safety Program: *Seymour G. Epstein*[1]; [1]The Aluminum Association, 900 19th St., NW, Washington, DC 20902 USA

The aluminum industry continues inits efforts to better understand molten metal explosions, the conditions under which they occur, and how they may be prevented. The Aluminum Association has long considered the handling of molten aluminum its single-greatest safety priority and has an ongoing, multi-faceted program to address the safe handling of molten aluminum. A number of aluminum companies, in the U.S. and abroad, participate in this program which includes research into the causes and prevention of molten aluminum-water explosions; guidelines for handling molten aluminum, for scrap receiving and inspection, and for sow casting and charging; molten metal incident reporting; scrap rejection notification; testing of fabrics to protect employees exposed to molten metal and pot bath; and a series of workshops, presentations and

training aids to increase awareness. An update on these efforts will be presented.

9:35 AM

Effect of Coating Cure Time on Adhesion and Explosion Avoidance: *Ray T. Richter*[1]; David D. Leon[1]; Thomas L. Levendusky[2]; [1]Alcoa, Inc., Ingot & Solid. Platform, 100 Technical Dr. ISP-B, Alcoa Center, PA 15069-0001 USA; [2]Alcoa, Inc., Pack. Coat. & Surf. Techn., 100 Technical Dr. PCST-A, Alcoa Center, PA 15069-0001 USA

During the period of 1995 August through 1997 March, research contracted by the Aluminum Association on behalf of a group of sponsoring companies, identified three alternate coating materials which would be an acceptable replacement for Tarset Standard to prevent molten metal and water steam explosions. These new coatings were: 1) Intertuf 132HS a coal tar epoxy by Courtaulds; 2) Multi-Gard 955CP a 100% solids epoxy by Carboline; and 3) WiseChem E-115 a 100% solids epoxy by ESP. These three coatings, as well WiseChem E-212-F, when tested in the past, were all evaluated for explosion avoidance using the recommended cure times as provided by the manufacturer. These recommended cure times are generally considered excessive from a productivity viewpoint because they can range from 12 to as high as 168 hours. The Aluminum Association contracted with Alcoa, Inc. in 1998 to investigate the affect of reduced cure times on adhesion and explosion avoidance of the four above coatings. This report reviews the resul ts of this investigation which was targeted at determining the potential for avoiding molten metal and water steam explosions when cure times were reduced to as low as one hour. Also investigated was the effect of reduced cure times on coating adhesion when exposed to direct water impingement and the ability of coatings to prevent explosions after repeated exposure to molten metal.

10:00 AM Break

10:10 AM

Fundamental Studies on Molten Aluminum-Water Explosion: *Rusi P. Taleyarkhan*[1]; Seokho Howard Kim[1]; [1]Oak Ridge National Laboratory, Eng. Techn. Div., Bldg. 9204-1 MS 8045, Oak Ridge, TN 37831-8045 USA

A joint project has been established between Oak Ridge National Laboratory (ORNL) and the Aluminum Association (AA). ORNL's work is composed of variously scaled experiments simulating key phenomenologies connected with "onset" of molten aluminum-water explosions coupled with development of alternate novel methods for prevention. This joint work is being conducted in two phases. Work conducted during Phase 1 has been reported during TMS99. Phase 2 work consisted of addressing issues related to curing times for coatings necessary for assuring protection from explosion onset, as well as on design and testing of confirmatory tests with 50-lb melt drops to demonstrate the importance of non-condensible gases on explosion prevention. Results from ORNL tests using the Steam Explosion Triggering Studies (SETS) facility were used to guide 50-lb melt drop tests sponsored by AA. Good agreement was obtained between ORNL and AA-sponso red tests. The paper discusses results of tests on selected coatings with various degrees of curing, the impact of water temperature, test data with non-condensible gas injection, and recent discoveries related to use of microwave technology for accelerating the curing process to the time frame of minutes from close to more than a hundred hours.

10:35 AM

Molten Metal-Water Explosions with Aluminum Alloys Containing Significant Amounts of Lead and Bismuth: *John E. Jacoby*[1]; [1]Consultant, 3398 North Hills Rd., Murrysville, PA 15668 USA

Explosion data collected by the Aluminum Association reveals that numerous severe molten aluminum explosions have occurred with alloys containing significant amounts (>0.8%) of lead plus bismuth. Are these alloys more dangerous than other aluminum alloys? The answer is obviously yes. This paper will include discussions of the thermite reaction, nature of the oxide film, volatility, alloying characteristics, reaction with casting lubricants and long solidification range which may help explain why these alloys have generated a poor safety record. The procedures that can be used to minimize the explosion risks during melting, transfer and direct chill casting will also be discussed in detail.

11:00 AM

Elements of Effective Safety Training Programs for Aluminum Casting Operations: *J. Martin Ekenes*[1]; [1]Hydro Aluminum Hycast, P.O. Box 603, Otis Orchards, WA 99027-0603 USA

In order to compete effectively in a global economy, aluminum casting operations must develop and maintain a qualified and competent workforce. A significant part of this effort must center on employee training, especially safety training. In addition to providing employees with accurate information, effective safety training programs will recognize certain principles governing human behavior. This paper reviews topical content appropriate for safety training programs to be used in aluminum casting operations and suggests ways to enhance the effectiveness of such training by incorporating principles of human behavior.

11:25 AM

Panel Discussion on Casthouse Safety: *Seymour G. Epstein*[1]; John E. Jacoby[4]; J. Martin Ekenes[5]; Ray T. Richter[2]; Rusi P. Taleyarkhan[3]; [1]Aluminum Association; [2]Alcoa; [3]Oak Ridge National Laboratory; [4]Consultant; [5]Hydro Aluminum Hycast

Authors of the previous papers will interactively discuss casthouse safety issues with the audience.

Chemistry and Electrochemistry of Corrosion and Stress Corrosion: A Symposium Honoring the Contributions of R.W. Staehle: Mechanisms and Modeling - I

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

Monday AM — Room: 222
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Russell H. Jones, Pacific Northwest National Laboratory, Mats. Sci. Dept., Richland, Washington 99352 USA; John P. Hirth, Herford, AZ 85625 USA

8:30 AM Opening Remarks: R. H. Jones

8:40 AM Keynote

Mean Value, Dispersion, and Initiation Time in Stress Corrosion Cracking as Objectives for Fundamental Investigations: *Roger W. Staehle*[1]; [1]University of Minnesota, 22 Red Fox Rd., North Oaks, MN 55127 USA

Most experimentation and fundamental studies of stress corrosion cracking (SCC) have concentrated on determining the mean value of time-to-failure, initiation time, or propagation rate. Such results are used to predict the occurrence of penetration or perforation of materials in components. However, the mean value, by definition, is concerned with 50% failure. On the other hand, most components either can tolerate no failures or only some relatively small number of failures, e.g. one failure of 1000 or one of 10,000. For such objectives the mean value, by itself, is not useful. It is necessary to determine the dispersion of data, with the intercept being the mean, from which small fractional amounts of failure can be determined. The dispersion is the slope of the cumulative distribution suitably linearized depending on the form of the statistical relationship being used. While there is an extensive literature that implicitly is concerned with mean values and with the fundamental aspects of its rational ization, there is no theory that provides a physical basis for the dispersion of either SCC or any other corrosion mode. There is also no physical theory for the initiation time as calculated statistically. The purpose of this discussion is to define the dispersion and initiation time and to identify physical factors

that influence their magnitudes. Also, the magnitudes of the dispersion and initiation time are greatly affected by the stressors such as temperature, stress and concentration of species in solution in such a way that the dispersion that is determined in accelerated testing may be quite different from the dispersion that is realized in the engineering application. Thus, an acceleration that is determined by comparing mean values may bear little similarity to the acceleration of data necessary to predict failure of small fractions such as 0. 01%. The implications of such possible disparities is explored.

9:25 AM

Mechanochemical Mechanisms in Stress Corrosion: *John J. Gilman*[1]; [1]University of California at Los Angeles, Dept. of Mats. Sci. and Eng., 6532 Boelter Hall, 405 Hilgard Ave., Los Angeles, CA 90095-1595 USA

This paper is concerned primarily with chemical reactions that are driven directly by mechanical potentials. This case is important in stress corrosion along with chemical and thermal potentials, but the mechanism has remained obscure until relatively recently because it was treated in an indirect fashion; and because it focussed on dilatational, rather than shear potentials. Chemical reactivity is determined by "chemical hardness". That is, by the gap in the bonding energy spectrum between the bonding and the anti-bonding energy states. It always increases during a chemical reaction. Bond-bending has a large effect on chemical hardness, while stretching has only a small effect. This leads to a characteristic rate law based on Zener's theory of electron tunneling that is consistent with observations of stress corrosion, and with the very direct experiments called "hammer chemistry".

9:55 AM

Vacancies in SCC Mechanisms: *Jose R. Galvele*[1]; [1]Com. Nac. Energia Atomica, Instituto de Tecnologia, Avda. Libertador 8250, Buenos Aires 1429 Argentina

Numerous publications have considered the influence of dislocations on the propagation of cracks by stress corrosion. On the other hand, a significant number of mechanisms include the action of vacancies in the SCC process, but no explicit analysis is made of their role. A critical review is made in the present paper of the role played by the vacancies in those SCC mechanisms. The source as well as the movement of the vacancies is considered, and their significance in the various mechanisms is analyzed.

10:25 AM

Physical and Numerical Modelling of the Stress Corrosion Cracking Behaviour of Austenitic Stainless Steels and Nickel Base Alloys in PWR and Chloride Solutions: *Thierry Magnin*[1]; D. Tanguy[1]; D. Delafosse[1]; [1]Ecole des mines de St Etienne, Centre SMS, 158 Cours Fauriel, 42023 St. Etienne Cedex 2 France

The main results of slow strain rate tests of austenitic stainless steels and alloy 600 in PWR and chloride containing solutions will be given, as a function of the applied electrochemical potentials. The role of both anodic dissolution and hydrogen effects will be emphasised through the "corrosion enhanced plasticity model" proposed some years ago by T. Magnin to describe the trans and the intergranular stress corrosion cracking mechanisms in fcc materials. Numerical simulations of the damaging effects will be presented: first at the scale of the micron to model the hydrogen-dislocation interaction at the stress corrosion crack tip and, secondly, at the atomic scale to model by molecular dynamics the hydrogen segregation at grain boundaries. Such results will be discussed in terms of modelling of the stress corrosion cracking velocity.

10:55 AM

Immunity, Thresholds, and Other SCC Fiction: *Peter L. Andresen*[1]; [1]GE Corporate R&D Center, 1 River Rd., K1-3A39, Schenectady, NY 12301 USA

The engineering view of SCC in hot water emphasizes regions of immunity and thresholds in stress intensity, corrosion potential, alloy/condition, radiation fluence, temperature, etc. However, fundamentally these concepts almost universally fail to stand up to the scientific challenge of modern measurements or mechanistic understanding. Detailed crack growth rate studies on austenitic stainless steels and Ni alloys reveal that conditions that were once widely assumed to represent SCC immunity do produce well-controlled, although often low, stress corrosion crack growth rates. Similarly, the concepts of a threshold stress intensity, threshold fluence, threshold corrosion potential, etc. provide a distorted perspective of SCC, because there is almost always a continuity in the response surface, not a genuine threshold-although a few examples of SCC immunity in hot water are discussed. The role of flawed test data in shaping our understanding of SCC, and the specific measurement factors, including SCC test design, thatgive rise to apparent immunity and threshold behavior are discussed.

11:25 AM

A Critical Potential for the Stress Corrosion Cracking of Fe-Cr-Ni Alloys and its Mechanistic Implications: *Gustavo A. Cragnolino*[1]; Darrell S. Dunn[1]; Yi-Ming Pan[1]; Narasi Sridhar[1]; [1]Southwest Research Institute, Ctr. for Nuc. Waste Regulatory Analyses (CNWRA), 6220 Culebra Rd., San Antonio, TX 78238-5166 USA

Since Uhlig introduced in the 70s the concept of a critical potential for the stress corrosion cracking (SCC) of austenitic stainless steel (SS) in hot concentrated chloride solutions to support his proposed mechanism of adsorption-induced SCC, several mechanisms have been suggested and discussed in the literature. SCC of Fe-Cr-Ni alloys has been interpreted in terms of hydrogen-induced cracking, slip dissolution/film rupture, film-induced cleavage, and surface mobility, as well as variations of these mechanisms. In this paper we discuss the validity of a critical potential concept on the bases of experimental results reported in the literature for Fe-Cr-Ni-Mo alloys and our own work using alloys with different Ni contents, such as type 316L SS (Fe-18Cr-12Ni-2. 5Mo), alloy 825 (29Fe-22Cr-42Ni-3Mo) and alloy 22 (4Fe-22Cr-58Ni-13Mo-3W), in concentrated chloride solutions at temperatures ranging from 95 to 120ºC. We conclude that the existence of this potential, although valid for alloys containing less than 42%Ni within certain ranges of chloride concentrations and temperatures, cannot be interpreted in support of any of the discussed mechanisms. Even though a critical potential located in the anodic potential range may exclude hydrogen influenced mechanisms and appears to be related to anodic dissolution processes that remove atoms in a narrow front from the metallic lattice, it does not unequivocally support any of the alternative mechanisms. The relationship of this critical potential with the repassivation potential for localized corrosion is discussed. In this context, some directions for experimental and theoretical research are discussed, particularly for conditions associated with very slow crack growth rates which are becoming relevant for applications requiring extremely long periods of performance.

Computational Thermodynamics and Materials Design: Thermodynamic Modeling - I

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

Monday AM Room: 201
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Y. Austin Chang, University of Wisconsin-Madison, Mats. Sci. and Eng., Madison, WI USA

8:30 AM Opening Remarks: Zi-Kui Liu

8:35 AM Keynote

Computational Thermodynamics and Materials Design: *Larry Kaufman*[1]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., Cambridge, MA 02139 USA

Computer coupling of phase diagrams and thermochemical data have been performed for the 30 years (1-4). These methods have proven valuable in the development of new and improved materials for a wide range of applications. Recent extensions of the method to include diffusional and kinetic factors have provided a rational basis for simulating rates of reaction at temperatures where equilibrium cannot readily be achieved(4). Ab Initio calculations of the stability of pure solutions and compounds have been carried out for nearly fifty years. The accuracy of these methods have been improved to the point where it is common to see numerical values quoted with an accuracy of 1-10 mRyd/atom or(1300-13000 J/g-atom). A variety of computer based software has been developed and successfully applied for examining the relative Gibbs energies of competing phases that can exist over wide ranges of composition,temperature and pressure. A number of software packages are also available to calculate diffusional and kinetic behavior. One of the key ingredients in the CALPHAD-based predictive method is the relative stability and the heats of formation of stable and metastable structures which cannot or have not been measured directly. The latter are precisely the quantities that the Ab Initio calculations can provide. Illustration of cases where combination of the CALPHAD-based methods with the First-Principles techniques have been applied(5-6) will be discussed.

9:20 AM

Recent Developments of the Kaufman-Cohen Model of Martensite Nucleation: *Mats Hillert*[1]; [1]Royal Institute of Technology, Mats. Sci. and Eng., Stockholm 10044 Sweden

Larry Kaufman's interest in the thermodynamics of alloys, which finally led to the development of what is now called CALPHAD, started with his need to describe the thermodynamic properties of the Fe-Ni system when developing a theoretical model of the martensitic transformation together with Morris Cohen. Their model will be reviewed and it will be shown how it has influenced the nucleation theories for martensite ever since Larry Kaufman's thesis was presented in 1955. A recent attempt to revive some aspect of the original theory, that has been neglected during the later development of the theory, will be described. It concerns their hypothesis of a series of obstacles for nucleation instead of a single one.

9:50 AM

On the Choice of "Geometric" Thermodynamic Models: *Arthur Daniel Pelton*[1]; Patrice Chartrand[1]; [1]Ecole Polytechnique, Mats. Eng., P.O. Box 6079, Station 'Downtown', Montreal, Quebec H3C 3A7 Canada

Several "geometric" models have been proposed for estimating the thermodynamic properties of a ternary solution from optimized data for its binary subsystems. Among the most common are the Kohler, Muggianu, Kohler/Toop and Muggianu/Toop models. The latter two are "asymmetric" in that one component is singled out and treated differently, whereas the first two are "symmetric." It is shown that the use of a symmetric model when an asymmetric model is more appropriate can often give rise to large errors. Equations are proposed for extending the symmetric/asymmetric dichotomy into N-component systems for N greater than 3, while still permitting the flexibility to choose either a symmetric or an asymmetric model for any ternary subsystem. Finally, some arguments in favor of the Kohler model over the Muggianu model are presented.

10:20 AM Break

10:30 AM

On the Relation Between Solution Hardening Parameters and Thermodynamic Interaction Parameters: *Peter A. Miodownik*[1]; [1]University of Surrey, Mats. Sci. and Eng. Dept., Guildford, Surrey GU2 5XH UK

Solid solution hardening is often expressed primarily through the sum of parameters involving modulus and size differences with a relatively minor role attributed to chemical interactions. By contrast, current CALPHAD methods of characterising solid solutions tend to depend almost totally on extracting more accurate parameters via optimisation techniques which, while they integrate a variety of thermodynamic data, do not explicitly include the elastic and other allied physical properties. Data bases for the elastic, plastic and thermodynamic properties of solid solutions have therefore tended to evolve separately despite the fact that, in the early days of determining unknown thermodynamic interaction parameters, Kaufman devised a procedure which incorporated an elastic misfit parameter. Historically, this methodology ceased to be used to determine thermodynamic interaction parameters when it was realised that insufficient input data was available for the required combination of elements and structures, particularly where meta-stable structures were concerned. Since that time, extensive additional data has accumulated on the properties of solid solutions, including metastable structures, and this paper will re-examine the relationship of these various parameters in selected alloys of the transition metals.

11:00 AM

Combining Crystallographic and Thermodynamic Databases: *Suzana Gomes Fries*[1]; Hans Leo Lukas[2]; Bo Sundman[3]; [1]ACCESS e.V., RWTH-Aachen, Intzestrasse 5, Aachen D52072 Germany; [2]Max-Planck-Institut fuer Metallforschung and Institut fuer Nichtmetallische Anorganische Materialien, Heisenbergstr. 5, Stuttgart D-70569 Germany; [3]Royal Institute of Technology, Dept. of Mats. Sci. and Eng., Stockholm SE-10044 Sweden

The development of multicomponent thermodynamic databases brings to evidence the necessity of crystallographic information in order to identify and model the many phases present. The combination of crystallographic and thermodynamic databases not only helps to solve the problem of naming phases but can also help to define some areas where a more systematic research on phase solubility/stability is needed. To identify phases which have the same prototype and decide if they will form a solid solution or not is not a trivial problem, furthermore, to identify phases that althought the different crystal structure are anyway related buy some special symmetry path, is a challenge task. The initial steps of an extended project of merging this kind of information isdiscussed and reported.

11:20 AM

Thermodynamic Behavior of Inorganic and Organic Systems with Eight and More Components: *Michael Hoch*[1]; [1]University of Cincinnati, Dept. of Matls. Sci. and Eng., Cincinnati, OH 45221-0012 USA

We investigated the partial enthalpy of mixing in the eight component system (Ag-Bi-Cd-Ga-In-Pb-Sn-Zn), calculated the solubility of Anthracene in a seven component organic solvent and studied the behavior of a wax containing straight alkanes with 20 to 42 carbon atoms. In systems with 8 components we have 28 binary interaction parameters: thus uncertainty in one, two or three parameters affects the calculated values only slightly. In the inorganic system adding Ag to the seven component system changes the partial enthalpies at the center of gravity of the components slightly. In the solubility data one cannot differenciate between two significantly different interaction parameters. In the case of the wax, from the composition of the liquid and solid at various temperatures we could calculate the melting point (liquidus) temperature. On the practical side we recovered the noble metals (Ag, Au, Pt) from the jewellery industry scrap, which involved an eight component oxide system, to which other oxides were added to lower the melting point, the viscosity and the surface tension.

11:40 AM

Cluster Variation Method in the Computational Materials Science: *R. Kikuchi*[1]; K. Masuda-Jindo[2]; [1]University of California, Mats. Sci. and Min. Eng., Berkeley, CA 94720-1760 USA; [2]Tokyo Institute of Technology, Dept. of Mats. Sci. and Eng., Nagatsuta 4259, Midori-ku, Yokohama 226-8503 Japan

Cluster Variation Method (CVM) has been very successful in the computations of alloy phase diagrams as well as in many problems of the materials science related to the phase transitions. Originally, CVM was developed in the framework of the so-called rigid lattice approximation, but it has recently been extended to include continuous atomic displacements due to thermal lattice vibration and local atomic distortion due to size mismatch of the constituent atoms. In the present study, we focus our attention on the latter continuous displacement treatment of CVM. The continuous displacement (CD) formulation of the CVM is applied to study the phase stability of the binary alloys. The basic idea is to treat an atom which is displaced by r from its reference lattice point as a species designated by r. The effects of continuous atomic displacement on the thermodynamic quantities of binary alloy systems are investigated in detail. We also discuss the extension of the continuous displacement of CVM to the calculations of liquid phases of metals and alloys.

Cyanide: Social, Industrial, and Economic Aspects: Politics and Spills I

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

Monday AM — Room: 225
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Ray Beebe, Consultant, Tucson, AZ 85751 USA; Courtney Young, Montana Tech, Metallurgical and Materials Engineering, Butte, MT 59701 USA

8:30 AM Invited

Process Considerations Before and After Failure of the Omai Tailings Dam, August 19 to 24, 1995: *Robert R. Beebe*[1]; [1]Consultant, P.O. Box 32048, Tucson, AZ 85751 USA

On the night of August 19-20, 1995, seepage was seen at the toe of the Omai tailings dam in Guyana. The mill was immediately shut down, and within two hours the flow, then mostly water, was diverted into an inactive mining pit nearby. At about the same time a second more serious break occurred at a point where water and solids could flow directly into the Omai River. By the time a coffer dam could be completed on the early morning of August 24, some 4. 2 million cubic meters of tailings water had escaped from the pond, of which nearly 2.9 cubic meters had reached the Omai River. This paper outlines some initial process considerations and operational decisions which might have contributed to the dam's failure, how the Government of Guyana and the operator, Omai Gold Mines Limited, cooperated in dealing with immediate problems, and how the Government set about investigating the accident. In particular, the paper details the work of a Process Review Committee comprising Guyanese and foreign experts convened to assess the gold processing and effluent management systems at Omai, and to recommend actions where appropriate.

8:55 AM Invited

Public Image of Cyanide, Dams, and Baia Mare: *Fred W. DeVries*[1]; [1]Chem-Mining Consulting, Ltd., Chadds Ford, PA USA

Because of Australian ownership and management, the TV program "60 Minutes (Australia)" chose to report on the dam breach and cyanide spill at Esmeralda's Aurul operation, January 30, 2000, at Baia Mare, Romania. The author was invited to participate because of experience in cyanide, mining, and environmental matters. The opportunity for gathering technical background was severely limited by time constraints. However, some observations on the implications of this incident are presented before and after showing a tape of the actual telecast. Challenges resulting from the spill are significant for the entire gold-mining industry, in our world of increasing environmental activism.

9:25 AM Invited

Valley Leach Facility Design Considerations for Cyanide Containment: *Terry Mandziak*[1]; John Lupo[2]; [1]Anglogold Corporation, 100 N. 3rd St., Victor, CO 80863 USA; [2]Golder Associates, Inc., Denver, CO 80228 USA

Typical leach mine operations recover gold values through the application of a dilute cyanide solution onto ore within an engineered facility. Key to the success of mining operations is the containment and management of the cyanide solutions within the facility. Properly engineered facilities will have containment systems that are both protective of the environment while enhancing solution recovery. This paper not only discusses general containment standards, both identifies state-of-the-art containment and monitoring practices.

9:50 AM Invited

Interactions Between Cyanide-Heap Leach Solutions and Acid-rock Drainage: Implications for Remediation and the Potential Environmental Impacts of Cyanide from Summitville: *Geoffrey S. Plumlee, Ph. D.* [1]; [1]Crustal Imaging and Characterization Team, U.S. Geological Survey, MS935 Denver Federal Center, Denver, CO 80225 USA

The Summitville Au mine, Colorado, received tremendous publicity in the early 1990's for leaks of cyanide solutions from its heap leach facility. A real-time assessment of the leaks' environmental effects was not done. Plumlee et al (1995, Colo. Geol. Survey Spec. Pub. 38) did simple mixing experiments to model the influx of alkaline CN-heap leach solutions into a nearby stream, whose waters were highly acidic due to severe acid-rock drainage (ARD) from the mine. These experiments indicated WAD cyanide rapidly degraded due to formation of Fe-Cu-CN solids and HCN volatilization. The alkaline heap leach solutions also precipitated iron-hydroxides (iron supplied by the ARD), which then sorbed copper and other heavy metals from the ARD. Thus, mixing of cyanide-heap leach solutions with ARD may be a cost-effective remedial method at mine sites where treatment of both is required. We are initiating new experiments to further refine a potential remedial process.

10:15 AM Break

10:30 AM Invited

The Critical Importance of Strong Cyanocomplexes in the Remediation and Decommissioning of Cyanidation Heap Leach Operations: *Craig A. Johnson*[1]; David J. Grimes[1]; Reinhard W. Leinz[1]; George N. Breit[1]; Robert O. Rye[1]; [1]U. S. Geological Survey, Box 25046, MS 963, Denver, CO 80225 USA

We have examined cyanide behavior at several sites in the southwestern U.S. including 3 active heap leach circuits, 3 heaps undergoing rinsing, and 2 sites with cyanide contaminated groundwater. We have found that strong Co- and Fe-cyanocomplexes play an important role in cyanide behavior and can be critical to decommissioning, even where these complexes are not regulated explicitly. In active circuits, we find evidence for significant precipitation or adsorption of cyanocomplexes within the heaps. On rinsing, the release of the complexes is likely controlled by the kinetics of dissolution of, or desorption from, solids that have gone largely uncharacterized. In effluent, rapid photodissociation of strong cyanocomplexes can impede regulatory compliance by producing free cyanide in sunlight-exposed channels or in translucent sample bottles prior to chemical analysis. There is a critical need for additional information on Co-cyanocomplexes, including their importance in heap effluent, behavior in groundwater, and impact on the environment.

10:55 AM Invited

Cyanide Leaching, Interrupted: *Dan A. Mackie, P. Eng., President*[1]; [1]INNOVAT, Ltd, P.O. Box 61018, Oakville, Ontario L6J 7P5, Canada

Cyanide leaching operations have taken a beating from the press and the public at large for its seemingly haphazard approach to control of cyanide solutions and containment of tailings. Major spills in Colorado, Guyana, Spain, and more recently, Romania have highlighted what had already been perceived to be a bad scene. Citizen initiatives and legislative actions are increasing. Whether these indictments are justified or not will be not debated in this paper. What this paper will present is that the technology to avoid these disasters and future ones has been around for more than ten years but has been blatantly ignored by the industry, despite demonstrated cost effectiveness. It is time for miners to wake up and use emerging, instead of old, technologies.

11:25 AM Invited

The Pharmacology & Toxicology of Cyanide and Its Derivatives: *Steven I. Baskin*[1]; [1]United States Army Medical Research of Chemical Defense, Pharm. Div., Aberdeen Proving Grounds, MD 21010-5400 USA

Cyanide and its derivatives are utilized in many industrial applications including chemical intermediates, electroplating, and mining. It is also synthesized and found in plants and animals including man where it is thought to perform a variety of functions. A variety of enzymes are known to detoxify cyanide. These include: rhodanese, 3-mercaptopyruvate sulfur transferase, thiosulfate reductase, cystathionase and albumin. Cyanide appears to exert its toxic effect primarily by inhibiting cytochrome oxidase although other enzymes are inhibited. Therapeutic antagonist categories to treat cyanide include: methemoglobin formers such as nitrite, sulfane-sufur donors such as thiosulfate, metal salts or complexes such as cobalt and cyanohydrin-forming compounds such as dihydroxy acetone. Advantages and disadvantages of each category will be discussed. Newer compounds under development have been found that may allow for prophylactic therapy for cyanide poisoning with minimal side effects. In addition, co-oximetry de vices may provide real time measurement of cyanide in portable field situations.

Defect Properties and Mechanical Behavior of HCP Metals and Alloys: Phase Stability and Bulk Properties

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, Ames Laboratory, Iowa State University, Ames, IA 50011-3020 USA; Carlos N. Tome, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Monday AM — Room: 211
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Kai-Ming Ho, Ames Laboratory, Physics & Astronomy, Ames, IA 50011-3160 USA; Linda L. Horton, Oak Ridge National Laboratory, Met. and Ceram. Div., Oak Ridge, TN 37381-6132 USA

8:30 AM Keynote

Some Recent Developments in Understanding the Properties of the HCP Metals: *David J. Bacon*[1]; [1]University of Liverpool, Dept. of Eng., Brownlow Hill, Liverpool L69 3GH UK

The papers presented at the Adriatico Research Conference on 'Defects in Hexagonal-Close-Packed Metals' in 1990 (Philos. Mag. A 63 (1991) 821-1116) illustrated the importance of this class of material from both the fundamental and technological points of view. The New Orlean's meeting in 2001 is expected to be equally impressive and demonstrate that significant advances have been achieved in many aspects of the understanding of the properties and applications of the hexagonal metals. This introductory overview will touch on several of them, including: the properties of point defects, their production in radiation damage and the characteristic features of their clusters; the structure and properties of crystal dislocations; and the nature of interfacial defects and their role in boundary mobility.

9:10 AM Invited

First-Principles Studies of Phase Stability and Short-Range-Order in HCP Metallic Alloys: *Mark D. Asta*[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Rd., Evanston, IL USA

First-principles methods for computational modeling of phase stability in intermetallic alloys have advanced significantly in the past decade, with attention being directed primarily at cubic (fcc and bcc) materials. In this talk I will review briefly the computational approach, using results of a phase diagram calculation for the hcp-based Cd-Mg alloy system as an illustrative example. Results are presented of a first-principles study of phase stability and chemical short-range-order (SRO) in Ti-rich, Ti-Al alloys. The degree of SRO in these alloys is calculated as a function of temperature, and used to estimate theoretically the energy (γ_{SRO}) of a "diffuse anti-phase boundary" created by slip in short-range-ordered Ti(Al) alloys. The calculated values of γ_{SRO} are compared to very recent estimates based upon transmission-electron microscopy measurements by N. S. Thirumalai and M. J. Mills.

9:40 AM

Higher Order Elastic Constants and Generalised Gruneisen Parameters of Elastic Waves and Low Temperature Thermal Expansion of Rhenium: *George Varambinakam Mathew*[1]; C. Sukumara Menon[1]; [1]Mahatma Gandhi University, Schl. of Pure and Appl. Phys., Priyadarsini Hills, Kottayam, Kerala 686560 India

Rhenium crystallises in hexagonal close packed structure, and has an axial ratio of 1. 615 at 25° celsius, slightly less than the ideal value of 1.633. Among all the metals studied, it has the second highest bulk-modulus (KT=3603 kbar). One of the most interesting

findings in research on this metal is the anomalous (negative) pressure dependence of the superconducting transition temperature observed at low pressures. Rhenium is the strongest metal known at high pressure and is widely used as a gasket material in diamond anvil cell experiments. Here we report,the second order elastic constants, pressure derivatives of second order elastic constants, third order elastic constants,and the low temperature limit of thermal expansion of Rhenium. The calculated values of second order elastic constants and their pressure derivatives are also compared with the corresponding measured values. The agreement between the present values and the measured values is good.

10:00 AM
Effect of Pressure on Zone-Center Phonons in HCP Metals: *Helmut Olijnyk*[1]; Andrew Paul Jephcoat[1]; [1]University of Oxford, Dept. of Earth Scis., Oxford OX1 3PR UK

Knowledge of vibrational properties is not only essential in deriving elastic and thermal properties, but it also serves to elucidate the role phonons play in various types of phase transitions. We report on studies of several hcp metals by Raman scattering techniques in the diamond anvil cell for pressures up to 60 GPa. The investigated metals include the divalent metals Be, Mg, and Zn, the transition metals Y, Zr, Fe, Re and Ru, and some regular lanthanides. The observed pressure response of the transverse-optical zone-center phonon mode includes positive pressure shifts as well as anomalies like mode softening in connection with phase transitions (Zr, lanthanides). It is shown that the phonon frequencies and their pressure dependence are related to macroscopic elastic parameters. More general, these results show that the measurement of Raman-active phonons in metals provides a direct probe of bonding, and agreement with theoretical models gives additional confidence in ab initio techniques.

10:20 AM Break

10:40 AM Invited
Intergranular Stresses in Zircaloy-2: *Thomas M. Holden*[1]; Judy W. L. Pang[2]; Richard A. Holt[3]; [1]Los Alamos National Laboratory, LANSCE, Los Alamos, NM 87545 USA; [2]Manchester Materials Science Centre, Grosvenor St., Manchester M1 7HS England; [3]Atomic Energy of Canada, Ltd., Chalk River, Ontario K0J 1J0 Canada

The intergranular strains in Zircaloy-2 with rod texture were measured by neutron diffraction in-situ under uniaxial tension applied along the rod axis. The stresses along <a> and <c>-axes perpendicular to the rod axis were found to be 230 MPa and -241 MPa after 5% plastic deformation and the in-situ experiments show how these stresses develop. An elasto-plastic self-consistent model, EPSC, was used to simulate the experiments. Pyramidal and prismatic slip are included in the model. The introduction of plastic deformation which resembles basal slip, {0001}<1-210> considerably improves the agreement with experiment. To test this model the residual strains in Zircaloy-2 sheet were calculated with the same mechanical and thermal parameters, but sheet texture. The results were in agreement with experiment for sheet subject to 1.5% tension along the rolling direction but not for sheet subject to a 1.5% reduction in thickness by cold-rolling, possibly because twinning is not included.

11:10 AM
Neutron Diffraction Studies of the Deformation of Beryllium: *Donald W. Brown*[1]; [1]Los Alamos National Laboratory, LANSCE, MS H805, Bldg. 622, TA-53, Los Alamos, NM 87544 USA

We have studied the plastic deformation of beryllium in uniaxial tension and compression at room temperature and at 350ºC using the Time of Flight (TOF) neutron diffraction technique. Since TOF diffraction records the entire pattern simultaneously, the response of individual diffraction planes (hkil) and lattice parameters (a and c) to stress and temperature can be monitored. This provides a unique insight into intergranular stresses which develop during loading/heating. The neutron diffraction results represent the average response of grains in approximately 300 mm^3 of the gauge volume of the sample. This averaging corresponds well to the results of self-consistent polycrystal deformation models and comparisons may be readily made. Finally, we will be demonstrate how the results of the uniaxial loading measurements, in conjunction with spatially resolved neutron diffraction strain measurements, can provide qualitative information about the plastic history of a real engineering sample, specifically girth welded beryllium rings.

11:30 AM
An Empirical Potential for Ti Using the Modified Embedded Atom Method (MEAM): *S. G. Srinivasan*[1]; Dallas R. Trinkle[1]; M. I. Baskes[2]; R. C. Albers[1]; [1]Los Alamos National Laboratory, T-11 Group, MS:B262, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, MST-8, MS:G755, Los Alamos, NM 87545 USA

Large-scale atomistic simulations of technologically important hexagonal close-packed metals like Ti have been hampered by the nonavailability of computationally efficient and accurate descriptions of the complex angular force components in the interatomic interactions. A MEAM potential, a Modified empirical extension of the Embedded Atom Method (EAM), which explicitly includes angular forces, is developed for Ti using data from experiments and first-principles LAPW electronic-structure calculations. The MEAM potential describes well the basic properties of Ti, such as lattice constants, elastic constants, point-defect energetics, and phase stability. The reliability of this model is tested by simulating the alpha-to-beta and beta-to-omega martensitic phase transformations in Ti.

11:50 AM
Lattice Dynamics of HCP Metals with the Analytic EAM Potentials: *Wangyu Hu*[1]; Yangchen Huang[1]; Xiaolin Shu[1]; Bangwei Zhang[1]; [1]Hunan University, Dept. of Appl. Phy., Changsha, Hunan 410082 China

The analytic EAM many-body potentials constructed by us is used to study the phonon dispersion relations of hcp metals. Dispersion curves along [100], [001], and [110] symmetry directions of Be, Co, Hf, Mg, Re, Ru, Sc, Ti, Y, and Zr are calculated. In this method, the total energy of the system is functioned analytically as embedding, pair potential and modification energies, and the elastic constants are reproduced exactly as input data. The agreement between the calculated and the experimental data of the phonon frequencies is found to be as good as obtained by other authors in the past using different methods.

General Abstract Sessions: Mechanical Properties: Methods and Applications

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

Monday AM — Room: 202
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Donald Koss, Penn State University, 202A Steidle Building, University Park, PA 16802 USA; Pedro D. Peralta, Arizona State University, Dept. of Mech. and Aerospace Eng., Mail Code 6106, Tempe, AZ 85287-6106 USA

8:30 AM
Thermography Detection and Material Fatigue Damage: *Bing Yang*[1]; Peter K. Liaw[1]; Hsing Wang[2]; Liang Jiang[1]; Yuehui He[1]; Lijia Chen[1]; J. Y. Huang[3]; R. C. Kuo[2]; J. G. Huang[3]; R. R. Seeley[4]; D. L. Klarstrom[4]; [1]The University of Tennessee, Knoxville, Mats. Sci. and Eng. Dept., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; [3]Institute of Nuclear Energy Research (INER), P.O. Box 3-14, 1000 Wenhua Rd., Chiaan Village, Lungtan, Taiwan 325; [3]Taiwan Power Company, Taipei, Taiwan; [4]Haynes International, Inc., Kokomo, IN 46904 USA

An infrared (IR) thermography technique, as a nondestructive evaluation technique, was applied to investigate the fatigue damage of Reactor Pressure Vessel (RPV) Steels and HR 120 Alloys during low frequency and high frequency fatigue testing. The relationship between the temperature, stress-strain state, and fatigue behavior

of different material is discussed. Both thermodynamic and heat-transfer theories are applied to model the observed temperature variation during fatigue. Back calculation has been conducted from the observed temperature profiles to the stress-strain state of the material. The predicted and measured temperature evolutions and mechanical behavior during fatigue were found to be in good agreement. Thermography appears to provide a useful method of investigating the stress-strain behavior during fatigue.

8:55 AM

Hot Ductility Measurements on in situ Solidified Specimens Using an Improved Gleeble Machine Technique: *Daryoush Emadi*[1]; Elhachmi Essadiqi[1]; [1]CANMET, Mats. Techn. Lab., 568 Booth St., Ottawa, Ontario K1A 0G1 Canada

To prevent transverse cracking in steels during continuous casting, it is critical to characterize their hot ductility behaviour using high temperature tensile test. The hot ductility tests without melting do not reproduce the actual solidification microstructure of continuous casting steels and therefore, this phenomenon is not very well simulated. The Gleeble machine makes it possible to partially melt the sample and to reproduce the structure similar to that obtained in continuous casting. The major factors controlling the Gleeble test are explained. Various parameters, including sample preparation, thermocouple attachments, solidification shrinkage, strain rate and thermal profile, which can affect the results, are evaluated. Based upon the results of this study, an improved experimental procedure for isothermal Gleeble hot ductility test is proposed.

9:20 AM

An Experimental Model of Void Growth and Coalescence During Ductile Fracture: Christopher R. Colepietro[1]; Peter A. Kirkham[2]; *Donald A. Koss*[1]; [1]Penn State University, 202A Steidle Building, University Park,. PA 16802 USA; [2]Ryerson Tull, Inc., 2621 W. 15th Place, Chicago, IL 60540 USA

In order to predict ductile fracture on the basis of damage, the growth and coalescence of neighboring voids must be addressed. This study utilizes a novel specimen geometry to model the growth and interaction behavior among small groups of voids. Specifically, we rely on tensile specimens containing blind-end holes with hemispherical ends and measurements of the inter-hole thinning behavior. The key assumption is the stain-induced thinning of the inter-hole ligament is similar to that between neighboring microvoids during void growth and coalescence. Results from both Cu and Al alloy specimens containing 2, 3, or 4 blind-end holes indicate very pronounced strain-induced void interaction effects that are strongest for clusters of 3 holes spaced 2 hole diameters apart or less. For this case, hole coalescence also results in the characteristic 3-fold symmetry pattern often seen among the ridges on a dimpled ductile fracture surface. Effects of void spacing and strain hardening will be discussed.

9:45 AM

Surface Replication as a Means of Monitoring Fatigue Crack Initiation and Propagation in Ferrous Powder Metallurgy Alloys: *Steven J. Polasik*[1]; Nikhilesh Chawla[1]; K. S. Narasimhan[2]; [1]Arizona State University, Dept. of Chem. and Mats. Eng., Tempe, AZ 85287-6006 USA; [2]Hoeganaes Corporation., 1001 Taylors Lane, Cinnaminson, NJ 08077 USA

Ferrous powder metallurgy components must have excellent fatigue performance for a variety of applications. Thus, an understanding of the mechanisms of fatigue crack initiation and propagation in these materials is very important. By using surface replication techniques, the fatigue crack initiation and growth was investigated in powder metallurgy alloys. In particular, the influence of surface porosity on fatigue crack initiation and propagation was characterized by this technique. A detailed description of the surface replication technique will be provided. Characterization of the crack initiation and growth processes in these materials will also be described. Finally, the effect of pore size, mean pore spacing, and alloying additions on the fatigue behavior of the alloys will be discussed. Research supported by Hoeganaes Corporation, under contract #AC5-1022.

10:10 AM Break

10:20 AM

A TEM Investigation of Void Nucleation at Inclusions in AF1410 Steel: *Bala Ramalingam*[1]; Luana Iorio[1]; Warren M. Garrison[1]; [1]Carnegie Mellon University, Dept. of Mats. Sci. and Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Ductile fracture in high strength steels proceeds by the growth and coalescence of voids nucleated at second phase particles such as inclusions. In these steels, gettering the sulfur as small inclusions of titanium carbosulfide (Ti_2CS), instead of as manganese sulfide, chromium sulfide (CrS) or lanthanum oxysulfide, results in better fracture properties. For instance, recent work on AF1410 steel has yielded Charpy impact energies of 176J and 108J for heats containing Ti_2CS and CrS inclusions respectively. While the reasons for the superior fracture properties of Ti_2CS containing heats are not completely understood, it is believed that the dominant reason might be the Ti_2CS particles being more resistant to void nucleation than the other inclusion types. TEM methods are being used to investigate, and understand, the differences in void nucleation characteristics at CrS and Ti_2CS inclusions in AF1410 steel. Our observations from this work and their i mplications for the understanding and design of tough high strength steels will be presented.

10:45 AM

Fracture Toughness Anisotropy in Monocrystalline Molybdenum Disilicide: *Pedro D. Peralta*[1]; Mehdi Hakik[1]; [1]Arizona State University, Dept. of Mech. and Aeros. Eng., Mail Code 6106, Tempe, AZ 85287-6106 USA

The fracture behavior of monocrystalline MoSi2 has been studied using Vickers hardness and 3-point bending tests. The published results based on hardness suggest that (001) is the preferred crystallographic cleavage plane for this material and that the fracture toughness in this plane is lower than on the {100} and {110} planes. However, 3-point bending results reported in the literature indicated that the fracture toughness of MoSi2 in the (001) plane is higher than in (110). Experimental and analytical work has been carried out to find the reasons for the discrepancy between the two aforementioned studies. Three-point bending and Vickers hardness experiments were performed to duplicate literature results, and the differences between these experiments examined. It was found that the contradiction can be partly explained in terms of the effect of Mo5Si3 precipitates in the three-point bending experiments and crack tip shielding due to dislocation plasticity during hardness testing.

11:10 AM

The Influence of Non-isothermal Aging on the Mechanical Behavior of a Cast 319 Type Aluminum Alloy: *Shannon Christine Weakley*[1]; John Allison[2]; J. Wayne Jones[1]; [1]University of Michigan, Mats. Eng., 2300 Hayward St, H. H. Dow Bldg., Ann Arbor, MI 48109-2136 USA; [2]Ford Motor Company, Sci. Res. Lab., 2000 Rotunda Dr., Mail Drop 3182, Dearborn, MI 48124 USA

The aging response of aluminum alloys is traditionally studied using isothermal aging treatments. Commercial heat treatments of large cast aluminum components are, however, not isothermal; and the time required to heat these components to aging temperature can account for a significant portion of the total aging cycle. In this study, the influence of heat up time on aging response of a 319-type aluminum alloy (W319) has been characterized. Three different ramp rates to aging temperature were investigated: 5 minutes (near-isothermal), 45 minutes (intermediate), and 100 minutes (slow). It was found that the time to aging temperature had no significant impact on the material properties. In addition, the effect of an "interrupted" or partial quench between solution treatment and aging was examined. In commercial applications, the quench from solution treatment temperature to room temperature can be time consuming and costly. If this quench was "interrupted" by cooling to a temperature higher than room temp erature, the quench time could be cut in as much as half. However, this interrupted quench was shown to significantly decrease the hardness and yield strength of the W319 material.

11:35 AM

Properties of Salt-Grown Uranium Single Crystals: *Jason C. Cooley*[1]; Robert J. Hanrahan[1]; W. Larry Hults[1]; Dan J. Thoma[1];

James L. Smith[1]; Jason C. Lashley[2]; Charles H. Mielke[3]; Robert G. Clark[4]; A. R. Hamilton[4]; Jeremy L. O'Brien[4]; Eddie C. Gay[5]; N. E. Lumpkin[5]; C. C. McPheeters[5]; J. L. Willit[5]; George M. Schmiedeshoff[6]; Sharon Touton[6]; B. F. Woodfield[7]; B. E. Lang[7]; J. Boerio-Goates[7]; [1]Los Alamos National Laboratory, Mats. Sci. and Techn., Mail Stop G770, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Mats. Sci. and Techn., Mail Stop G730, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Mats. Sci. and Techn., Mail Stop E536. Los Alamos, NM 87545 USA; [4]University of New South Wales, Centre for Quantum Comp. Techn., Sydney 2052 Australia; [5]Argonne National Laboratory, CMT Div., Argonne, IL 60439 USA; [6]Occidental College, Dept. of Phys., Los Angeles, CA 90041 USA; [7]Brigham Young University, Dept. of Phys., Provo, UT USA

Recently single crystals of alpha-uranium were grown from a liquid salt. The electrical, magnetic and thermal properties of these crystals have been surveyed. The ratio of the room temperature resistivity to the saturation value at low temperature is three times larger than previously reported demonstrating that these crystals are of higher purity and quality than those in past work. The resistive signatures of the CDW transitions at 43, 37 and 22K are obvious to the naked eye. The transition at 22K exhibits temperature hysteresis that increases with magnetic field. The superconducting transition temperature from resistivity is 820mK and the critical field is 80mT. The Debye temperature from heat capacity measurements is 254K in good agreement with the predicted value of 250K. In time, measurements made on these uranium crystals may help to understand the origin of superconductivity and its relation to the CDW transitions.

General Abstract Sessions: Mechanical Properties C

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Monday AM — Room: 212
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Ozer Unal, Ames Laboratory, Metall. and Ceramics Program, Ames, IA 50011 USA; Joseph W. Newkirk, University of Missouri-Rolla, Rolla, MO 65409-0340 USA

8:30 AM
The Fracture Toughness and Toughening Mechanisms of Nickel-Base Wear Materials: *Brian V. Cockeram*[1]; [1]Bechtel Bettis Laboratory, ZAP 08D/MT, P.O. Box 79, 814 Pittsburgh-McKeesport Blvd., West Mifflin, PA 15122 USA

Nickel-base wear materials are typically used as weld hardfacing deposits, or cast or Hot Isostatically Pressed (HIP) inserts that provide the needed wear resistance to a base material with the desired mechanical properties. Most nickel-base wear materials contain high levels of chromium, silicon, carbon, and boron, which results in complex microstructures that are comprised of high volume fractions of silicide, carbide, and/or boride phases. The volume fraction of nickel-phase dendrite regions typically ranges from 40% to 70%, and these dendrite phase particles are individually isolated by a matrix of silicide, carbide, and boride phases. The continuous matrix of brittle silicide, carbide, and boride phases results in a low damage tolerance for nickel-base wear materials, which is a concern in applications that involve high stresses, thermal transients, or shock loading. Fatigue crack growth and fracture toughness (Kic) testing in accordance with ASTM E399 methods has been used to quantify the damage tolerance of various nickel-base wear materials. Fractographic and microstructure examinations were used to define a generic toughening mechanism for nickel-base wear materials. The toughness of nickel base wear materials is primarily controlled by plastic deformation of the nickel-phase dendrites in the wake of a crack moving through the matrix of brittle silicide, carbide, and/or boride phases, i.e. crack bridging. Measured Kic values are compared with calculated Kic values based on the crack bridging model. Microstructure examinations are used to confirm and define the important aspects of the crack-bridging toughening model. This model can be used to predict the toughness values of nickel-base wear materials, and direct processing methods to improve the Kic values.

8:55 AM
Prediction of the Core Structure of the 90° Partial Dislocation in Si: *Karin S. Lin*[1]; D. C. Chrzan[1]; [1]University of California, Dept. of Matls. Sci. and Eng., Berkeley, CA 94720 USA

The prediction of the atomic scale structure of a dislocation core using ab initio electronic structure total energy techniques is an area of active interest. For compact dislocation cores, it is a common practice to employ supercells containing a dislocation dipole and periodic boundary conditions. These boundary conditions, however, may impose unusual stress states upon the dislocation cores, and these stress states may influence the final predictions. This talk considers the influence of periodic boundary conditions on the prediction of dislocation properties. Specifically, it is demonstrated that one may use supercells and periodic boundary conditions to obtain a reasonable description of the properties of a dislocation. The energy difference between two competing core structures for the 90-degree partial dislocation in Si (as predicted by Tersoff potentials) is studied. It is demonstrated that the stable core structure of the dislocation may be altered by the application of hydrostatic pressure, and the application of shear stresses. This work is supported by the Office of Science, Basic Energy Science, U.S. Department of Energy.

9:20 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, Matls. Sci. and Eng. Dept., 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. 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.

9:45 AM Break

10:00 AM
Effects of Frequency and Specimen Self-Heating on The Fatigue Life of Type 316 Ln Stainless Steel: *Hongbo Tian*[1]; Peter K. Liaw[1]; D. Fielden[1]; Liang Jiang[1]; Bing Yang[1]; C. R. Brooks[1]; D. D. Bruns[1]; M. D. Brotherton[1]; Xin Wang[2]; J. P. Strizak[2]; L. K. Mansur[2]; J. R. DiStefano[2]; K. Farrell[2]; D. C. Lousteau[2]; S. J. Pawel[2]; G. T. Yahr[2]; [1]The University of Tennessee, Knoxville, Matls. Sci. and Eng. Dept., Knoxville, TN 37996 USA; [2]Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

Fatigue tests were performed at 0.2 Hz, 10 Hz, and 700 Hz on 316 LN stainless steel using both conventional and advanced high-frequency machines. The two main factors governing the influence of test frequency on the fatigue life: strain rate and temperature effects, were investigated. Phenomena for fatigue tests conducted in the mercury environment were also studied. The mechanism for the shorter fatigue lives at higher frequencies was proposed. The shorter fatigue lives at higher frequencies are attributed mainly to specimen heating. Fatigue tests at higher frequencies were carried out while cooling the specimen with liquid mercury or with nitrogen gas during experiments, which provides evidence to support the notion that specimen self-heating is the factor controlling the influence of test frequency on fatigue life. Modeling work on the temperature

distribution around the test specimen in both air and mercury is discussed.

10:25 AM

Hot Cracking Propagation along with Interdendritic Fluid Flow and Cohesion: *Eisaku Tokuchi*[1]; Kimioku Asai[1]; Shozaburo Ohta[1]; [1]Musashi Institute of Technology, Mech. Eng., 1-28-1 Tamazutumi, Setagaya-ku, Tokyo 158 Japan

The effect of interdendritic fluid flow and the liquid film cohesion on the hot cracking mechanism was first investigated. We examined those behaviors of Al alloy 5052 which have been mainly used in our series of research. TIG spot welding was performed in the center of its sheets of 1mm thickness, and the high-speed breaking test and the Varestraint test were carried out. The tests which were automatically controlled every 0.1 second in breaking time were followed by the fractography of the fractured sections with FE-SEM. The observation with the acceleration voltage of 1kV clearly showed the traces of the above behaviors. The results indicated that the crack would propagate toward the later stage of the solidification, be gradually difficult to propagate, and almost completely stop before the solid coheres the adjacent solid. All the data were obtained at Musashi Institute of Technology in 1994 and 1995 as a graduate program.

10:50 AM

Electromechanical Behavior of Open Cellular 7075-T6 Al: I. Nieves[1]; F. Arceo[1] ; *J. C. Earthman*[1]; [1]University of California, Dept. of Chem. & Biochem. Eng. and Matl. Sci., Irvine, CA 92697-2575 USA

The electrical resistance response during tensile deformation was investigated for an open-cellular 6101-T6 Al foam. Because of a slight asymmetry in the cell structure, both specimens with longitudinal cell orientation and those with transverse cell orientation were tested. Resistance was measured using a four-point potential drop technique that consists of periodic potential measurements during brief applications of constant current. An anomalous reduction in resistance was measured for all specimens during the initial stages of elastic deformation of the structure. This reduction was consistently measured regardless of the locations of the potential probes on the specimen or grips. An expected increase in resistance then followed for greater strains which primarily results from the failure of individual struts within the cellular structure. Possible explanations for the observed anomalous electromechanical behavior will be discussed.

11:15 AM

Effect of Silicon Content on the Strength and Toughness of a Martensitic Stainless Steel: Aytekin Hitit[1]; Warren M. Garrison, Jr.[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Martensitic stainless steels containing sufficient amounts of molybdenum and of cobalt exhibit significant age-hardening which is attributed to the precipitation of particles of R-phase. Ferritic stainless steels containing about 5wt.% molybdenum also exhibit pronounced age-hardening if they contain 2 to 4 wt.% silicon and this age-hardening is also attributed to the precipitation of particles of R-phase. For both systems the precipitates contains large amounts of iron, chromium and molybdenum but only small amounts of silicon or cobalt. These results suggest that silicon and cobalt play similar roles in the precipitation of R-phase. The objective of this work is to determine whether or not silicon additions can enhance precipitation strengthening in low carbon martensitic stainless steels modified by cobalt additions to achieve R-phase precipitation strengthening. To investigate this possibility, the effects of an addition of 2 wt.% silicon on the strength and toughness of a 0.005C/12Cr/12Co/5Mo/4.5Ni alloy have been investigated. Precipitation reactions in these two compositions have been studied.

General Abstract Sessions: Microstructures

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

Monday AM — Room: 230
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Steven P. Marsh, Naval Research Laboratory, Washington, DC 20375-5343 USA; Alan J. Ardell, University of California, Dept. of Mats. Sci. and Eng., Los Angeles, CA 90095-1595 USA

8:30 AM

Quantitative Analysis of Secondary Phases: *Janice Lynn Klansky*[1]; [1]Buehler Ltd., 41 Waukegan Rd., Lake Bluff, IL 60044 USA

When designing composites, it is important to consider the shape, size, amount and distribution of the reinforcing particles. Likewise, these are key considerations for alloy development of two-phase materials such as cast irons or aluminum-silicon alloys. This study evaluates the use of standard metallographic preparation and image analysis techniques to evaluate the morphology and distribution of secondary phases. It will focus on the use of traditional mathematical parameters used to examine two-dimensional cross sections of complex shapes.

8:55 AM

Decay Analysis of Coarsening Kinetics in Pb-Sn Alloys: *Steven P. Marsh*[1]; [1]Naval Research Laboratory, Code 6325, Washington, DC 20375-5343 USA

Phase coarsening is generally treated as a growth phenomenon in which the average domain size increases during isothermal aging. However, it is a relaxation process driven by a reduction in the excess interfacial energy of a two-phase structure. Analysis of coarsening as the temporal decay of the specific surface area of the microstructure permits accurate determination of coarsening rate constants. This regression approach relates directly to the governing physics, and it minimizes many experimental and theoretical sources of error that are present in conventional analyses based on growth kinetics of the average length scale. Rate constants obtained from micrographs of coarsening lead-tin alloys using this technique will be presented and compared with theoretical predictions. Advantages of this relaxation-based approach will also be discussed.

9:20 AM

Coarsening of Ni_3Ge Precipitates under Uniaxial Compression: Natalia V. Starostina[1]; *Alan J. Ardell*[1]; [1]University of California, Dept. of Mats. Sci. and Eng., 6531-G BH, Los Angeles, CA 90095-1595 USA

The effect of applied uniaxial compressive stress on the coarsening behavior of Ni_3Ge precipitates in a Ni-11. 6 at. % Ge alloy aged at 625°C is under investigation. The specimens are [001]-oriented monocrystalline cylinders aged for 48, 144 or 288 h under stresses ranging from 6 to 101 MPa; the deformation is primarily elastic over this range. The microstructures were examined by transmission electron microscopy. The applied stress causes a significant reduction in the coarsening kinetics, producing a decrease of ~20% or more in the average size at relatively low stresses (below 25 MPa) and retards coarsening gradually over the rest of the range. The applied stress tends to promote rounded interfaces as well as elongation along the cube directions, though the effect of stress is not dramatic. This research is supported by the Dept. of Energy.

9:45 AM

The Coarsening of Gamma in Gamma Prime (Ni_3Al): Jaykumar Joshi[2]; *Alan J. Ardell*[1]; [1]University of California, Dept. of Mats. Sci. and Eng., 6531-G BH, Los Angeles, CA 90095-1595 USA;

[2]University of California, Dept. of Mats. Sci. and Eng., 6531 BH, Los Angeles, CA 90095-1595 USA

We report initial results of an investigation of precipitation of the γ phase (Ni-rich solid solution) in a Ni_3Al matrix in alloys containing 22 to 23 at. % Al. The aging temperatures used range from 600 to 800°C. Microstructures were examined by TEM in [001]-oriented thin foils. The kinetics of precipitation of γ in Ni_3Al are much slower than in the reverse case; this is true for nucleation as well as coarsening. The γ precipitates are spherical when small, but become cuboidal in size as they grow. Unlike the reverse case, γ precipitates become plate-shaped at quite small sizes. We attribute this to the absence of anti-phase boundaries, which do not exist in γ precipitates, thereby eliminating an important factor impeding coalescence of closely spaced particles. This issue, as well as the kinetics of coarsening, will be discussed. This research is supported by the National Science Foundation.

10:10 AM Break

10:20 AM

Observation of the Dendritic Growth of Grain Boundary Precipitates in Ni-Cr-Fe Alloy: Jinsung Jang[1]; *Yong Bok Lee*[2]; Dokyol Lee[2]; Woo-Seog Ryu[1]; Il-Hiun Kuk[1]; [1]K.A.E.R.I, Nuclear Materials Technology Research Team, P.O. Box 105 Yusong, Taejon, Korea; [2]Korea University, Div. of Mats. Sci. & Eng., 5-1 Anam-dong, Sungbuk-gu, Seoul, Korea

The morphology of grain boundary precipitates in corrosion resistant Ni-Cr-Fe alloy was investigated with regard to crystallographic orientation. In contrast to the previous observations of the evolution of grain boundary carbides with heat treatment time, i. e. from fine discrete particles through a semi-continuous layer to large discrete particles, this study revealed that grain boundary carbides in this Ni-base alloy grow in a dendritic manner. The crystallographic characteristic of the dendrite precipitates were investigated by TEM (Transmission Electron Microscopy) and EBSD (Electron Backscattered Diffraction). The dendritic precipitates were revealed as M23C6 type carbides growing along the <110> direction on the {100} grain boundary plane, maintaining a cube-to-cube relationship with one adjacent grain, and thickening asymmetrically toward the incoherent interface within the grain boundary.

10:45 AM

Characterization of Micro Phase Properties in Ultra-fine Grained Steels Using Nanoindentation Technique: *Yeol Choi*[1]; Jong-Kyo Choi[2]; Wung Yong Choo[2]; Dongil Kwon[1]; [1]Seoul National University, Schl. of Mats. Sci. and Eng., San 56-1, Shinlim-Dong, Kwanak-Gu. Seoul 151-742 Korea; [2]Pohang Iron & Steel Co., Ltd., Pohang-shi, Kyungbuk, Korea

The nanoindentation technique was used to evaluate the mechanical properties of ultra-fine grained steels. Ultra-fine grained steels, whose ferrite grain sizes are reduced to 1~2 mm, are composed of various micro phase constituents such as ferrite, bainite and martensite. These single phases could not have been easily tested by conventional technique due to its small size until present time. An array of nanoindentation marks spaced sub-micro meter was made on the ultra-fine grained steel specimen. AFM was used for micro phase and grain boundary identification. The obtained hardness and elastic modulus values reflected the strength characteristics of each micro phase. These properties were compared to those from coarse grain steels before decreasing the grain size by hot-rolling procedure. Discontinuous loading curves were observed and could be analyzed in terms of inhomogeneous yielding behavior. The curve excursion loads were different near the grain boundary. These results relate to the dislocation emission and local dislocation distributions. Finally, overall mechanical properties of multiphase materials were predicted by evaluating the micro phase constituent properties. These values were compared with those values obtained from the micro Vickers hardness test and the tensile test.

11:10 AM

Design and Optimization of Hot Working Processes for Ti-6al-4v with Microstructural Control: *William G. Frazier*[1]; T. Seshacharyulu[1]; Steve C. Medeiros[1]; Y. V. R. K. Prasad[2]; [1]Air Force Research Laboratory, AFRL/MLMR, 2977 P St., Wright-Patterson AFB, OH 45433 USA; [2]Indian Institute of Science, Metall., Bangalore 560012 India

Among all titanium alloys, Ti-6Al-4V is the most widely used in aerospace applications due to attractive properties. An essential step in Ti-6Al-4V component manufacture is hot working which includes microstructural conversion from a lamellar structure to an equiaxed structure and finish forging in the alpha-beta phase field along with an appropriate heat treatment to obtain the desired final microstructure. The oxygen content in this alloy is varied from 0.13 wt% (ELI grade) to 0.20 wt% (regular grade) depending on whether the end use is toughness or strength critical. A detailed study has been conducted to understand the effect of oxygen (commercial vs. ELI) and starting microstructure (lamellar vs. equiaxed) on the hot working response over wide temperature and strain rate ranges, and processing maps have been developed by identifying the microstructural mechanisms. The process of globularization occurring at slow strain rates (<0.1 s^{-1}) is the most preferred for conversion and is bounded by th e prior beta cracking at lower temperatures and beta transus at high temperature. A regime of void nucleation, specific to ELI grade near the transus, has been identified which further restricts the higher temperature limit of conversion processing. Hot working of the equiaxed microstructure is best done in the alpha-beta superplasticity regime at slow strain rates (<0.01 s^{-1}) while working at higher strain rates results in a dynamically recovered microstructure. The usefulness of the mechanism maps in the design and optimization of industrial hot-working schedules without inducing microstructural defects will be discussed. As an example, optimization of a cogging sequence for an ELI grade will be presented in detail.

11:35 AM

Microstructure Development in Al-Based Amorphous Alloys with Pb: *Robert I. Wu*[1]; Zhenfu Dong[1]; John H. Perepezko[1]; [1]University of Wisconsin-Madison, Dept. of 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.

General Abstract Sessions: Microstructures/Brazing

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

Monday AM — Room: 214
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Xiang-Ming Cheng, University of Kentucky, Chem. and Mats. Eng., Lexington, KY 40506 USA; John J. Stephens, Sandia National Laboratories, Albuquerque, NM USA

8:30 AM

The Anisotropic Behavior of the Portevin-Le Chatelier Effect in Al-Mg Alloys: *Xiang-Ming Cheng*[1]; James G. Morris[1]; [1]University of Kentucky, Chem. and Mats. Eng., 177 Anderson Hall, Lexington, KY 40506 USA

The Portevin-Le Chatelier effect (PLC) was studied in Direct Chill Cast (DC) and Strip Cast (SC)Al-Mg alloys. The as received SC hot band shows a strong anisotropy of the intensity of the PLC effect with regard to the rolling direction (RD), transverse direction (TD) and 45 degrees from the rolling direction (QD). The magnitude of the stress drops of the serrations is largest for TD, smallest for RD, with QD in between but closer to TD. However, no such serration intensity difference exists between RD, QD and TD in DC hot band and soution treated SC hot band although it reappears after subsequest cold rolling. The elongated grains or effective grain size anisotropy is considered to play a major role in the serration anisotropy. In addition, anisotropic distribution of dislocations and solute atoms also appears to be important to the serration anisotropy. Thus, the combined anisotropic microstructures mentioned above are the main reason for the serration anisotropy.

8:55 AM

Effect of Mn on Pearlite Growth within the (γ+α+Fe_3C) Three Phase Field of the Fe-C-Mn Phase Diagram: *C. R. Hutchinson*[1]; R. E. Hackenburg[1]; G. J. Shiflet[1]; [1]University of Virginia, Dept. of Mats. Sci. and Eng., 116 Engineer's Way, Charlottesville, VA 22904 USA

It is a well known effect that substitutional alloying elements have a very strong effect on the formation of pearlite. An attempt at understanding this effect must begin with an examination of the role of the alloying elements in altering the Fe-C phase diagram. Additions of Mn to the Fe-C systems lowers the upper Ae1 and opens up an (γ+α+Fe_3C) three phase field. The transformation to pearlite within this three phase field (which is necessarily less than 100% pearlite at equilibrium) exhibits some very interesting characteristics, namely a non-constant growth rate and a continually changing γ/pearlite interfacial Mn concentration with time. Examination of the growth of pearlite within this three phase field is the topic of this investigation and will be compared with growth within the (α+Fe_3C) two phase field. Analytical transmission electron microscopy (ATEM) has been used to measure the interfacial Mn concentrations with time and an explanation for the unusu al solute profiles and growth kinetics observed is presented in the framework of the local equilibrium model. Support by the NSF-DMR is acknowledged.

9:20 AM

Effect of Faceting on Grooving of Polycrystalline Tungsten: *Pavlo Sachenko*[1]; Joachim H. Schneibel[2]; Wen Zhang[1]; [1]Oakland University, Mech. Eng., Rochester, MI 48309 USA; [2]Oak Ridge National Laboratory, Mets. and Ceram., P.O. Box 2008, Oak Ridge, TN 37831 USA

Grain-boundary grooving was studied on polished surfaces of polycrystalline tungsten annealed at 1350°C. Atomic force microscope images were taken in the same area for each groove after different annealing times. The presence or absence of faceting on adjacent grains strongly affected the grooving kinetics and groove shapes. The profiles of the grooves developed between unfaceted grains were in excellent agreement with predictions of Mullins theory of grooving by surface diffusion; both secondary maxima and minima next to the main groove maxima were regularly observed. Grooves forming between faceted and smooth grains showed unusual shapes and kinetics. Contrary to Mullins' assumption, the surface fluxes at the root of such grooves were found to be non-zero. Simulations based on the differential equation for surface diffusive mass transport are being carried out to understand this observation. This material is based upon work supported by the National Science Foundation under grant DMR-9996087. Research at the Oak Ridge National Laboratory SHaRE User Facility was sponsored by the Division of Materials Sciences and Engineering, U. S. Dept. of Energy, under contract DE-AC05-96OR22464 with UT-Battelle LLC.

9:45 AM

Microstructural Evolution in Non-Equilibrium Sputter Deposited $Cu_{1-x}W_x$ Alloys: *Wilbur L. Walters*[1]; G. M. Janowski[1]; J. M. Rigsbee[2]; [1]University of Alabama at Birmingham, Mats. and Mech. Eng., 1150 Tenth Ave. S., BEC Rm. 254, Birmingham, AL 35294-4461 USA; [2]North Carolina State University, Dept. of Mats. Sci. and Eng., Campus Box 7907, Raleigh, NC 27695-7907 USA

A series of non-equilibrium copper-tungsten ($Cu_{1-x}W_x$) alloys have been synthesized by dc magnetron sputter deposition using elemental Cu and W targets. Using Cu foil substrates, 15-20 micron (μm) thick films were deposited with compositions ranging from 0 to 5 atomic percent W. Evolution of as-deposited microstructures was determined by analyzing samples annealed at 900ºC for up to 100 hours. Scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) verified film composition and a columnar morphology for the sputter deposited films. X-ray diffraction showed the presence of bcc tungsten and fcc copper in the annealed films. Transmission electron microscopy (TEM) of annealed materials revealed a high density of discrete, nanoscale W particles dispersed within and along the grain boundaries of a Cu matrix. These results will be interpreted in terms of atomic misfit and W redistribution as a function of W content, particle size and particle spacing.

10:10 AM Break

10:20 AM

A Study of Zirconium as a Active Element for Brazing: *John J. Stephens*[1]; F. Michael Hosking[1]; Charles A. Walker[1]; Frederick G. Yost[2]; [1]Sandia National Laboratories, Joining and Net Shape Dept., P.O. Box 5800, MS0367, Albuquerque, NM 87185-0367 USA; [2]Trapezium Technology, 1901 Cleopatra Ct. NE, Albuquerque, NM 87112 USA

This study was motivated by the observation of Ti scavenging, and non-hermetic performance, in a commercial Ag-Cu-Ti active braze alloy when Kovar/alumina ceramic braze joints were evaluated. These observations motivated consideration of Zr as an alternative active element to Ti. A eutectic Ag-Cu alloy with a 2. 1 wt. % Zr addition as an active element has been found to produce hermetic Kovar/alumina ceramic braze joints, with minimal amounts of active element scavenging. Since ZrO2 is more stable than Al2O3 at temperatures in excess of ~650°C, a small amount of alumina is eroded from the surface, and a continuous reaction layer of ZrO2 is observed in hermetic joints. However, the "Zr-Cusil" alloy requires excellent atmosphere control to be successful, and a relatively high braze process temperature (~950°C) since the Zr is incorporated in the intermetallic compound Cu4AgZr. We have studied alternative means of delivering Zr to the ceramic interface during the braze operation, and will discuss the merits of these approaches. *Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. Dept. of Energy under Contract DE-AC04-94AL85000.

10:45 AM

Effects of Cu, Ag and Sb on the Creep-Rupture Strength and Thermal Fatigue Behavior of Lead-Free Solder Alloys: *Noboru Wade*[1]; Johji Kunii[2]; Seiji Yamada[1]; Kazuya Miyahara[3]; [1]Topy Industrial Limited, Tech. R&D Lab., Toyohashi 441-8510 Japan; [2]Nagoya University, Grad. Sch., Nagoya 464-8603 Japan; [3]Nagoya University, Dept. of Mole. Des. and Eng., Nagoya 464-8603 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 12N/mm2 and 353 to 403K, respectively. Thermal fatigue tests were conducted at a temperature range of 223 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.

11:10 AM

Influence of Thermal Expansion Mismatch on the Strength of Metal-Ceramic Brazed Joints: *Jocelyn L. Wiese*[1]; Thomas W. Eagar[1]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., 77 Massachusetts Ave., Cambridge, MA 02139 USA

The effect of thermal expansion coefficient (CTE) mismatch on the strength of metal-ceramic brazed joints is being investigated. Metal rods (Invar, Hastelloy B2, and Inconel 600) have been brazed inside alumina ceramic tubes using commercially available Ag-Cu-Ti brazing alloy. The shear strength of the concentric lap joints was measured. Two distinct types of failure mode were observed: duc-

tile failure in the braze alloy and reaction layers and brittle failure of the alumina tube. Preliminary results indicate that when the CTE of the metal is larger than that of the ceramic, ductle failure in the braze occurs, however when the CTE of the metal is smaller than that of the ceramic, brittle failure in the ceramic occurs. A finite element model is being used to further understand the role of CTE mismatch and geometric constraints on joint strength and fracture mode.

11:35 AM

Alumina Ceramic Surface Finish and Brazing Reactions: *Floyd Michael Hosking*[1]; Charles H. Cadden[2]; Ronald E. Loehman[1]; John J. Stephens[1]; Nancy Y. C. Yang[2]; [1]Sandia National Laboratories, Albuquerque, NM 87185 USA; [2]Sandia National Laboratories, Livermore, CA 94551 USA

Active brazing alloys (ABA's) are a relatively new class of filler metals that directly react with ceramics. Of particular interest to the process engineer is how the "active" constituents in these alloys, such as Ti, Zr, and V, affect wetting, capillary flow, and joint properties. Experimental evidence suggests that the metallurgical reaction with oxide ceramics is usually a simple reduction-oxidation product or a more complex spinel. The interfacial bond can be further enhanced by reactions with the glassy phase that binds the polycrystalline grains together. The presentation examines the effects of alumina grade, glassy phase, and surface modifications (silicon, silica, and mullite coatings; air firing) on interfacial reactions and joint strength. Understanding the fundamental role of ceramics composition and surface finish is necessary for controlling ABA reactions. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S. Dept. of Energy under Contract DE-AC04-94AL85000.

General Abstract Sessions: Ceramics and Intermetallics

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

Monday AM — Room: 228
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Brian V. Cockeram, Bechtel-Bettis, ZAP 08D/MT, P.O. Box 79, West Mifflin, PA 15122 USA; J. Daniel Whittenberger, NASA-Glenn Research Center, Mats. Div., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA

8:30 AM

Creep Mechanisms in Binary Titanium Aluminides: *E. Cerreta*[1]; S. Mahajan[1]; T. Pollock[2]; [1]Arizona State University, Chem. & Mats. Eng., P.O. Box 876006, Tempe, AZ 85287-6006 USA; [2]University of Michigan, Dept. of MSE, 2300 Hayward St., HH Dow 2041, Ann Arbor, MI 48109-2136 USA

The early stages of creep were investigated in three binary gamma based titanium aluminide alloys with nominal compositions of Ti 48, 50, and 52at% Al. Additionally, a second set of alloys, with slightly lower interstitial oxygen contents, was examined. Tests were conducted at 760°C and 200MPa and interrupted before and after the creep minimum was reached. The untested as well as the tested materials were studied by TEM to understand the substructural evolution in the early stages of the creep curve and to elucidate the role that oxygen content plays in the early stages of creep. Additionally tests were performed at 150 and 250 MPa to gain insight into the affects of stress on creep rate controlling mechanisms. From these observations conclusions about creep mechanisms are drawn and the effects of aluminum concentration, oxygen content, and stress on the creep response in single phase TiAl will be discussed.

8:55 AM

Effects of Addition Elements on Structures and Properties of TiAl Alloy: *Bo Young Hur*[1]; Yoon Hur[1]; [1]Gyeong Sang National University, Matls. Eng. Dept., Kajwa-dong 900, Chinju, Gyeongnam 660-701 Korea

The effects of Al content (44-54at. %Al) and ternary additions such as Mo, V and Si in as cast-based alloys made by plasma arc melting on solidification structures and mechanical properties were studied. Columnar /a2 lamellar structures in Al-lean alloys due to primary a solidification had higher room temperature(RT) fracture strength and strain than phase structures through the reaction of L +a in Al-rich alloys. The fraction of a2 phase was found to decrease with increasing Al content in binary alloys. Fractography revealed that fine translamellar fracture is a main fracture mode in Ti-48at. %Al alloy which led to a high fracture strain(>5%). The RT fracture strain was improved by the addition of 1.5 at. %Mo and 1 at. %Si. The mechanical properties have been discussed in term of changes in unit cell volume and axial ratio. In the case of Si, tensile properties coincided well with the change of axial ratio c/a. Again, the effects of Nb content (11-16at. %Nb) in as cast a2 based Ti-25at. %Al-Nb alloys made by plasma arc melting on structures and mechanical properties were also studied. Prior ß/B2 grain boundaries were found to increased and the size of a2 plates that resulted in finer a2-cleavage facet decreased with increasing Nb content, which, we speculate, caused higher fracture strain and strength in higher Nb content alloys.

9:20 AM

Deposition and Characterization of TiAlCr and TiAlCrN Coatings: *Feng Huang*[1]; John A. Barnard[1]; Mark L. Weaver[1]; [1]The University of Alabama, Metallur. & Mats. Eng., Box 870202, A129 Bevill, Tuscaloosa, AL 35487-0202 USA

TiAl-based alloys are gaining prominence in aerospace applications and beyond because of their attractive density-specific properties and oxidation resistance. To investigate the modification effect from chromium and nitrogen addition, TiAlCr and TiAlCrN coatings were prepared by dc magnetron sputtering of a TiAlCr target in both pure Ar and Ar+N2 plasma. In this paper, investigation of the growth of TiAlCr-based coatings will be presented first, and is followed by structural and mechanical characterization of the coatings as a function of deposition parameters and heat treatment. It is found that the structure of as-deposited coatings is insensitive to deposition parameters while mechanical properties such as hardness and modulus are a function of the deposition. The development of coating's structure and mechanical properties after various heat treatments is presented.

9:45 AM

Creep and Toughness of Cryomilled NiAl Containing Cr: *J. Daniel Whittenberger*[1]; Beverely Aikin[2]; Jonathan A. Salem[3]; [1]NASA-Glenn Research Center, Mats. Div., MS 24-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [2]Case Western Reserve University at NASA-Glenn Research Center, Mats. Div., 21000 Brookpark Rd., Cleveland, OH 44135 USA; [3]NASA-Glenn Research Center, Structures Div., 21000 Brookpark Rd., Cleveland, OH 44135 USA

NiAl-AlN+Cr composites were produced by blending cryomilled NiAl powder with ~ 10 vol % Cr flakes in order to improve the room temperature toughness without severely reducing the elevated temperature strength. NiAl-7. 4 vol % AlN w/o Cr were consolidated by both hot isostatic pressing (HIP) and hot extrusion, while NiAl-12. 4 vol % ALN w/o Cr were densified by HIP. In comparison to the as-consolidated matrices, neither HIP'ed Cr-modified material demonstrated a significant improvement in toughness over that of NiAl-AlN. Hot extruded NiAl-AlN+10. 5Cr, however, possessed a toughness twice that determine for the base NiAl-AlN alloy. Measurement of the 1200 to 1400K plastic flow properties revealed that the strength of the composites was completely controlled by the properties of the NiAl-AlN matrices. This behavior could be successfully modeled by the Rule-of-Mixtures, where load is shed from the weak Cr to the strong matrix.

10:10 AM Break

10:20 AM

Corrosion Behavior of Fe3Al in Environments Containing Chlorine and Oxygen: *Weol D. Cho*[1]; Gilsoo Han[1]; [1]University of Utah, Metallur. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112 USA

The corrosion behavior of iron aluminides in environments containing chlorine and oxygen has been investigated at the temperature range of 600-800ºC using thermogravimetric analysis and various analytical tools including SEM, XRD and EDS. The rate of corrosion of the aluminides has been obtained as a function of gas chemistry, temperature and gas flowrate. Based on the kinetic results from thermogravimetric measurements and microstructure studies, the corrosion mechanism has been elucidated. The interaction between alumina developed on the iron aluminides and chlorine is also studied.

10:45 AM

Fabrication of Porous Sintered α-Alumina with Uniform Pore Size Distribution: *Tetsu Umeda*[1]; Yoshio Uchida[1]; [1]Sumitomo Chemical Co. Ltd., Tsukuba Res. Lab., Kitahara 6, Tsukuba-City, Ibaraki-ken Japan

Porous sintered α-alumina with uniform pore size distribution is valuable in metallurgical field for molten metal filtration. It has been developed using Sumicorundum®, which is single crystal α-alumina powder with narrow particle size distribution and high purity. Green bodies were prepared by slip-casting or uniaxial pressing, then calcined at 1100-1500°C and they changed into porous α-alumina sintered bodies. Total through pore size distribution and pore capacity of porous bodies were measured and found they have very narrow through pore size distribution and good permeation properties. Pore size of sintered bodies could be controlled by particle size of starting alumina powder. Compression strength of sintered bodies could be enlarged by adding a small amount of alumina sol as sintering agent. Porous sintered α-alumina also had pore size stability against high temperature, these properties are suitable for molten metal filters.

11:10 AM

The Fracture Strength of Plate and Tubular Forms of Monolithic Silicon Carbide (SiC): *Brian V. Cockeram*[1]; Jim L. Hollenbeck[1]; [1]Bechtel-Bettis, ZAP 08D/MT, P.O. Box 79, West Mifflin, PA 15122 USA

The fracture strength of Silicon Carbide (SiC) plate deposits produced by Chemical Vapor Deposition (CVD) was determined from room-temperature to 1500°C using a standard 4-point flexural test method (ASTM C1161). Two different types of CVD SiC material are shown to have slightly different flexural strength values, which appear to result from differences in microstructure. Although CVD deposition of SiC results in a textured grain structure, the flexural strength was shown to be independent of the CVD growth direction. The orientation of machining marks was shown to have the most significant influence of flexural strength, as expected. The fracture strength of tubular forms of CVD SiC produced by machining from a plate deposit are compared with tubular products produced by CVD deposition on a mandrel. The differences in microstructure between the plate deposits and CVD deposits made on a mandrel, and the influence of microstructure on fracture strength is discussed.

11:35 AM

Analytical Method and Optimization of Metallurgical Processes Based on the Colored Phase Diagrams: *O. O. Rodnov*[1]; P. V. Polykov[2]; P. D. Stont[2]; A. I. Berezin[2]; S. S. Gorjaev[3]; [1]Krasnoyarsk Non-Ferrous Metals and Gold Academy 95, Krasnoyarsky Rabochy St., Krasnoyarsk 660025 Russia; [2]Toks-Soft-Light Metals, Ltd., Russia; [3]Toks-Soft-Sibiria, Ltd., Russia

There are known algorithms and programs which realize methods of phase diagrams property-composition based on the condition that sum of three parameters is 100%. These phase diagrams can be built only for the quarterly systems, using specially planned experiments. These programs are applied widely in metallurgic and chemistry where dosage of the mixture components should be planned. Algorithms and program of their realizations, which permit to build colored phase diagrams made on the base of passive experiments (from statistics), have been developed. This property of diagram opens principally new possibility to compact large amount of statistical date into the form of colored phase diagrams very convenient for practical use. In particularity for aluminium production this system permits to receive dependencies of the technical and economical parameters on the technological conditions. Proposed method offers for the technical manager's very good tool for analyses of the practical situation and the searching of the optimal technological solutions. This method can be applied in the Decision Support Systems.

General Abstract Sessions: Thin Films, Granulation, and Aluminum

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

Monday AM — Room: 213
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Lee Eeles, Australian Greenhouse Office, Greenhouse Challenge, Canberra, Australian Capital Territory 2601 Australia; Ian J. Barker, Mintek, Measure. and Cont. Div., Private Bag X3015, Randburg 2125 South Africa

8:30 AM

Oxidation Resistance of Ultrathin TiB2 Films for the Protection of Magnetic Underlayers: *Feng Huang*[1]; William S. Epling[2]; John A. Barnard[1]; Mark L. Weaver[1]; [1]The University of Alabama, Metallur. & Mats. Eng., Box 870202, A129 Bevill, Tuscaloosa, AL 35487-0202 USA; [2]The University of Alabama, Chem. Eng., Box 870203, A127 Bevill, Tuscaloosa, AL 35487-0203 USA

Ultrathin titanium diboride (TiB_2) films are receiving increased attention as potential protective overcoats for high-density magnetic storage media. This is due to its high hardness, chemical inertness and good thermal stability. This paper focuses on the oxidation resistance of ultrathin TiB_2 overcoats. Multilayer films consisting of an ultrathin TiB_2 overcoat (<100 nm) and a 100 nm Co interlayer were deposited on Si substrates via magnetron sputtering. XPS and XRR techniques were then utilized to characterize the surface oxide layer. XPS results indicate that a 5 nm TiB_2 film is sufficient to protect the underlying regions from oxidation if annealed in air to 200°C. At higher temperatures, thicker films are required, however, even a 10 nm coating still protected the Co layer during annealing at 400°C.

8:55 AM

Assessment of Thin Film Residual Stress Using Contact Stress Analysis and Indent Morphology: *Yun-Hee Lee*[1]; Yeol Choi[1]; Eun-chae Jeon[1]; Dongil Kwon[1]; [1]Seoul National University, Schl. of Mats. Sci. and Eng., San 56-1, Shinrim-Dong, Kwanak-Gu, Seoul 151-742 Korea

Residual stress reduces the mechanical reliability of various thin film materials by decreasing interfacial adhesion and strength. Thus, the researches for finding the formation mechanism and the strength of residual stress are very important. However, conventional measuring methods are limited in regard to both specimen preparation and in the analyses of local properties. Therefore, we proposed a new method to evaluate the residual stress using nanoindentation technique and indent morphology. First, we superposed the elastic/plastic indentation stress fields with the elastic residual stress field. Anisotropic plastic deformation around the indent by the effect of the residual stress was modeled using the change in yield condition. Concrete values of the size and shape of the plastic zone around the nano-contact were obtained using the atomic force microscopy. The quantitative value of residual stress was calculated by inputting these values into the proposed modeling equation. The residual stresses of hard D LC coating on Si wafer were evaluated using this method. Finally, the value of residual stress from nanoindentation analysis was compared with the value obtained from the conventional curvature method.

9:20 AM

A Study of Ramped and Constant Loading Nanoscratch Techniques Utilized to Characterize the Nanotribology of Chromium Thin Films: *G. Wei*[1]; T. W. Scharf[1]; J. N. Zhou[1]; J. A. Barnard[1]; [1]The University of Alabama, Dept. of Metall. and Mats.

Eng. and Ctr. for Mats. for Info. Tech., Tuscaloosa, AL 35487-0202 USA

This paper presents a study of ramped and constant loading nanoscratch techniques applied to Cr thin films. Constant and ramped loading scratches were carried out using a Nano Indenter II system at various loads (1mN, 2. 5mN and 5mN). Cr thin films were deposited by DC magnetron sputtering. The load dependencies of the displacement, residual wear depths, percent elastic recovery, and friction coefficient in constant load and ramped load tests are compared. The scratch wear tracks were viewed by scanning electron microscopy (SEM) and atomic force microscopy (AFM). AFM was also used to quantify the scratch profile in cross-section. Under the same (maximum) load, constant load tests exhibit higher displacements, residual depths and friction coefficients but lower percent elastic recoveries. The mechanism of the displacement difference between ramped and constant scratch has been analyzed. A series of experiments have been performed to assess the analyses.

9:45 AM

Surface Tension Effects in Molten Metal Granulation: *Ian J. Barker*[1]; [1]Mintek, Measure. and Cont. Div., Private Bag X3015, Randburg 2125 South Africa

Recent technical developments have provided a new way to estimate the surface tension of granules during the process of granulation. Examples will be given in the full paper of various materials and their surface tensions during granulation in water. These values of the surface tensions are relatively low and are more typical of the values reported in the literature for oxides than those for metals. This indicates that the surface of such a granule is behaving as an oxide, even though the interior of the granule itself is a metal. This finding has a number of implications, and these will be discussed in more detail in the final paper.

10:10 AM Break

10:25 AM

The Aluminium Industry, a Greenhouse Challenge, the Australian Experience: *Lee Eeles*[1]; [1]Australian Greenhouse Office, Greenhouse Challenge, GPO Box 621, Canberra, Australian Capital Territory 2601 Australia

To meet its international commitments and address the issue of climate change, the Australian Government has established the Australian Greenhouse Office as its lead agency on climate change. The Government has developed a National Greenhouse Strategy and has committed almost $1 billion to a range of greenhouse gas abatement and energy efficiency programs. Key programs include the development of environmental management strategies for HFs, FPCs and SF6, and the Greenhouse Challenge-a voluntary, self-regulatory program. The Australian aluminium industry is a Greenhouse Challenge participant. The industry has been at the forefront of research into controlling anode effects and reducing emissions of PFCs that occur in the smelting process. Since 1990, emissions of PFCs have declined by more than 70 per cent. Future emissions reductions are likely to be in energy supply and use.

10:50 AM

Opportunities for Aluminum R&D and Best Practices Partnerships: *Thomas P. Robinson*[1]; Sara Dillich[1]; William T. Choate[2]; [1]U.S. Department of Energy, Ofc. of Indust. Techns., 1000 Independence Ave. SW, Washington, DC 20585-0121 USA; [2]BCS, Inc., Ste. 306, 5550 Sterrett Place, Columbia, MD 21044 USA

The Aluminum Industry Technology roadmap published, May 1997 was a bold technology planning initiative developed by aluminum industry companies, the Aluminum Association and the Dept. of Energy-Office of Industrial Technologies (DOE-OIT). Since its publication, DOE-OIT has been a partner to more than seventy firms in over thirty Research and Development projects that address priorities identified in the Road map. DOE-OIT will present an overview of its Aluminum R&D portfolio. The overview will cover the technical progress, expected benefits, demonstration status and market projections for the portfolio's core R&D projects. It will emphasize the energy savings and environmental reduction profiles of the portfolio. The authors will also describe DOE-OIT's extended portfolio of relevant projects and other DOE-OIT programs and activities that benefit industry including: BestPractices (training, plant audits and assessments, and technology implementation facilitation), state level Industries of the Future activities, and technology showcase events.

11:15 AM

Smelting Reduction of Red Mud for the Recovery of Iron and Titania Slag/Metal Equilibrium Studies: Srikanth Srinivasan[2]; Amitava Bandopadhyaya[3]; Thomas C. Alex[3]; *Animesh Jha*[1]; [1]University of Leeds, Dept. of Matls., 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. Process. 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 1400-1600°C. 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.

General Recycling: Topics Related to Light Metals and Aluminum Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee
Program Organizer: Guy Fredrickson, Hazen Research, Thermal Processing Group, Golden, CO 80403 USA

Monday AM — Room: 219
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Guy Lawrence Fredrickson, Hazen Research, Thermal Processing Group, Golden, CO 80403 USA

8:30 AM Opening Remarks

8:35 AM

Some Insights into Loss of Aluminum During its Recovery fron Salt-Cake: Benjamin W. Rockwell[1]; Ray D. Peterson[2]; John P. Hager[1]; *Gerard P. Martins*[1]; [1]Colorado School of Mines, Metallur. and Materials Eng., Golden, CO 80401 USA; [2]IMCO Recycling Inc., P.O. Box 268, 397 Black Hollow Rd., Rockwood, TN 37854 USA

Secondary production of aluminum from scrap has achieved prominent status both in the U.S. and worldwide. The primary incentive being the overwhelming savings in costs offered by this route. Saltcake and black-dross byproducts generated by reverbatory and rotary-barrel furnaces, employed for this scrap-recycling purpose, contain significantly high aluminum metal-content–3 to 5% and 10 to 20%, respectively. These byproducts have been disposed into landfills (primarily in the U.S.). Restrictions placed on landfill practices in several European countries have fostered the development of processes for the treatment of these materials–the recovery of the chloride salts and the aluminum metal being the primary objectives. The paper to be presented reports on the dissolution of pure aluminum and a 3004 alloy, in a 0. 5 molal chloride electrolyte. The intrinsic-rate measured has been extrapolated to forecast retention times associated with a range of (spherical) Al particles of sizes, 50 to 5000 μm, for 100% and 10% loss, at 22°C and 60°C.

9:00 AM

Magnus Separation of Dross and Image Analysis During Separation of Non-Ferrous Metal Scrap: *Gerrit H. Nijhof*[1]; Peter C. Rem[2]; E. A. Schokker[2]; [1]Nijhof Consultancy, Heemsteedse Dreef 92, 2101 KN Heemstede, The Netherlands; [2]Delft University of Technology, Mijnbouwstraat 120, 2638 RX Delft, The Netherlands

Results will be presented of the ongoing research on separation technologies as follow up on the presentations on previous Light Metals Conferences. The work on the image analysis of materials separated by the Eddy Current is finalized. Directives for commercial applications are given. The technique is suitable for shredder scrap but not for household refuse. Details will be discussed. The Magnus separation technique is further developed. Experiments have been performed on the separatin of aluminum and aluminumoxide from dross. After milling, the main problem, the particles are sieved in three fractions: 0.2 to 0.8 mm, 0.8 to 1.6 mm, and 1.6 to 3.0 mm. Results of the separation trials will be presented. Separation with a high throughput is possible.

9:25 AM

Technical Progress in the Aluminium Industry–A Scenario Approach: Bernd Friedrich[1]; Joachim Krüger[1]; Wilhelm Kuckshinrichs[2]; *Georg Rombach*[1]; Petra Zapp[2]; [1]RWTH Aachen, IME Dept. of Nonfer. Proc. Metall., Intzestr. 3, Aachen 52066 Germany; [2]Juelich Research Center, STE Programme Group Systems Analysis and Technology Evaluation, Juelich 52425 Germany

Analysis and modeling of material flows in complex production systems are appropriate instruments to show existing potentials for an efficient use of resources following the idea of sustainable development. Using scenario techniques significant future developments of aluminium production, manufacturing and use can be evaluated. This article focuses on technological progress along the material flow of aluminium from mining, smelting, to recycling and disposal. For this a technology-orientated process chain model has been developed. As an example the German packaging industry and its special recycling concept, including material and energy supply and transport has been chosen. The 1997 basis scenario is compared with a calculation considering newest technologies known today and a further one with regard to their possible application in 2010. The results help to identify technical potentials in different process steps of packaging life cycle and to analyse their impacts on the environment.

9:50 AM Break

10:10 AM

Recycling Activities for Aluminium Packaging under the Focus of the Preparation for the Re-Melting Process: *Stefan Mutz*[1]; Jan Meier-Kortwig[1]; [1]RWTH Aachen, Chair for Proc. und Recy. of Solid Waste Mat., Wuellnerstrasse 2, Aachen 52064 Germany

Aluminium is often regarded as a material which is very suitable for recycling e. g. due to large energy savings compared to primary production. When recycling of aluminium is discussed the focus point is mainly in the area of re-melting and refining. Nevertheless, the processing of scrap material prior to its reuse in secondary smelters becomes more and more important as aluminium and its alloys are typically used in combination with other materials. Within this paper the most important recycling activities taking place in the field of aluminium packaging are presented. First the different possible input materials are described under the aspect of raw material properties e.g. metal content, average peace size, impurities, etc. Then an overview of the different technical processes of recycling of aluminium is given within some examples. For measuring the success of such recycling activities there are some tools used like metal yield, moisture, organic content, piece size, etc. Finally the technical possibilities in preparing the input materials to get better results in the re-melting process are shown.

10:35 AM

Removal of Magnesium from Recycled Aluminum by Gaseous Chlorine Injection into Liquid Metal: Estéfano Aparecido Vieira[1]; *Jorge Alberto Soares Tenório*[1]; [1]University of São Paulo, Metall. and Mats. Dept., Av. Prof. Mello Moraes, 2463, Cidade Universitári-Butantã, São Paulo, SP 05508-900 Brazil

Due its chemical composition, UBCs are not easily accepted for other applications besides aluminum cans' production. This restriction is mainly related to Manganese and Magnesium concentrations. Each can is composed of AA3004 and AA5182 alloy, representing 75% and 25% of the entire mass, respectively. In the aluminum industry, chlorine injection have been used extensively with many purposes such as hydrogen reduction, alkaline metals removal and improvement of the molten metal cleaning. Chlorine was injected vertically into a bath containing liquid aluminum by a porous plug and nozzle to produce magnesium chloride under laminar flow conditions in a laboratory device. The use of Chlorine is viable to remove Magnesium. The tests showed several efficiencies. The yield can be controlled through bubbles size, so it is possible to avoid aluminum chloride emissions.

11:00 AM

Treatment of Industrial Waste Material in a Reverberatory Furnace at the Onahama Smelter: *Shigeru Ishikawa*[1]; Nobuo Kikumoto[1]; Kiyotaka Abe[1]; Michio Nishiwaki[1]; [1]Onahama Smelting & Refining Co., Ltd, Smelter Dept., 1-1 Nagisa Onahama Iwaki, Fukushima 971-8101 Japan

Recently, the treatment of industrial waste materials has been a serious problem in Japan. In these conditions, Onahama Smelter has begun to treat automobile shredder dusts (SD) in reverberatory furnaces without the generation of dioxin since 1995. Onahama Smelter has been planning to treat about 20% of SD generated in Japan (total one million metric tonnes per year) by using tonnage oxygen, and simultaneously to replace coal with SD as fuel at everberatory furnaces. At 2000 TMS Annual Meeting, the plan of expansion of SD consumption was outlined. Modifications of the reverberatory furnaces, storage and transportation system of SD and new oxygen plant are now under construction. They will not be completed until November 2000, and then regular treatment of SD and the replacing the coal with SD will be started. In this session, recent operations will be presented.

International Symposium on Deformation and Microstructure in Intermetallics: Deformation

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

Monday AM — Room: 220
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Peter M. Hazzledine, UES, Inc., AFRL/MLLM Bldg. 655, 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA; Christopher Woodward, UES, Inc., Matls. and Process. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

8:30 AM Invited

Strain Compatibility and Stress-Strain Relationships for Lamellar Gamma Titanium Aluminides: *Dennis M. Dimiduk*[1]; Peter M. Hazzledine[2]; T. A. Parthasarathy[2]; [1]Air Force Research Laboratory, Mats. and Manuf. Direc., AFRL/MLLM Bldg. 655, 2230 Tenth St., Wright-Patterson, AFB, OH 45433-7817 USA; [2]UES, Inc., AFRL/MLLM Bldg. 655, 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA

Lamellar Ti-Al alloys are plastically inhomogeneous and exhibit anisotropic flow. The origin of this behavior is that there are at least four different length scales in the microstructure: the grain size, the domain size, the lamellar thickness and the separation between either dislocations or twins. They range from mm to nm and they give rise to strain incompatibilities and internal stresses over a similar range of lengths. Traditional engineering finite element analysis of plastic deformation ignores all microstructural length-dependent aspects of the deformation but uses instead constitutive equations to describe plasticity. Such an approach may succeed in homoge-

neous, isotropic materials but it is unlikely to be accurate in Ti-Al. The gap between the scientific and engineering analyses of plasticity may be bridged, on average, by using Ashby's strain-gradient arguments. These capture most of the microstructural scale effects and may, with further development, deliver descriptions of plasticity which are capable of being used in FEM simulations. In this study, strain gradient arguments are used to interpret experimental stress-strain measurements of both PST and polycrystallineTi-Al.

9:00 AM Invited

Use of Weibull Statistics to Quantify Variability in TiAl Alloys: Nicholas Biery[2]; Rafael Raban[2]; Andrew Elliott[2]; Marc De Graef[2]; *Tresa M. Pollock*[1]; [1]University of Michigan, MSE Dept., 2300 Hayward St., HH Dow 2042, Ann Arbor, MI 48109 USA; [2]Carnegie Mellon University, MSE Dept., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

In recent years it has become apparent that intermetallics can effectively function in critical structural applications if they possess some degree of tensile ductility at low and intermediate temperatures. Cast gamma titanium aluminides are a good example of this. Unfortunately, for a given composition, these materials often suffer from a high degree of variability in tensile properties. In this study, variability in the tensile properties of cast gamma titanium aluminides has been quantified with the use of Weibull analyses. Tensile experiments have been performed on cylindrical specimens of several TiAl based alloys, including Ti-47.9Al-2Cr-2Nb and Ti-47Al-2Cr-2Nb alloys with two different cooling rates during casting, a Ti-47Al-2Cr-1Nb-2Ta alloy and a Ti-45Al-1.3Mn-2Nb-0.5B alloy. In all materials, failures were "intrinsic" in character, typically initiating within a grain or group of grains subject to high levels of local straining. Weibull moduli (based on a two parameter fit) were typically a factor of 2 to 3 higher than engineering ceramics such as silicon nitride, silicon carbide or PSZ. Use of the three parameter form of the Weibull relation, with the yield strength as the minimum failure strength, is most effective for quantification of variability and scaling properties with volume. Weibull moduli are similar for alloys with similar grain sizes, independent of composition, over the range of alloys investigated. Large variations in lamellar volume fraction, segregation and phase distribution influence variability much less than changes in grain size that arise from variations in cooling rates during casting or from the addition of grain nucleants.

9:30 AM Invited

Micromechanical Mechanisms of Texture Formation in Gamma-TiAl: *Arno Bartels*[1]; Wolfram Schillinger[1]; [1]TU Hamburg-Harburg, Mats. Sci. and Techn., 5-06, Eissendorfer Str. 42, Hamburg D-21071 Germany

The texture evolution in TiAl depends not only on the different types of deformation, for instance, compression or rolling, but also on the initial microstructure. Cast TiAl shows a lamellar microstructure with a strong texture due to an alignment of the lamellae perpendicular to the direction of the heat flow during solidification. A deformation in compression parallel to the aligned lamellae results in a plain strain deformation and ends with a brass texture. The orientation rotation caused by ordinary and superdislocation slip and the role of the mechanical twinning will be discussed. At higher temperatures the deformation of TiAl with equiaxed microstructure results in typical texture components which are determined by a strong mechanical twinning activity which leads to an orientation of the [001]-direction transverse to the direction of the compression stresses. Using the model of the TiAl yield surfaces the characteristics of the textures can be simulated.

10:00 Break

10:15 AM Invited

Elevated Temperature Deformation of the Cubic Laves Phase Cr2Nb: Antonios V. Kazantzis[2]; Ian P. Jones[2]; *Mark Aindow*[1]; [1]University of Connecticut, Metall. and Matls. Eng., 97 N. Eagleville Rd., U-3136, Storrs, CT 06269-3136 USA; [2]University of Birmingham, School of Metall. and Matls., Elms Rd., Edgbaston, Birmingham B15 2TT UK

The mechanical behavior of the C15 Laves phase Cr2Nb at temperatures of 1150°C-1550°C has been studied using compression testing and the deformation mechanisms were identified by using transmission electron microscopy to assess the microstructures of the deformed samples. It was found that the samples deformed by the glide of extended dislocations and/or twinning depending upon the testing temperature, strain rate and (in mixed-mode microstructures) the grain orientation. It will be shown that this complex behavior arises because of the variation in stacking fault energy with temperature and the necessity of synchroshear processes for both the glide of extended dislocations and twinning in this structure.

10:30 AM Invited

Deformation and Microstructure of Transition-Metal Disilicides: *Haruyuki Inui*[1]; Masaharu Yamaguchi[1]; [1]Kyoto University, Dept. of Matls. Sci. and Eng., Sakyo-ku, Kyoto 606-8501 Japan

Many transition-metal disilicides have attracted considerable interest as possible candidates for very-high-temperature structural applications. These disilicides include MoSi2 and WSi2 with the tetragonal C11b structure, VSi2, CrSi2, NbSi2 and TaSi2 with the hexagonal C40 structure and TiSi2 with the orthorhombic C54 structure. The generation of the three different structures is accomplished by changing the stacking order of the MeSi2 layers; these three structures are based on the AB, ABC and ADBC stacking sequences, respectively. Thus, there are equivalent slip systems operative in each of the three structures. We have investigated microstructure and deformation of single crystals of these transition-metal disilicides. When the CRSS and temperature are normalized respectively to the shear modulus and melting temperature, the normalized CRSS-temperature plot indicates that these disilicides are classified into two groups (conventional shear and synchroshear groups) in terms of the onset temperature for plastic flow, regardless of crystal structure.

11:00 AM

Dislocation Microstructures of Mo_5Si_3 Single Crystals Deformed at High Temperatures: *Kyosuke Yoshimi*[1]; Eiji Aoyagi[1]; Man H. Yoo[2]; Shuji Hanada[1]; [1]Tohoku University, Inst. for Matls., 2-1-1 Katahira, Sendai, Miyagi 980-8577 Japan; [2]Oak Ridge National Laboratory, Mets. and Ceram. Div., Oak Ridge, TN 37831-6115 USA

Slip systems and nature of operative dislocations in $D8_m$-type Mo_5Si_3, have not been established yet. In order to determine these, deformation properties and dislocation microstructures in Mo_5Si_3 were investigated using single crystals. Depending on the load axis, several slip planes are identified on the surfaces of the single crystals compressed at high temperatures. Complexity of the observed slip planes is interpreted as multiple combinations of fundamental slip planes, (001), {100} and {110}. Dislocations activated on the fundamental slip planes were observed by TEM, and their Burgers vectors are determined for each dislocation-type. Based on the observed results, dislocation process for the high temperature deformation of Mo_5Si_3 will be discussed.

11:20 AM

Deformation and Microstructures of NbTiCr Laves Phase Alloys at Elevated Temperatures: *Katherine C. Chen*[1]; Paul G. Kotula[2]; Carl M. Cady[3]; Robert D. Field[4]; Dan J. Thoma[4]; [1]California Polytechnic State University, Mats. Eng. Dept., Cal Poly, San Luis Obispo, CA 93407 USA; [2]Sandia National Laboratory, Albuquerque, NM USA; [3]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA; [4]Los Alamos National Laboratory, MST-6, MS-G770, Los Alamos, NM 87545 USA

Specific alloy design methodologies have been utilized to develop a Laves phase intermetallic alloy based on C15-NbCr2. Previous studies have demonstrated an increase in fracture toughness and deformability with strategic alloying additions, such as Ti to NbCr2. In addition, Laves phase intermetallics have also been shown to exhibit enhanced deformation as discrete particles within a more ductile matrix. Thus, two-phase alloys (C15+bcc) in the NbTiCr system have been processed with a novel technique to produce optimized microstructures. Results from compression tests as a function of temperature will be discussed. Microstructures and deformation mechanisms are revealed by transmission electron mi-

croscopy (TEM). Establishment of the structure-property relationships at elevated temperatures will assist in the development of Laves phase intermetallics as viable engineering materials.

11:40 AM
Tensile Properties of Ti3SiC2 in the 25-1300ºC Temperature Range: *Miladin Radovic*[1]; Michel W. Barsoum[1]; *Tamer El-Raghy*[1]; Sheldon Wiederhorn[2]; [1]Drexel University, Mats. Eng., 32nd and Chestnut Sts., Philadelphia, PA 19104 USA; [2]National Institute of Standards and Technology, MISEL, 100 Bureau Dr., Gaithersburg, MD 20899 USA

The functional dependence of the tensile response of fine (3-5 μm) and coarse (100-300 μm) grained Ti3SiC2 samples on strain rates in the 25-1300ºC temperature range was measured. At temperatures above 1100ºC there is a distinct non-linearity in stress-strain curves and the stress at which this transition occurs is not a yield point but an inelastic deformation stress (IDS). Upon unloading the samples from the stress above IDS, inelastic relaxation was observed. The high value of strain rate sensitivity (0. 42-0. 56) of Ti3SiC2 was obtained from tensile tests and confirmed by strain rate jump/drop test and stress jump creep tests. Large plastic deformations (up to >25%) is achieved at temperature above 1100oC and low strain rates (<10-4 s-1). The high value of strain achieved during the tensile tests above 1100ºC is mostly a consequence of void and microcrack formation, indicating a high damage tolerance of Ti3SiC2.

International Symposium on Shape Casting of Aluminum: Science and Technology: Industrial Trends, Challenges and Opportunities

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

Monday AM — Room: 224
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: John E. Allison, Ford Motor Company, Ford Res. Lab., Dearborn, MI 48124-2053 USA; Ray Peterson, Imco Recycling Inc, Rockwood, TN 37854 USA

8:30 AM Welcome
John Allison, Ford Motor Company

8:40 AM Keynote
Cast Aluminum for the Automotive Industry: Trends, Opportunities and Challenges: *Linda Miller*[1]; [1]Ford Motor Company, Casting and Forging Operations, MD 1 POEE Building, Dearborn, MI 48124 USA

Abstract Text Unavailable

9:10 AM Keynote
Industrial Trends for Automotive Casting Production and Technology: *Masamichi Sato*[1]; [1]Toyota Motor Company, Foundry Eng. Div., 1, Nishiyama, Myochi, Miyoshi, Nishikamo-gun, Aichi 470-0214 Japan

In automotive industry, reduction of carbon dioxide for the earth environmental problem has become an important and urgent subject, and in recent years, lots of efforts in improvement of fuel consumption of conventional gasoline/diesel engine and development of light-weight vehicle, new hybrid engine, fuel-cell vehicle etc. have been being done. Aluminum casting processes, that are high-pressure die-casting, squeeze-casting, metal-matrix composite casting, permanent mold casting etc. should play very important role in those development. In this presentation, the production trend of automotive casting parts, for example cylinder block, cylinder head, steering housing, knuckle parts, transmission housing etc. will be presented. And new trend of casting technology for high-functional, high-strength, thinner, net-shaped casting and application of CAD/CAE will be introduced.

9:40 AM Keynote
Castings in Commercial Airplanes, an Overview: *Fred J. Feiertag*[1]; [1]The Boeing Company, Commercial Airplane Group, Manuf. Res. & Dev., P.O. Box 3707 MC 5H-82, Seattle, WA 98124-2207 USA

Castings have always been an important means of achieving the design goals for commercial airliners. The historic development of aerospace casting technology will be used as the background to explain the current challenges. The current emphasis on reducing cost creates new opportunities for castings. Along with the potential for expanding the use of castings are a number of barriers to be addressed by industry, government, and academia. The obstacles to be overcome will be described in three areas; foundry capacity and capability, human resources, and research needs. This presentation highlights the needs for applied research and development to enable castings to be a more effective tool for the cost conscious designer.

10:10 AM Break

10:25 AM Invited
Optimization of Material and Foundry Process for Aluminum Automotive Components: *Philippe Meyer*[1]; [1]Montupet, 202 Quai de Clichy, Cedex 92112 France

In order to meet the booming demand of aluminum engine components, MONTUPET has put in place a chain of tools ranging from in depth analysis of the interactions between microstructure and properties to dedicated manufacturing processes. In particular, it is as essential to shorten the development time as to fulfill the requirements of severe fatigue specifications for cylinder heads and blocks. This has led to implement original development routes and tools. A variety of foundry processes and material/heat treatment selection also appears necessary to bring case by case the right answer to the customer.

10:55 AM Invited
Research Needs and Opportunities in Shape Forming of Aluminum: *John Green*[1]; Sara Dillich[2]; Philip Sklad[3]; [1]The Aluminum Association, 900 19th St. N.W., Washington, DC 20006-2168 USA; [2]U.S. Department of Energy, Ofc. of Indut. Techn., EE-20, 1000 Independence Ave. S.W., Washington, DC 20585-0121 USA; [3]Oak Ridge National Laboratory, Met. and Ceram. Div., P.O. Box 2008, MS6065, 1 Bethel Valley Rd., Oak Ridge, TN 37831 USA

Through the Aluminum Industry Technology roadmap (May, 1997) and the Aluminum Industry roadmap for the Automotive Market (May,1999), the aluminum industry has identified ambitious technology research and development goals in the area of shape forming of aluminum. These goals are being actively pursued through collaborative, cost-shared R&D with the Department of Energy, other federal agencies, and the USAMP AMD cast light metals initiative. Enabling technologies and on-going R&D in sheet forming, pressure casting, lost-foam casting, semi-solid forming, ultra-large castings, metal compression forming, etc., will be discussed, as will opportunities for future research efforts in these areas. Challenges facing the integration of aluminum components into technology driven markets such as the automotive market will also be addressed. These challenges include low-cost ingot, defect-free castings and sheets, and process reengineering.

11:25 AM Panel Discussion

Lead-Free Solder Materials and Soldering Technologies I: Emerging Technologies, Materials, Processing

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 Chemistry 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

Monday AM Room: 227
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Sung K. Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; Carol Handwerker, NIST, Metall. Div., Gaithersburg, MD USA

8:30 AM Opening Remarks

8:35 AM Invited

A Metallurgical Study of Pb-Free Solders for Flip Chip Interconnects: *Darrel Richard Frear*[4]; Jin Wook Jang[1]; Jong Kai Lin[1]; Charles Zhang[1]; Li Li[1]; [1]Motorola, Interco. Sys. Lab., 2100 East Elliot Rd., MDEL725, Tempe, AZ 85284 USA

A variety of Pb-free solder alloys have been proposed for use as flip chip interconnects including Sn-Ag, Sn-Cu, Sn-Ag-Cu, Sn-Ag-Bi, and Sn-Sb, among others. This study was performed to characterize the reaction behavior and reliability of these solders for a fine pitch flip chip configuration on a variety of under bump metallurgies. The intermetallic reaction products and kinetics were determined and related to mechanical integrity. The reliability tests performed included shear strength, isothermal fatigue, and thermomechanical fatigue all as a function of the number of reflows and solid state aging. The solder microstructure and interfacial intermetallics dominate the performance of the solder alloys and will be documented along with a recommendation of an optimal solder alloy for flip chip applications.

9:00 AM Invited

Sn-Zn-Al Pb-free Solder-An Inherent Barrier Solder for Cu Contact: *Kwang-Lung Lin*[1]; Hui-Min Hsu[1]; [1]National Cheng Kung University, Dept. of Mats. Sci. and Eng., 1 Ta-Hsuey Rd., Tainan, Taiwan 701

Flip chip solder bumps were produced on Cu contact applying Sn-9Zn-xAl Pb-free solder by dipping method. The solder bumps were treated under 85ºC/85% RH (relative humidity) or at 150°C for 1000 hours to explore the interfacial interaction behavior. Experimental results revealed that Al and Zn, not Sn, diffuse to the Cu/solder interface during the extended period test. A Al4. 2Cu3. 2Zn0. 7 compound, characterized by XRD, was formed at the interface for the as produced solder bump. The long time test resulted in the formation of Cu5Zn8 and Al4Cu9 compounds. The gathering of Al at the interface forms a barrier to Sn diffusion toward Cu substrate and thus no Cu-Sn compound was detected. This is the first time to find a Sn-containing solder which, in contact with Cu, does not form Cu-Sn intermetallic compound during heat treatment and thus the Sn-Zn-Al solder is termed an inherent barrier solder.

9:25 AM

IMS Injection Molded Solder Technology for Pb-free Solders: *Peter A.. Gruber*[4]; [1]IBM, Research, Thomas J. Watson Res. Cen., P.O. Box 218, Yorktown Heights, NY 10598 USA

A new solder process technology called Injection Molded Soldering, or IMS, has been developed. This process is amenable to many solder alloys, including lead free compositions. Key features of this technology are flexibility, simplicity and the potential for reducing solder processing costs. The process hinges on a new Injection Molded Solder (IMS) head that melts bulk solder and subsequently dispenses same into molds of various configurations, depending on application. The solder in the molds is then transferred to substrates. The flexibility of IMS allows it to process BGA and CGA packages as well as bump wafers. The simplicity of the process permits various solders to be readily compared. Thus it is ideal for evaluating lead free alloys to determine their compatibility with various under bump metallurgies, as well as their joining properties. Other characteristics of the process are minimal solder waste and array uniformity. IMS reduces manufacturing steps and thus potentially solder processing costs, an attractive feature as lead free solders grow in usage.

9:45 AM

Influence of Alloy Composition on Fillet-Lifting Phenomenon in Sn-Ag-Bi Alloys: *Hisaaki Takao*[1]; Hideo Hasegawa[1]; [1]Toyota Central R&D Labs., Inc., Mats. Div. I, Lgtwgt. & Envi. Labs., Nagakute-cho, Nagakute Yokomichi 41-1, Aichi-gun, Aichi pref. 480-1192 Japan

The influence of Bi on the fillet-lifting (FL) in Sn-3Ag-xBi alloys was investigated. The fillet-lifting doesn't occur below 1mass%Bi. The FL occurrence rate increases up to 10mass%Bi and then decreases followed by no occurrence of FL in 30mass%Bi. This peculiar composition dependence was found to be related to the temperature change near the solidus temperature in the cooling curve. Namely, the latent heat release is considered to contribute to the alleviation of the temperature gradient raising FL in the solder joint. We propose a fillet-lifting index (FRI) from the analysis of the cooling curve as a new predictable index for the FL occurrence, which is well correlated with the FL occurrence rate. On the contrary, the formation of Bi concentrated layer at the solder/Cu land interface, thought to cause FL, is considered to result from the Sn consumption by the interfacial reaction between Sn and Cu.

10:05 AM Break

10:20 AM Invited

The NCMS High Temperature Fatigue-Resistant Solder Project: *Frank W. Gayle*[1]; Gary Becka[2]; Alan Gickler[3]; Jerry L. Badgett[4]; Gordon C. Whitten[4]; Tsung-Yu Pan[5]; James Slattery[6]; Christopher G. Olson[7]; Ahmer Syed[8]; Angela Grusd[9]; Brian Bauer[9]; Iver Anderson[10]; James Foley[10]; Duane Napp[11]; [1]NIST, Metall. Div., Mail Stop 8555, 100 Bureau Dr., Gaithersburg, MD 20899-8555 USA; [2]Allied Signal, FM&T, D/836, MS 2C43, P.O. Box 419159, Kansas City, MO 64141-6159 USA; [3]Johnson Manufacturing, 114 Lost Grove Rd., P.O. Box 96, Princeton, IA 52768-0096 USA; [4]Delphi Delco Electronics Systems, One Corporate Center, MS 8186, P.O. Box 9005, Kokomo, IN 46904-9005 USA; [5]Ford Motor Company, Ford Res. Lab., 2101 Village Rd., Bldg. R, M/D 3135, Dearborn, MI 48124 USA; [6]Indium Corporation of America, P.O. Box 269, Utica, NY 13502 USA; [7]Rockwell Collins, MS 107-110, 400 Collins Rd. NE, Cedar Rapids, IA 52498 USA; [8]Amkor Technology, 1900 South Price Rd., Chandler, AZ 85248 USA; [9]Heraeus, Circuit Mats. Div., 24 Union Hill Rd., West Conshohocken, PA 19428 USA; [10]Ames Laboratory, 126 Mets. Dev., Ames, IA 50011 USA; [11]National Center for Manufacturing Sciences, 111 Hazeltine Dr., Georgetown, TX 78628 USA

In addition to the move toward environmentally friendly lead-free solders, the automotive, avionics, and oil exploration industries are seeing an increased number of applications for microelectronics where temperatures exceed 125°C. As a follow on to the original Lead-free Solder Project, the National Center for Manufacturing Sciences has sponsored the High Temperature Fatigue-Resistant Solder project to determine the performance of several new lead-free solders, as well as the common Pb-free alternatives, in high temperature applications. Participants include OEMs (Delphi, Ford, Rockwell, AlliedSignal), a component supplier (Amkor), solder suppliers (Johnson Manufact., Indium Corp., and Heraeus) and other labs (NIST, Ames Lab.). This four-year effort has focused on the thermal fatigue performance of a number of lead-free, Sn-based solders cycled to temperatures of 125° to 160°C, with a goal of meeting the thermal fatigue performance of eutectic tin/lead solder cycled at -55 to +125°C. This talk will present results from the study.

10:45 AM

Lead-Free Solders for Automotive Body Construction: *Tsung-Yu Pan*[1]; Alan Gickler[2]; Howard D. Blair[1]; John M. Nicholson[1]; [1]Ford Motor Company, Ford Res. Lab., 2101 Village Rd., Bldg. R, M/D 3135, Dearborn, MI 48124 USA; [2]Johnson Manufacturing Company, 114 Lost Grove Rd., P.O. Box 96, Princeton, IA 52768-0096 USA

Lead (Pb) based solders had been used successfully as dent and seam filler for automotive body panels and were commonly refered to as "body solders". The usual composition was 70Pb/30Sn with a pasty range from 182 to 258°C. New lead-free body solders were developed in the early 80's with a Sn-Cu-Zn composition to mimic the pasty characteristics and processability of the lead-containing solders. This material was used in vehicle mass production until the silicon bronze materials were developed. However, the body solders still have the advantage of corrosion resistant, compatibility with e-coat, and lower processing temperature and lower material cost than the silicon bronze materials. In this presentation, the historical background of body solders, lead-free solders, process, microstructures, and the advantages of using such solder materials will be discussed.

11:05 AM Invited

A Lead-Free Active Solder for Joining Electronic Packaging: *Ronald W. Smith*[1]; Paul Vianco[2]; Cynthia L. Hernandez[2]; Ino Rass[3]; Frank Hillen[3]; [1]Materials Resources International, 811 W. Fifth St., Unit 2, Lansdale, PA 19446 USA; [2]Sandia National Laboratories, M. S. 1411, P.O. Box 5800, Albuquerque, NM 87185 USA; [3]Euromat GmbH, Rheinstrase 7, Huckelhoven D-41836 Germany

Active solders incorporating Ti, Hf, Zr and other active elements have been recently developed and are being introduced in a range of applications. In particular, an Sn-Ag-Ti active solder has been developed that has been shown to wet and bond a wide range of ceramic, metal and composite materials that have application in electronic packaging and thermal management components for electronics. The reported work investigated the stability and interaction of the active Sn-Ag-Ti alloy with a range of materials that are used in electronic packages, including Al, Cu, Al2O3, AlN, Al:SiC, and low expansion alloys such as Kovar®. Excellent joints with these materials have been achieved and utilize a fluxless joining method that processes in air at 250°C without the need for premetallization. Processing steps will be reviewed and as-joined interface microstructures will be reported and compared to joints that have been exposed to hold times and temperatures ranging from 10-100 hours at temperatures from 100°C-200°C are reported. Reported results will show that the active alloy Sn-Ag-Ti base with Ga and rare earth additions interact and effectively join electronic packaging materials and could be used to replace metallization pretreatments used to prepare materials for conventional solder joining or active braze joining now used in electronic applications that require ceramics or composites and/or dissimilar material joints.

11:30 AM

Characterization of Pb-Free Solders and Pb-Free Electroplating Systems for Memory Semiconductor Applications: *Seung Wook Yoon*[1]; Ik Seong Park[1]; Heung Sup Chun[1]; [1]Hyundai Electronics Industries Company, Ltd., Semicon. Grp., Ichon, Kyunggi-do 467-701 Korea

This study focuses on the application of Pb-free solder technologies for memory device packaging and SMT of memory modules. For memory devices such as DRAM, SRAM and Flash memory, they use the TSOP, QFP type packages as well as CSP. To evaluate the Pb-free solders and Pb-free electroplating systems for leaded packages, various solder systems and electroplating systems were characterized in terms of manufacturing feasibility, wetting properties, mechanical properties and board level reliability. For board level solder joint reliability test, daisy chain samples with various electroplating system were prepared, and SMTed on daisy chain PCB with Pb-free solders. After T/C tests (-65C~150ºC and -55C ~ 125ºC) of PCB board level mounting, solder joint lifetime was electrically observed and compared. After board level reliability test, interfacial reaction and microstructure such as crack initiation site and crack propagation, were investigated. Their fractured surface, microstructure of solder joint interface and of solder ball were examined and analyzed by optical microscopy, XRD, SEM and EDX. Almost of solder joint cracks were found at the heel joint region and the crack propagated along the interfacial region as well as bulk solders.

11:50 AM

Fluxless Flip Chip Bonding of Si-Wafer/Bumps/Glass by Plasma Treatment: *Soon Min Hong*[1]; Chang Bae Park[2]; Jae Pil Jung[2]; Choon Sik Kang[1]; [1]Seoul National University, Schl. of Mats. Sci. and Eng., San 56-1, Shinrim-Dong, Kwanak-Gu, Seoul 151-742 South Korea; [2]University of Seoul, Dept. of Mats. Sci. and Eng., 90 Jeonnong-Dong, Dongdaemun-Gu, Seoul 130-743 South Korea

In flip chip assembly, as the packaging density grows high, the cleaning of flux used in conventional process becomes increasingly difficult. The flux residue can seriously affect the reliability and performance of flip chip assemblies by corrosion. In addition, the chemical solvent for flux cleaning process can also cause the environmental problem which is a world-wide concern in recent years. The purpose of this research is to evaluate the fluxless flip chip bonding properties between Si-wafers and glass substrates using micro solder bumps. Instead of flux, we used the plasma cleaning to remove the oxides and other contaminants of solder bumps and substrates before flip chip bonding. The mechanical properties of the solder bump joint were examined. The the intermetallic compounds between solder and UBM and TSM were analyzed. The effect of flip chip reflow parameters were also discussed.

Magnesium Technology 2001: Magnesium Reduction–Lloyd M. Pidgeon Memorial Session

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

Monday AM — Room: 203-205
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: R. Neelameggham, Magnesium Corporation of America, Salt Lake City, UT 84116 USA

8:30 AM

Introduction by Ruth Pidgeon Bryson, Daughter of Late Lloyd M. Pidgeon

8:35 AM Keynote

Lloyd M. Pidgeon-Magnesium Pioneer: *Robert E. Brown*[1]; [1]Magnesium Assistance Group, Inc., 226 Deer Trace, Prattville, AL 36067-3806 USA

Lloyd Montgomery Pidgeon was an unusual man in an unusual time. His contributions to the development of the magnesium industry have never been appreciated (or even known) by many of today's magnesium followers. Dr. Pidgeon, working with one technical graduate, achieved commercial development of a process to produce magnesium by reducing calcined dolomite with ferrosilicon i.e. the silicothemic process. He also received patents for electrolytic magnesium processes. He worked with engineers to design and build 6

magnesium production plants in a very short period. The original plant at Haley, Ontario is still operating. He received many technical honors, but was always quick witted, with a humorous approach to life.

9:20 AM

The Pidgeon Process in China and its Future: Jing Chun Zang[1]; Weinan Ding[2]; [1]Gold River Magnesium Plant, Ningxia Huayuan Magnesium Group, No. 50 Wenhuadong St.,Yinchuan, China 750004; [2]Sinomag, 1204 Floor 1 Landmark Tower, 8 Dongsanhuan Bei Lu, Beijing, China 100004

Magnesium production in China has been growing steadily over the past 10 years. Most of the metal has been produced by the Pidgeon process. This process uses horizontal steel tubes called retorts, in furnaces and under vacuum. In the retorts mixtures of finely ground calcined dolomite and ferrosilicon formed into briquettes react to form magnesium vapors which are condensed and later remelted into ingots. The Pidgeon process was long thought to be uneconomic and obsolete. The Chinese have used the advantages of excellent raw material, location, large skilled labor supply, and low capital costs to produce magnesium by this process. The Chinese magnesium is being sold at the lowest prices in the world and lower than aluminum on a pound for pound basis.

9:45 AM

Chinese Adaptation of the Pidgeon Process: *Gerald S. Cole*[1]; [1]Ford Motor Company, Dearborn, MI 48121 USA

The author recently participated in the first Chinese Magnesium conference in Beijing and visited 5 plants, 4 of which were primary producers and 1 which was only a recycler. He will discuss the Chinese method for producing ultra low-cost Mg and will examine the potential impact of this low cost metal on the West. He will support his observations through video analysis of the modified Chinese Pidgeon process.

10:10 AM Break

10:20 AM

Vertical Larger-Diameter Vacuum Retort Magnesium Reduction Furnace: *Xiaoming Mei*[1]; *Alfred Yu*[1]; *Shixian Shang*[1]; *Tianbai Zhu*[1]; [1]Nanjing Welbow Metals Co., Ltd, Jingqiao, Lishui, Nanjing, Jiangsu 211224 China

A new magnesium reduction technology has been developed to improve the Pidgeon reduction process. A demo-plant of 1000t magnesium per year succeeds in applying this new technology. Firstly, a new furnace is developed and a larger-diameter vertical settled vacuum retort is used instead of traditional horizontal retort. So the furnace can be designed with more compact structure to raise the magnesium output per furnace volume Secondly, Calcined dolomites and ferrosilicon is compressed into given unitary shape for enhancing heat and mass transfer during the reduction and shorten remarkably the reduction time. The shape is designed with reference to the numerical simulation result. Demo operation showed that, with application of the technology, significantly production capacity is increased in the same furnace, reduction period is decreased (only two thirds of the traditional reduction period), energy consumption is decreased, retort's life-span is extended, operation is easy and the total production cost is reduced.

10:45 AM

A Computational Thermodynamic Analysis of Atmospheric Magnesium Production: *Mellisa Lee Marshall*[1]; Zi-Kui Liu[1]; Roy Christini[2]; [1]The Pennsylvania State University, Dept. of Mats. Sci. and Eng., Steidle Bldg., University Park, State College, PA 16802 USA; [2]ALOCA, Technical Center, 100 Technical Dr., ALCOA Center, PA 15069 USA

The Magnetherm process is the most widely used thermal reduction process for commercial magnesium production. This process requires a vacuum atmosphere, ferrosilicon reductant, and dolomite ore. The vacuum atmosphere is typically 0.1 atm. However, the vacuum atmosphere creates two major problems: air leakage and batch operation to tap excess slag. The air leakage contaminates the magnesium vapor and the batch operation lowers productivity. Atmospheric production of magnesium could eliminate the vacuum requirement. By increasing the pressure inside the furnace to atmospheric pressure, a pressure difference would not exist between the outside and the inside of the furnace. Air would not leak into the furnace and excess slag could be tapped without stopping the production. However, the atmospheric magnesium process will require a different reaction temperature and slag composition since under current operating parameters, magnesium cannot be produced when the pressure is over 0.63 atm. A computational thermodynamic analysis was completed on a variety of slag compositions and reaction temperatures. The data collected was used to determine three key factors: (1) purity of the magnesium vapor; (2) aggressiveness of the slag; and (3) fraction of solids in the bulk slag. All slags were ranked according to how well they scored in the three key areas.

11:10 AM

Producing Magnesium for Use in the Titanium Manufacturing Process: *Laura K. Simpson*[1]; [1]Titanium Metals Corporation, P.O. Box 2128, Henderson, NV 89009 USA

The introduction in 1993 of vacuum distillation technology to Titanium Metals Corporation (TIMET) has greatly reduced the need to purchase make-up magnesium for the titanium manufacturing process. Therefore, the production of magnesium metal in-house has become even more critical. As recently as 1998, the TIMET magnesium plant has produced at its maximum capacity of 15,000 tpa, all for internal use. Production of magnesium metal for use in the titanium reduction process, however, poses several unique operational problems. Variability of feed stock volumes, feed stock impurities, handling molten feed and the trials of running a continuous operation balanced with two sister processes are some of the challenges. This paper will discuss the production of magnesium as it relates to titanium sponge manufacturing at TIMET, including overviews of the current titanium vacuum distillation and Alcan monopolar technologies, and solutions to the unique operational problems posed by the process.

11:35 AM

Modernization at Magcorp-Coming of Age in the 21st Century: Howard I. Kaplan[1]; Ron Thayer[1]; R. Neelamegham[1]; Ray Bassani[1]; [1]Magnesium Corporation of America, Salt Lake City, UT 84116 USA

Magnesium Corporation of America(Magcorp) is the oldest and largest magnesium plant in the United States. Magcorp is modernizing and upgrading the facility into one of the most modern, technically, economically and environmentally efficient plants in the world via a capital investment that is less than 10% of that needed for a greenfield plant. This paper will describe the technical improvements made in three major areas: Magnesium chloride purification, Electrolytic cell technology, and direct chill casting and other magnesium casting technology improvements. The economic benefits of the improvements will be quantitatively defined including production cost benefits associated with energy reductions and manpower improvements as well as production capacity increases. Cast house modernization via direct chill and other technology will be discussed along with metallurgical analyses that demonstrate metal cleanliness levels that are state of the art. The environmental benefits from the technology insures that the Magcorp plant is well ahead of all the clean air act requirements and meets all modern environmental air quality limits.

Materials Processing Fundamentals I

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

Monday AM Room: 218
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Ramana G. Reddy, University of Alabama, Dept. of Metall. & Mats. Eng., Tuscaloosa, AL 35487-0202 USA

8:30 AM Introduction

8:40 AM
Gravitational Acceleration Effects on Microstructure: Experiments and Computational Modeling: *Jose Felix Leon-Torres*[1]; *Doru M. Stefanescu*[1]; S. Sen[2]; P. A. Curreri[2]; [1]The University of Alabama, P.O. Box 865518, Tuscaloosa, AL 35486 USA; [2]USRA Marshall Space Flight Center, Huntsville, AL USA

Experiments were performed under terrestrial gravity (1g) and during parabolic flights (10-2 g) to study the solidification and macrosegregation patterns of Al-Cu alloys. Alloys having 2% and 5% Cu were solidified against a chill at four different cooling rates. Microscopic characterization was used to analyze the final microstructure in each sample. Measurements of primary and secondary dendrite arm spacing are presented. Macrosegregation results from each alloy and casting condition are presented. Experimental relationship between dendrite arm spacing, gravity levels, macrosegregation, and cooling rate are discussed for each alloy. A 3-D computational model is used to explain the experimental results. The continuum formulation was employed to describe the macroscopic transports of mass, energy, and momentum, associated with the solidification phenomena, for a two-phase system. The model considers that liquid flow is driven by thermal and solutal buoyancy, and by solidification shrinkage. The solidification event was divided into two stages. In the first one, the liquid containing freely moving equiaxed grains was described through the relative viscosity concept. In the second stage, when a fixed dendritic network was formed after dendritic coherency, the mushy zone was treated as a porous medium. The cooling curves obtained during experiments were used for validation of the solidification model. The model is used to predict the dendrite arm spacing for each alloy. The model can explain the solidification and microstructure results for each cooling rate and the differences between low- and high-gravity results.

9:05 AM
Microstructural Characterization of Two-Phase Alloys: *Paula J. Crawford*[1]; M. E. Glicksman[1]; [1]Rensselaer Polytechnic Institute, Mats. Sci. and Eng. Dept., 110 Eighth St., Troy, NY 12180 USA

Coarsening in two-phase alloys is usually characterized in terms of the kinetics of the dispersed particles, using quantities derived from the well-known particle size distribution (PSD). The corresponding behavior of the continuous matrix phase, however, is more subtle, usually considered "passive" and often ignored in experimental and theoretical phase coarsening studies. Correlations between the coarsening behavior of the matrix and the dispersed phase are expected. This study employs an analysis to characterize the microstructure of a two-phase CuCo alloy, prepared as a uniform solid-state dispersion of cobalt particles. Our method employs a Voronoi tessellation to obtain characteristic geometrical parameters of cross-sections of the coarsening matrix. The microstructures are then evaluated via an automated image analysis system. Experimental data are collected with image processing software. Correlations found among the matrix parameters and those of the dispersoid are being evaluated and will be discussed.

9:30 AM
Properties of Extruded Alloy Honeycomb of Varying Cell Geometry: *Joe K. Cochran*[1]; [1]Georgia Institute of Technology, Mats. Sci. and Eng., Atlanta, GA 30332-0245 USA

A technology developed at Georgia Tech allows fabrication of thin-walled metal honeycomb. Using powder paste, honeycomb shapes are extruded with non-metallic precursors and subsequently converted to the metallic state by a direct reduction process. This process fabricates alloy honeycomb with selectable cell geometry and integral face sheets. These "linear cellular alloys" may be in a multitude of cell geometries including square and triangular and are intended for high heat transfer and high strength applications. Mechanical properties of square and triangular honeycomb from maraging steel and Inconel 617 will be presented for principal axial orientations and compared to mechanics models. For square cell structures, a cell size of 1. 5 mm with wall thickness of 150 microns has been achieved. Relative densities for these alloy honeycomb is in the 15-20% range but efforts are underway to reduce density significantly. Fabrication goals include high volume production capability, composition compatibility with existing systems, and affordability.

9:55 AM
Effect of Alloying Additions on the Reaction Synthesis of Aluminide Intermetallics: *Khaled M. Morsi*[1]; Sherif O. Moussa[1]; [1]University of Missouri, Mech. & Aeros. Eng., E3411 Eng. Bldg East, Columbia, MI 65211 USA

Aluminide intermetallics have extremely advantageous properties including structural, shape memory, magnetic and superconductive. The fabrication of such materials using energy-efficient processes such as reaction synthesis have been conducted in the past. The process involves the mixing of elemental powder e. g. nickel and aluminum in the correct proportion to form the intended nickel aluminide. The powder is then compacted into a pellet and heated under vacuum or inert atmosphere to a temperature of ~700ºC. At this temperature an exothermic reaction occurs converting the elemental powders into the intended intermetallic material. The heat of reaction heats the reacting pellet to very high temperatures. The notorious reaction induced porosity can be overcome by applying a novel process that uses extrusion during the high temperatures achieved during the reaction, which presents great possibilities and is the subject of a detailed study by the authors. For reaction synthesis to fully realize their industrial potential, the effect of alloying element additions on the reaction synthesis must be conducted. Surprisingly little work has been published on the subject, when it is well known that aluminides will almost always need to be alloyed before they can be used in industry. The work presented in this paper investigates the effect of alloying additions on the reaction synthesis of aluminide intermetallics. The effect of processing variables on the developed microstructure is discussed.

10:20 AM Break

10:40 AM
Microstructural Characterization of Alloys Reduced from Oxide Precursors: *Thomas H. Sanders*[1]; [1]Georgia Institute of Technology, Mats. Sci. and Eng., Atlanta, GA 30332-0245 USA

This paper will discuss the bulk properties of alloys reduced from oxide powder precursors. The oxide powders are processed through a paste extrusion method to produce honeycomb structures of various geometries and densities. Once dried, the green honeycomb structures are heated in a hydrogen-containing atmosphere to reduce the oxides to their metallic constituents. The result is a metal honeycomb, termed a linear cellular alloy, that shows significant potential for use in weight sensitive applications. Several relationships are available to predict the mechanical performance of such honeycomb structures based on the properties of the bulk material. Hence it is necessary to study the bulk material to determine the effects of the oxide powder processing route on the material properties and ultimately the performance of the linear cellular alloy. Three different alloy systems will be discussed: high conductivity copper and dilute copper alloys, the high temperature nickel alloy 617 with a nominal composit ion Ni-21Cr-12Co-9Mo, and a 350-grade maraging steel with a nominal composition of Fe-18Ni-12. 5Co-4Mo. Each of these alloys have properties that are ideally suited for hon-

eycomb structures designed to dissipate heat or absorb energy.

11:05 AM
On the Relation Between Softening Processes and the Gamma-Alpha Phase Transformation in Steel: *David N. Hanlon*[2]; Jilt Sietsma[1]; Sybrand van der Zwaag[1]; [1]Delft University of Technology, Lab. of Mats. Sci., Rotterdamseweg 137, Delft 2628 AL, The Netherlands; [2]Netherlands Institute for Metals Research, Rotterdamseweg 137, Delft 2628 AL, The Netherlands

The effect of high-temperature plastic deformation of the austenitic phase (γ) on the subsequent phase transformation to ferrite (α) has been experimentally investigated, and modelled by a physically-based model. The effect is of importance for steel sheet, in view of the significance of the phase transformation for the eventual properties of the material. In this paper the kinetics of the γ-α phase transformation has been studied after different degrees of prior deformation. The degree of deformation is varied by applying different deformation temperatures and strain rates, and by allowing a varying extent of recovery and recrystallisation. Consequently, both dynamic and static recovery and recrystallisation have been involved. The dilatometric observations on the phase transformation have been modelled using an interface-mobility model, in which the effect of the dislocation structure is introduced as an additional driving-force term. Both nucleation and growth are regarded in relation with the deformed structure.

11:30 AM
Effect of Electropulsing on the Ductility of Steel Wire: Di Yang[1]; *Hans Conrad*[1]; [1]North Carolina State University, Dept of Mats. Sci. and Eng., P.O. Box 7907, Raleigh, NC 27695-7907 USA

A preliminary study into the influence of high density(10^4-10^5) electric current pulsing (70µm duration) and continuous DC (~10^3A/cm^2) on the strength and ductility in tension of a high carbon steel wire was carried out at a strain rate of 10^4S^-1 at 400°F (204°C), 800°F (427°C) and (1200°F(649°C). Studies employing single pulse yield an electron wind-dislocation push coefficient Bew=3. 7x10^-3dyn-s/cm^2, which is in accord with that previously obtained for BCC Nb. Multipusing(100puse per second) with an effective current density of 100-200A/cm^2 give an enhancement by a factor of 1.10 in the uniform elongation of the wire at 400°F, but had no significant effect at 800°F and 1200°F. In contrast, continuous DC either had no significant effect on the ductility or gave a decrease. The results suggest that the enhanced ductility by electropulsing may be further increased by raising the effective current density through increases in pulsing frequency, maximum current density or pulse duration.

Materials & Processes for Submicron Technology: Processing Related Issues

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 Engineering, Tampa, FL 33620 USA; Ashok Kumar, University of South Florida, Department of Mechanical Engineering, Tampa, FL 33620 USA; Sailesh Merchant, Lucent Technologies, Orlando, FL 32819 USA; Mahesh Sanganeria, Novellus Systems, Inc., San Jose, CA 95134 USA

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Session Chair: Mark Anthony, University of South Florida, Coll. of Eng., Tampa, FL 33160 USA; Raj. Singh, OSRAM Sylvania, Chem. Res. Dev., Hawes St., Towanda, PA 14848 USA

8:30 AM Invited
Thermal Processing: The Evolution of Fast Ramp Furnaces: *Pradip K. Roy*[1]; Sailesh M. Merchant[1]; [1]Lucent Technologies, Bell Lab., 9333 South John Young Pkwy., Orlando, FL 32819 USA

The capabilities and advantages of advanced batch furnaces in meeting semiconductor process requirements, till at least the 10nm technology node, are reviewed. Hot wall batch furnaces continue to provide accurate temperature control, low cost of ownership and process advantages compared to single wafer tools. Recent advances by various furnace vendors have addressed the hot walled furnace shortcomings of thermal response, process times and automation overload, resulting in improved manufacturing economics. The inherent benefit of large load size, isothermal processing, uniform film growth, high reliability and low capital cost, has ensured a substantial cost of ownership advantage over single wafer processes. Accurate temperature modulation at high temperatures, during both heating and cooling cycles, has allowed synthesis of ultra thin gate oxides for devices below sub-20nm design rule.

9:00 AM Invited
Mechanism of HSG Formation for DRAM Cells by RTCVD: *Shlomo Berger*[1]; Avishy Captain[2]; Hedvi Spielberg[2]; Eli Iskevitch[2]; Sagy Levy[2]; [1]Technion, Mats. Eng., Haifa 32000 Israel; [2]Steag CVD Systems, Ramat Gabriel, Migdal, Aemek, Israel

An uneven coating made of Hemispherical-Grained Si(HSG) was formed on an amorphous Si layer by a rapid thermal CVD (RTCVD) process. The uneven coating is aimed to increase the effective surface area of a capacitor electrode in dynamic random access memory(DRAM) cells. The formation of the HSG consists of "seeding" and subsequent isothermal annealing stages. During the "seeding" stage nanometer size Si single crystals are formed and distributed uniformly on the surface of the amorphous Si layer. During the rapid annealing at 665ºC, under high vacuum, the Si grains grow up to 95nm after 20sec. The nucleation and growth of the HSG are done under a narrow range of temperatures and times that enable short diffusion path of Si atoms on the surface of the amorphous Si layer but prevent crystallization of the amorphous Si layer. The HSG coating increases the capacitance of the cell by a factor of 2.

9:30 AM Invited
Silicon Device Processing in H-Ambients: H-Diffusion Mechanisms and Influence on Electronic Properties: *Bhushan Sopori*[1]; Yi Zhang[1]; Nuggehalli M. Ravindra[2]; [1]National Renewable Energy Laboratory, 1617 Cole Blvd., Golden, CO 80401 USA; [2]NJIT, Dept. of Phy., 161 Warren St., Newark, NJ 07102 USA

Hydrogen is a ubiquitous, and electronically important, impurity in silicon. The influences of H on Si device properties are only recently being observed and are partially understood. It is now well established that hydrogen and provide passivation of interface and bulk defects, dangling bonds, and impurities in Si. Early observations of improvements in the performance of Schottky and MOS devices, following a forming gas anneal, are now verified as resulting from a decrease in the interface state density and a concomitant interface passivation. Passivation of bulk defects and impurities in Si is exploited commercially for improving the performance of silicon solar cells. However, there is no unified theory of hydrogen diffusion mechanisms. Furthermore, the experimental values of diffusivity of H in Si differ strongly. This paper will review fundamental issues of hydrogen in silicon-diffusion mechanisms, states of hydrogen, and interactions with other impurities and defects.

10:00 AM Invited
Etch Issues for Interconnects at 0. 13µm and Beyond: *Steve Lassig*[1]; [1]Lam Research Corporation, 4650 Cushing Pkwy., Fremont, CA 94588 USA

Subtractive aluminum etch for interconnects is being replaced by copper damascene processes. At the 0. 13 um node, the dielectric choice for insulating the back end wiring is changing to lower dielectric constant materials. These changes are radically changing the technology requirements for patterning. The most common integration method for copper is dual damascene, which requires multiple passes through lithography and etch before further deposition and planarization occurs. This adds challenge in that some of the patterning steps occur over severe topography. As the dielectric material changes to lower dielectric constant we are encountering many diverse new materials that need to be etched. This is driving etch technologists to new and wide ranging chemistries. Of course, all of

this must be done with ever increasing demands for the reaction chambers in a manufacturing environment.

10:30 AM Break

10:50 AM

Study on Via Etching Variation and Post-Clean Treatment on Via Electrical Performance: *Chiew Nyuk Ho*[1]; Yeow Kheng Lim[2]; Higelin Gerald[1]; Wang Ling Goh[2]; Man Siew Sze[2]; Alex See[3]; [1]Nanyang Technological University, Scl. of Mats. Eng., Nanyang Ave. 639798 Singapore; [2]Nanyang Technological University, Sch. of Elect. and Electr. Eng., Nanyang Ave. 639798 Singapore; [3]Chartered Semiconductor Manufacturing Pte, Ltd., Techn. Dev., 60, Woodlands Industrial Park D, St. 2 738406 Singapore

In this body of works, effects of via etching variation as well as post-clean treatment (PCT) on via electrical performance were studied. For this content, stress-migration test was performed to investigate temperature effect while both thermal and electrical factors were accounted in the electromigration test. It is found that via etching with TiN anti-reflection coating (ARC) etch-through results in not only lower initial via resistance, but also higher resistance to stress-migration and longer EM lifetimes. Although this is contradicting with the reported phenomena, the subsequent Ti/TiN barrier acting as the stress buffer is believed to be the reason. On the other hand, the initial via resistance and stress-migration resistance are deteriorated with the implementation of the additional PCT. This may due to the introduced CxFy residues (by PCT step) at the bottom of the via, thereby degrading the interface properties. However, the EM lifetimes seem to be unaffected by this contamination layer. The better EM performance might be related to the removed TiOxNy layer. Reliability of vias, the interconnections between two metal lines, is indeed a very complex phenomenon because the use of multiple materials and structures. The interfaces between dissimilar materials become the dominating factor affecting the via performance.

11:10 AM

Laser-Induced Titanium Disilicide Formation for Sub-Micron Technologies: *Yung Fu Chong*[1]; Kin Leong Pey[1]; Andrew Thye Shen Wee[2]; Alex See[3]; Chih-Hang Tung[4]; Yong Feng Lu[1]; [1]National University of Singapore, Elect. and Comp. Eng., 4 Eng. Dr. 3, Singapore 117576 Singapore; [2]National University of Singapore, Phys., Lower Kent Ridge Rd., Singapore 119260 Singapore; [3]Chartered Semiconductor Manufacturing, Ltd., Techn. Dev., 60 Woodlands Industrial Park D. St. 2, Singapore 738406 Singapore; [4]Institute of Microelectronics, Fail. Anal. and Reli., 11 Science Park Rd., Singapore Science Park II, Singapore 117685 Singapore

Currently, a two-step anneal process is employed for the silicidation of titanium. The first rapid thermal anneal (RTA) step is to achieve the C49 $TiSi_2$ phase, and the second step is to form the low resistivity C54 phase. However, as the width of the polysilicon line decreases, conversion of C49 to C54 $TiSi_2$ becomes extremely difficult. This is because the C49 to C54 phase transformation nucleates only at locations where three C49 grains intersect and the number of such intersection points (triple points) is reduced as the gate length decreases. In this paper, we have investigated the effect of replacing the first RTA step by a laser anneal step on the formation of C54 $TiSi_2$, with all other steps remain unchanged. The results show that a pseudo-crystalline $TiSi_2$ precursor layer can be formed by the laser annealing process. Upon subjecting this precursor layer to a second RTA step, fine-grained C54 $TiSi_2$ with low sheet resistance can be obtained.

11:30 AM Invited

Thin Film Metal Process Monitoring with Picosecond Ultrasonics: *Robert J. Stoner*[1]; Sailesh M. Merchant[2]; Guray Tas[3]; Christopher J. Morath[3]; [1]Brown University, Eng., Box D, Providence, RI 02912 USA; [2]Lucent Technologies, Orlando, FL 32819 USA; [3]Rudolph Technologies, 1 Rudolph Rd., Flanders, NJ 07836 USA

Picosecond ultrasonics is a non-contact optical measurement technique which has found wide acceptance in the semiconductor industry for monitoring thin film processes. Such processes may include single and multiple film deposition, etch and CMP. In this paper we briefly describe the measurement principles, and give examples of novel applications. We show how picosecond ultrasonics has been used to make film thickness and density measurement for ultrathin films including copper barrier layers such as reactively sputtered TaN. We also describe how the technique has been used to characterize RTP formed silicides. Finally we describe how picosecond ultrasonics has recently been applied to measuring complex patterned structures including copper damascene line and via arrays.

12:00 Noon Invited

Surface Modification of Thin Films by Gas Cluster Ion Beam Processing: *Lisa P. Allen*[1]; David B. Fenner[1]; John Hautala[1]; Allen Kirkpatrick[1]; Yan Shao[1]; [1]Epion Corporation, Billerica, MA 01821

Gas cluster ion beam processing of materials is an emerging technology for ultra-shallow surface modification with significant potential for applications in microelectronic technology. Formed from a pressurized gas expansion through a Laval nozzle into a vacuum, the condensed atomic clusters are held together by van der Waals forces. The method has produced beams of clusters from a variety of sources (Ar, N2, O2, CO2, N2O, SF6.), with cluster sizes ranging from several atoms to several thousands of atoms. Each gas cluster shares a single electrical charge such that a single cluster ion has high mass and momentum with a low energy (several eV) per atom. With the total energy of the gas cluster ion shared among several thousand atoms, the surface effect of the cluster ion impingement is contained within the first few atomic layers. Both experiment and simulation of the kinetic process show that sharp steps and asperities are rapidly eroded. Examples of gas cluster ion beam thin film smoothing of semiconductor, metallic, ceramic, superconductor, optical, and magnetic materials will be presented. Application for improved gate oxide integrity of silicon-on-insulator material will be shown.

Solution Concentration and Purification in Aqueous Processing

Sponsored by: Extraction & Processing Division, Aqueous Processing Committee

Program Organizer: Akram Alfantazi, Laurentian University, School of Engineering, Ontario P3E 2C6 Canada

Monday AM — Room: 221
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Akram Alfantazi, Laurentian University, Sch. of Eng., Sudbury, Ontario, Canada; David Dreisinger, UBC, Dept. of Mets. and Mats. Eng., Vancouver, Canada

8:30 AM

Application of Solvent Extraction to the Production of Two Component Composite Powders: *Junji Shibata*[1]; [1]Kansai University, Dept. of Chem. Eng., Suita, Osaka 564-8680 Japan

Some fundamental studies were carried out in order to produce composite powders comprising of two or three metals by using liquid-liquid extraction and develop the control technology of the particle size. The combination of copper and zinc, and cobalt and samarium was selected as the two component composite powders. Both stripping and crystallization occurred at the same time by emulsifying the organic phase loading the two metals extracted with Versatic Acid 10 and the aqueous solution containing oxalic acid. The effect of aqueous pH, oxalic acid concentration and agitation speed was examined on some properties such as crystallization speed, crystallization percent, particle size and shape of each metal powders obtained in this process. The analysis by using EDX showed that the two metals were distributed uniformly at the same ratio as the organic phase composition.

8:50 AM

Silver Ion Extraction Reactions by Dibutylthiourea (DBT) or Disulfiram (DSF) from Concentrated Chloride Solutions: *Ana Paula Paiva*[1]; [1]Faculdade de Ciências da Universidade de Lisboa, Departamento de Química e Bioquímica, Centro de Electroquímica e Cinética da Universidade de Lisboa (CECUL), Rua Ernesto de Vasconcelos, C8 Piso 5, Lisboa, 1749-016 Lisboa, Portugal

Silver separation from complex aqueous solutions usually involves processes such as precipitation as silver chloride or cemen-

tation. However, when high chloride concentrations are present in those aqueous phases, silver ion concentration in solution increases considerably, due to the formation of anionic chlorocomplexes. Therefore, investigation on profitable ways to recover significant quantities of silver remaining dissolved in highly concentrated chloride solutions may pass through the development of solvent extraction routes. In this work, the general pathways involving Ag(I) extraction by dibutylthiourea (DBT) or tetraethylthiuram disulfide (disulfiram, DSF), from concentrated acidic chloride solutions, are investigated. With this purpose, the dependence of Ag(I) distribution coefficient on ligand and chloride concentrations has been analysed. Furthermore, in order to check whether there is some protonation at any site of DBT or DSF after contact with adequate chloride phases, 1H NMR has also been used. The collected spectroscopic data suggest that anionic Ag(I) extraction reactions are not likely to occur. Based on the overall results achieved, proposals for the most probable Ag(I) extraction reactions are made.

9:10 AM

Purification of Cobalt Solutions by Ion Exchange: *Raj P. Singh*[1]; [1]OSRAM SYLVANIA, Chem. Rsch. and Dev., Hawes St., Towanda, PA 14848 USA

Lewatit TP207, a chelating resin with iminodiacetate functional group, is used for the purification of cobalt hexammine chloride solution for divalent cationic impurities (US Patent No. 5,154,757). The method is employed at commercial scale in OSRAM SYLVANIA, Inc. cobalt chemical production since 1992. It is observed that after long use in the purification of cobalt hexammine chloride solution, a large number of resin beads turns black from their original beige color. The purpose of this paper was to characterize these black-colored resin beads for their ion exchange behavior. The resin sample used in this work was an exhausted production TP207 resin containing large number of black-colored beads. The results indicated that blackened resin beads can be regenerated to almost full capacity and their ion exchange behavior for the purification of cobalt hexammine chloride solution is comparable to the new resin.

9:30 AM Break

9:50 AM

Arsenic and Selenium Removal from Aqueous Streams Using Pozzolan: P. Kerr[1]; *Rajendra Kumar Mehta*[1]; M. Misra[1]; C. Turrieta[2]; [1]University of Nevada, Univ. Ctr. for Environ. Sci. and Eng./199, 328 Appl. Rsch. Fac., Reno, NV 89557-0187 USA; [2]Turr Corporation, Fallon, NV USA

The conventional technologies for the removal of arsenic and selenium present as oxy-anions in aqueous streams and ground water are commonly based on adsorption/precipitation mechanisms. The inherent problem is that the adsorbents and precipitants are expensive, need regeneration and the contaminant adsorbed media often don't pass TCLP criteria. The use of natural pozzolan which was demonstrated to be quite effective in removing heavy metals and radionuclides in the past has recently been used in removing the oxy-anions of arsenic and selenium. The use of this material was demonstrated to achieve the concentration of arsenic to be as low as 5ppb and the pozzolan passed the TCLP criteria. Adsorption isotherms were generated and the effect of various parameters such as material loading and contact time were investigated in the batch tests. Finally, on the basis of stagewise batch tests, a flowsheet was developed to remove arsenic and selenium levels to the MCL levels from aqueous streams having the contaminant levels in the ppm range.

10:10 AM

Recovery of Cobalt, Gold and Bismuth from the Nico Deposit, NWT, Canada-Part III: Recovery of Cobalt: *A. Mezei*[1]; C. J. Ferron[1]; R. E. Goad[1]; [1]Lakefield Research Limited, Lakefield, Ontario Canada

A process has been developed to recover cobalt, gold and bismuth from the NICO deposit in Canada. The process consists of a bulk rougher flotation followed by an optional selective flotation of the bismuth. Cobalt is leached by pressure oxidation at 97% efficiency, producing a pregnant solution suitable for further processing. About 38% of the bismuth is recovered in a selective concentrate, where-from it is recovered by ferric chloride leaching followed by cementation, at overall efficiencies exceeding 98%. The gold is recovered from the cobalt and bismuth leach residues by cyanidation, at 95% efficiency. Previous work described the results of the leaching of the concentrates (Part I) and the recovery of bismuth (Part II). This work describes the results of the cobalt recovery testwork and the resulting conceptual flowsheet. The sulfur dioxide-oxygen system was the most efficient for the precipitation of iron and arsenic from cobalt bearing pregnant leach solutions, producing purified solutions assaying <1 mg/L As and <8 mg/L Fe. The efficiency of cobalt solvent extraction (from the resulting purified PLS containing about 1 g/L Co) ranged from 91 to 98%; the co-extractions ranged from 88 to 100% for copper, from 28 to 33% for magnesium, from 6 to 7% for calcium and from 96 to 98% for zinc. Sulphide precipitation processes (using either sulfur dioxide/elemental sulfur or sodium sulfide) produced residual copper concentrations in the cobalt strip solution ranging from 0. 1 to 2 mg/L Cu. About 90% of the calcium and magnesium were separated by scrubbing the organic with cobalt sulfate, producing residual concentrations of 12 mg/L Ca and from 180 to 330 mg/L Mg in typical feed solutions directed to cobalt carbonate precipitation tests. Cobalt carbonate precipitation efficiencies ranged from 75 to 78% when calcium and magnesium co-precipitations were restricted from 8 to 17% and 0. 3 to 0. 4%, respectively. The limits required by most stringent specifications on commercial grade cobalt carbonate products were met or exceeded.

10:30 AM

Study of the Batch Purification Process of Solutions from Leaching of Used Alkaline Batteries: *Cleusa Cristina Bueno Martha de Souza*[1]; Jorge Alberto Soares Tenório[1]; [1]Polytechnic School-University of São Paulo, Dept. of Metall. and Matls. Eng., Av. Prof. Mello Moraes 2463, São Paulo, SP 05508-900 Brazil

Hydrometallurgical techniques for processing metals are becoming an efficient method for recovering metals. Although batteries' manufacturers have got rid of mercury content, some efforts have been made for recovering other metals enclosed in the battery formula. This paper discusses the bench scale experiments in the purification process of leached solutions from alkaline spent batteries. After leaching process using sulfuric acid as leachant, the solutions containing Fe, Cd, Pb, Mn, Zn, K were neutralized to precipitate iron hydroxide. In a second step, the purification experiments were carried out with excess of zinc dust to remove Cd while Mn and Zn still remain in the solution to be recovered in an electrolysis stage. The purification experiments procedures were conducted at different temperatures and times to find out properly conditions from the viewpoint of metals removal. After filtration the solutions were submitted to chemical analysis to certify the metals content extracted.

Structural Biomaterials for the 21st Century: Metallurgy of Biocompatible Metallic Materials

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

Program Organizers: Mitsuo Niinomi, Toyohashi University of Technology, Department of Production Systems Engineering, Toyohashi 441-8580 Japan; Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; Henry E. Lippard, Allvac R&D, Monroe, NC 28110 USA; Toru Okabe, Baylor College of Dentistry, Texas A&M Health Science Center, Department of Biomaterials Science, Dallas, TX 75246 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Monday AM Room: 229
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Toru Okabe, Baylor College of Dentistry, Texas A&M Htlh. Sci. Cen., Dept. of Biomats. Sci., Dallas, TX 75246 USA

8:30 AM Keynote

Recent Biocompatible Metallic Materials: *Mitsuo Niinomi*[1]; [1]Toyohashi University of Technology, Product. Sys. Eng., 1-1 Hibarigaoka Tempaku-cho, Toyohashi 441-8580 Japan

Metallic biocompatible materials are mainly used for replacing failed hard tissue. The main metallic biocompatible materials, that is, metallic biomaterials, are stainless steels, cobalt-based alloys, titanium and its alloys. Recently, titanium alloys are getting much attention for application as biomaterials. The various kinds of new high strength alpha+beta and low modulus beta type titanium alloys composed of non-toxic elements like Nb, Ta, Zr, etc. are being developed for biomedical applications. These new alloys can solve the problems with toxicity of alloying elements and lack of mechanical biocompatibility of conventional titanium alloys, like Ti-6Al-4V. Recent research and development in other metallic alloys, like stainless steels, cobalt-based alloys, etc. will be also discussed.

9:00 AM Invited

Isothermal Aging Behavior of Ti-29Nb-13Ta-4. 6Zr New Beta Alloy for Medical Implant: *Masahiko Ikeda*[1]; Shin-ya Komatsu[1]; Isao Sowa[2]; Mitsuo Niinomi[3]; [1]Kansai University, Dept. of Matls. Sci. and Eng., 3-3-35, Yamate-cho, Suita, Osaka 564-8680 Japan; [2]Student of Kansai University; [3]Toyohashi University of Technology, Dept. of Product. Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580 Japan

Ti-29Nb-13Ta-4. 6Zr alloy comprised of highly biocompatible metals was developed by Prof. Ninomi and his group of Toyohashi University of Technology. It is very important to establish heat treatment conditions to develop appropriate microstructures and mechanical properties. However, aging behavior is not systematically and fully investigated. In this study, the aging behavior of the Ti-29Nb-13Ta-4. 6Zr alloy quenched from 1033K was investigated employing electrical resistivity and Vickers hardness (HV) measurement, optical microscopy and X-ray diffraction (XRD). On 573K aging, resistivity at room and liquid nitrogen temperatures increased up to 120ks and 30ks, respectively. Then, both resistivities decreased with continued aging. HV started to increase from 0. 06ks and isothermal omega phase was identified at 300ks aging by XRD. On 773K aging, an incubation period for resistivity change at both temperatures of about 6ks was observed, following which resistivity decreased. An incubation period for HV change was extended up to 12ks. HV stared to increase from 30ks and precipitated alpha was observed by optical microscopy and reflections of alpha phase were identified by XRD, whereas no reflections of isothermal omega phase were identified during whole aging time at 773K. It is considered that isothermal omega phase was precipitated by aging at a temperature below 773K.

9:20 AM Invited

Shape Memory Ti-Nb-Sn Alloys: *Shuji Hanada*[1]; Naoya Masahashi[1]; Sadao Watanabe[1]; Kei Nitta[1]; Hideki Hosoda[1]; [1]Tohoku University, Instit. for Matls. Rsch., Katahira 2-1-1, Aoba-ku, Sendai, Miyagi-ken 980-8577 Japan

Shape memory effects of Ti-Nb-Sn alloys consisting of biologically safe elements were investigated to develop an alternative to shape memory NiTi alloys containing the harmful element Ni. As-quenched Ti-Nb-Sn alloys undergoes a martensitic transformation from bcc to orthorhombic. The martensitic transformation start temperature (M_s) decreases by about 50°C with 1 at% increase of Nb content and by about 100°C with 1 at% increase of Sn content. Ti-10~18at%Nb-4~6at%Sn alloys exhibit a shape memory effect depending on alloy composition and deformation temperature. Tensile strain given at temperatures below the reverse transformation finish temperature (A_f) is recovered on heating above A_f completely or partially, depending on deformation temperature and given strain. A retained strain of 3. 5% at a maximum after tensile deformation is recovered on heating. Shape memory effects of Ti-Nb-Sn alloys will be discussed in comparison with Ti-Ni alloys.

9:40 AM

Structure and Properties of Binary Ti-Mo Alloys: *Jiin-Huey Chern Lin*[1]; Wen-Fu Ho[1]; Chien-Ping Ju[1]; [1]National Cheng-Kung University, Dept. of Matls. Sci. and Eng., #1 Da-Sha Rd., Tainan, Taiwan

Structure and properties of a series of binary Ti-Mo alloys with molybdenum contents ranging from 6 to 20 wt% have been investigated. Experimental results indicated that the hexagonal alpha phase c.p. Ti exhibited a feather-like morphology. When 6 wt% Mo was contained, a fine, acicular martensitic structure of orthorhombic alpha phase was observed. When 7.5 wt% Mo was contained, the entire alloy was dominated by the martensitic alpha structure. When Mo content increased to 10 wt% or higher, the retained beta phase became the only dominant phase. The bending strength of Ti-7.5Mo was similar to those of Ti-15Mo and Ti-13Nb-13Zr, and higher than c.p. Ti by nearly 60%. The bending modulus of the alpha-dominated Ti-7. 5Mo alloy was lower than Ti-15Mo by 22%, than Ti-6Al-4V by 47%, than Ti-13Nb- 13Zr by 17%, and than c.p. Ti by 40%.

10:00 AM Break

10:10 AM

Thermomechanical Analysis of Ti40Ta and Ti50Ta Alloys: *Celina R. Ortiz*[1]; Rudy Villa[1]; Gabriela Gonzalez[1]; Elizabeth Trillo[1]; Stephen W. Stafford[1]; Lawrence E. Murr[1]; [1]The University of Texas at El Paso, Dept. of Metall. and Matls. Eng., 500 W. University Ave., El Paso, TX 79968 USA

Currently, titanium-tantalum is being studied as an alternative to the popular biomaterial Ti6Al4V. This material has demonstrated possible toxic metal ion release. Preliminary studies have shown Ti40Ta and Ti50Ta to exhibit high strengths and excellent corrosion resistance when subjected to various heat treatments. Ti50Ta aged at 400°C for 50 hours and at 500°C for 50 hours exhibits a tightly woven tweed structure as seen through transmission electron microscopy. The second phase particles, exhibiting a short needle-like morphology, have aligned in the tweed structure resulting in an increase in hardness. The goal of this research is to find a similar optimum heat treatment for the Ti40Ta samples which have not shown an increase in hardness during preliminary testing. Once the heat treatment has been optimized, potentiodynamic testing will be performed to determine the corrosion resistance of both the Ti40Ta and Ti50Ta. This research is funded by a General Services Administration (GSA) Grant #PF-90-018.

10:30 AM

Effect of Heat Treatment on the Microstructure and Hardness of a Precipitation Hardenable Nickel-Chromium Base Denture Alloy: E. E. Al Wakeel[2]; *R. A. Fournelle*[1]; V. B. Dhuru[3]; J. M. Toth[4]; [1]Marquette University, Mech. and Indust. Eng., P.O. Box 1881, Milwaukee, WI 53201-1881 USA; [2]Mansoura University, Fac. of Dentist., Mansoura Egypt; [3]Marquette University, Sch. of Dentist., P.O. Box 1881, Milwaukee, WI 53101-1881 USA; [4]Medi-

cal College of Wisconsin, Ortho. Surgery, 8700 W. Wisconsin Ave., Milwaukee, WI 53226 USA

The effect of solution heat treatment and subsequent aging heat treatments on the microstructure and hardness of an as-cast nickel-chromium base denture alloy (Ticonium Premium 100 "Hard") were studied by light microscopy, scanning electron microscopy, transmission electron microscopy and x-ray diffraction. The as-cast alloy exhibited an inhomogeneous dendritic structure consisting of fcc gamma phase dendrites with an inhomogeneous distribution of gamma prime precipitates and an interdendritic eutectic mixture of fcc gamma phase and NiBe. This structure had a hardness of 336 HV. Solution treatment of the alloy for 60 minutes at 1,050°C resulted in the dissolution of the gamma prime precipitates in the gamma dendrites as well as their homogenization. Solution treatment also resulted in a decrease in hardness to 200 HV. Subsequent aging at 600°C, 750°C and 900°C resulted in the formation of fine uniform distributions of gamma prime precipitates in the gamma phase dendrites and a consequent increase in hardness. A maximum hardness of 359 HV was attained after 10 hours of aging at 750°C.

10:50 AM

Effect of Heat Treatment on the Strength and Ductility of a Precipitation Hardenable Nickel-Chromium Base Denture Alloy: S. Suryawanshi[2]; V. B. Dhuru[2]; *R. A. Fournelle*[1]; [1]Marquette University, Mech. and Indust. Eng., P.O. Box 1881, Milwaukee, WI 53201 USA; [2]Marquette University, Sch. of Dentist., P.O. Box 1881, Milwaukee, WI 53201 USA

The effect of annealing an as-cast nickel-chromium base denture alloy (Ticonium Premium 100) on its microstructure, tensile properties, hardness and fracture behavior was studied by light microscopy and scanning electron microscopy. The as-cast alloy exhibited an inhomogeneous dendritic structure consisting of fcc gamma phase dendrites with an inhomogeneous distribution of gamma prime precipitates and an interdendritic eutectic mixture of fcc gamma phase and NiBe. This structure had a yield strength of 760 M/Pa, a tensile strength of 825 MPa, a percent elongation of 3. 4% and a hardness of 348 HV. Annealing at 600°C for various times up to 10 hours resulted in little change in microstructure and mechanical properties. Annealing at 750°C resulted in precipitate formation in the center of the dendrites, a small increase in strength and hardness and no change in ductility. Annealing at 900°C resulted in a coarsening of the as-cast precipitate structure, a decrease in the strength and hardness and an increase in the ductility. Annealing at 1,050°C resulted in the dissolution of the as-cast precipitate structure, a large decrease in strength and hardness and a large increase in ductility. The fracture surfaces of the as-cast alloy and alloy annealed at 600°C and 750°C exhibited brittle features such as clevage through intermetallic particles. Alloy annealed at 900°C and 1,050°C exhibited ductile dimple rupture. Annealing the as-cast alloy for 1 hour at 1,050°C, quenching and then aging at 750°C for 1 hour, resulted in a fine distribution of precipitates in the dendrites, higher strength and hardness than the as-cast alloy, but much improved ductility.

11:10 AM

Effect of Alloying Addition on Structure and Properties of Ti-7. 5 Mo Alloy: *Chien-Ping Ju*[1]; Wen-Fu Ho[1]; Jiin-Huey Chern Lin[1]; [1]National Cheng-Kung University, Dept. of Matls. Sci. and Eng., #1 Da-Sha Rd., Tainan, Taiwan

The present work is a study of the effect of 1 wt% addition of a series of alloying elements on the structure and properties of the Ti-7.5Mo system. The results indicate that the phase/crystal structure, microstructure, and mechanical properties of Ti-7.5Mo can be significantly changed by small amounts of alloying addition. Ti-7. 5Mo is a typical orthorhombic alpha"-phase alloy. When 1 wt% Zr or Nb is added, its fine, acicular martensitic structure of alpha" phase remains, microhardness increases 25-29%, bending strength increases 13-21%, while modulus only slightly increases. When Cr is added, a significant amount of equi-axed, beta phase is retained and microhardness, strength, and modulus all increase. When Fe is added, the equi-axed beta phase becomes the only dominant phase and its hardness, strength, and modulus all become the highest among all Ti alloys in this study.

NOTES

MONDAY PM

2001: An Odyssey of Materials in Space: Advanced Systems and Materials for Space: Materials with High Structural Efficiency

Sponsored by: Extraction & Processing Division, Light Metals Division, Aluminum Committee, Copper, Nickel, Cobalt Committee

Program Organizers: Daniel B. Miracle, Wright Laboratory, Materials Directorate, Building 655, WPAFB, OH 45433 USA; Enrique V. Barrera, Rice University, Department of Mechanical Engineering and Materials Science, Houston, TX 77251 USA

Monday PM Room: 223
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: TBA

2:00 PM Invited

Advanced Space Systems and Materials Needs at Boeing: *Cliff Bampton*[1]; John Halchak[1]; Bill Bozich[2]; [1]The Boeing Company, Rocketdyne Propu. & Power, Canoga Park, CA USA; [2]The Boeing Company, Space & Commun. Sys., Huntington Beach, CA USA

Boeing legacy organizations have a long history of involvement in all the major United States' space programs. More than in most industries, this has required development and insertion of new and enabling technologies concurrently with operational systems development. This has always been especially challenging for new materials and processes since their traditional development cycles are considerably longer than a systems development cycle. This has led to many critical on-the-job learning experiences where solutions to unanticipated problems had to be engineered in very short time frames. This presentation will describe some past, recent and current examples for launch system propulsion and vehicle systems. Implications of the current trend toward avoidance of the traditional test-fail-fix development paradigm for new materials and processes development and insertion will be discussed.

2:30 PM Invited

Finding Focus for the Future Space Odyssey: Materials Challenges Facing the Space Sector: *Diana Carlin*[1]; [1]AF Research Laboratory, Matls. and Manufact. Direct., Wright-Patterson AFB, OH 45433 USA

Abstract text unavailable.

3:00 PM Invited

Materials Needs for Liquid Rocket Engines: *J. S. Shelley*[1]; [1]AF Research Laboratory, Prop. Direct., AFRL/PRRE, 4 Draco Dr., Edwards AFB, CA 93524-7160 USA

Several Government programs, including the Integrated High Payoff Rocket Propulsion Technology (IHPRPT) project and NASA Second and Third Generation Shuttle projects, are developing new materials and processes for application to Liquid Rocket Engines (LREs). The continuing drive for increased system performance and reliability with decreased cost is motivating the need for new materials and repeatable processes in the rocket industry. The extremes of the LRE environment is challenging for any material, components are complex, and reusability requirements make accurate life prediction a necessity. This paper will discuss the liquid rocket engine environment and specific design challenges that create challenges for materials. Recent progress in applying new materials will be presented. The research needs of the rocket community will also be presented.

3:30 PM Break

3:40 PM Invited

Processing and Qualification of Al-Based Materials for Space Applications: *Mark van den Bergh*[1]; B. Braun[2]; K. Segal[3]; C. Bampton[4]; [1]DWA Al Composites, Chatsworth, CA USA; [2]Naval Research Laboratory, Washington, DC USA; [3]NASA Goddard Space Flight Center, Greenbelt, MD USA; [4]The Boeing Company, Rocketdyne Propulsion and Power, Canoga Park, CA USA

Although Discontinuously Reinforced Aluminum (DRA) is not a pervasive material for space, it has been successfully inserted into a number of structural applications ranging from deck panels, longerons and structural booms. DRA was selected for reasons of dimensional stability, high specific stiffness, and reduced structural weight. Because knowledge of these applications is not widely disseminated, it is worthwhile to review the manufacturing and qualification efforts undertaken, and reinforce the systems level pay-offs and lessons learned. Nanophase aluminum is processed to possess very fine grain size, high strength, ductility, and toughness. High strength results from the combined contributions of fine grain size, alloying, and deformation substructure. Nanophase aluminum has been earmarked for applications in rocket turbo-machinery. Efforts are underway to develop scaled-up, robust processing of powder and billet, with an emphasis on affordability. This section of the presentation will focus on present processing capabilities and highlight the improvements necessary to assure technology progression to insertion of flight qualified hardware.

4:10 PM

Discontinuously-Reinforced Aluminum for Elevated Temperature Applications: *Awadh B. Pandey*[1]; Kevin L. Kendig[2]; Daniel B. Miracle[2]; Thomas J. Watson[3]; [1]Pratt & Whitney, Liquid Space Prop., P.O. Box 109600, M/S 706-06, West Palm Beach, FL 33410-9600 USA; [2]Air Force Research Laboratory, Matls. and Manufact. Direct., 2230 10th St. Ste 1, Wright-Patterson Air Force Base, Dayton, OH 45433 USA; [3]Pratt & Whitney, Matls. and Process. Eng., 400 Main St., East Hartford, CT 06108 USA

Discontinuously-reinforced aluminum (DRA) composites have been used in aerospace structures such as Ventral Fins and Fan Exit Guide Vanes owing to their superior specific stiffness, specific strength, wear resistance, and thermal resistance as compared to the unreinforced aluminum alloys. DRA materials are now being considered for elevated temperature applications. The matrix alloy has crucial role on the ambient and elevated temperature mechanical properties of DRA. The matrices in the conventional DRA materials are commercial precipitation hardened aluminum alloys which can be used only up to 150°C due to rapid coarsening of strengthening precipitates. The past effort on the development of high temperature aluminum alloys showed limited success. Therefore, it is important to design an alloy which can resist coarsening at elevated temperatures. In this paper, a new matrix alloy based on Al-Mg-Sc with improved thermal stability is proposed. The monolithic alloy and the composite with 15 volume percent SiC particles were processed using a powder metallurgy approach. The influence of processing parameters on the microstructures and mechanical properties of the monolithic and composite materials is discussed. The thermal stability of the monolithic and composite materials is also evaluated. The strength of the composite is discussed in terms of solid solution strengthening, Orowan strengthening, and load transfer models.

4:30 PM

Deformation and Strengthening Mechanisms of an Al-Mg-Sc Alloy from Cryogenic to Elevated Temperatures: *Kevin L. Kendig*[1]; Daniel B. Miracle[1]; Awadh B. Pandey[2]; [1]U. S. Air Force, Mets. Brnch., AFRL/MLLM Bldg. 655, 2230 Tenth St., Wright-Patterson AFB, OH 45433-7817 USA; [2]Pratt & Whitney, Liquid Space Prop., P.O. Box 109600, M/S 706-06, West Palm Beach, FL 33410-9600 USA

An Al-Mg-Sc alloy is being developed to meet high performance goals in rocket engine applications. Very promising specific strength has been demonstrated. Careful microstructural investigations have been done to elucidate strengthening and deformation mechanisms over a range of temperatures, from -160°C to 200°C. Available strengthening models were used to calculate expected strengthening

contributions due to solid solution, grain size, and fine Al_3Sc precipitates. Observations of the microstructure provide the basis for application of grain size and precipitate strengthening models. Microstructural information, theoretical calculations, and measured material properties will be presented and discussed.

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Amorphous Al Alloys for High Specific Strength: *Michael Scott*[2]; Kevin Kendig[1]; Oleg Senkov[2]; Daniel Miracle[1]; Tom Watson[3]; Ken Davis[4]; [1]AF Research Laboratory, Matls. and Manufact. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH USA; [3]Pratt and Whitney, Matls. Lab., E. Hartford, CT 06108 USA; [4]DWA Al Composites, Chatsworth, CA USA

Many applications in advanced liquid-fueled rocket propulsion systems require enabling levels of specific strength. Amorphous Al alloys (AAA) provide a promising approach toward achieving these goals. An Al-Ni-Y alloy being considered for advanced space applications has been produced via inert gas atomization. The powder was outgassed and extruded at 450°C. The structure of the as-produced powder was characterized and compared with the structure following the thermal exposure associated with outgassing and extrusion. The mechanical properties of the extruded alloy have been measured. The observations and measurements will be presented and discussed.

5:10 PM

Hydrogen Effects on Material Behavior: *Jerry W. Sheldon*[1]; Jeff D. Haynes[1]; Steven J. Gentz[2]; [1]United Technology Corporation, Pratt and Whitney Liquid Space Propulsion, East Hartford, CT USA; [2]NASA, Marshall Space Flight Center, Huntsville, AL USA

IN 100 (PWA-SP 1074), a powder metallurgy nickel base super alloy is used in the Space Shuttle Main Engine (SSME) High Pressure Fuel and Oxidizer Turbopumps (HPFTPs and HPOTPs). This material is exposed to a variety of severe thermal and environmental conditions throughout the start transient, main stage, and shut down cycles of the SSME. Previous experience for most materials indicated that one of the most life limiting exposures is to ambient temperature high pressure gaseous hydrogen. Recent developmental testing of the HPFTP revealed hydrogen assisted cracking in the Turbine Housing. Thermal and structural analyses, supported by instrumented hot fire units, suggested the cracking area of the housing was being exposed to 300 to 500° F high pressure hydrogen. A characterization program was initiated to increase the understanding of IN 100 in this temperature and environmental regime. This paper presents the findings of this material property characterization investigation and of the examination of a hydrogen barrier coating and an alternative thermal treatment to improve the hydrogen properties of IN 100. Data presented will show that hydrogen rich steam has a greater effect on properties than pure hydrogen and that the minimum strength properties occur at a temperature greater than ambient for IN 100.

Alumina & Bauxite: Bayer Process Chemistry

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

Monday PM — Room: 217
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Jacques Mordini, Aluminium Pechiney, Gardanne 13541 France

2:00 PM Opening Remarks

2:10 PM

The Composition of DSP Formed under Predesilication and High Temperature Bayer Digestion Conditions: *Peter Graham Smith*[1]; Russell Pennifold[1]; Alan Kane[2]; [1]AJ Parker CRC for Hydrometallugy, CSIRO Mins., P.O. Box 90, Bentley, WA 6982 Australia; [2]AJ Parker CRC for Hydrometallugy, Queensland Alumina Ltd., Parsons Point, Gladstone, Queensland 4680 Australia

The composition of Desilication Product (DSP) has been determined as a function of liquor composition from which it was made, under the conditions of predesilication and high temperature digestion characteristic of the QAL refinery. DSPs were formed in synthetic liquor by reacting kaolin with lime and gibbsite. The soda content of DSP is related to lime charge, but is unrelated to liquor composition. Carbonate and chloride contents are predicted well by regression models. The low levels of sulphate in these preparations meant that this species is less dominant than may otherwise have been expected (Riley et al.,1999). DSP formed under predesilication conditions was exclusively of the hydroxysodalite type whereas after subsequent digestion, additional compounds identified were hydrogarnets with a limited range of silica contents, and a form of carbonate containing sodium calcium cancrinite. Semi-quantitative estimates of the phase composition indicated that under conditions of medium to high lime charge and high carbonate in solution, DSP is dominated by the cancrinite, heavily influencing its chloride and sulphate sequestering capacity.

2:40 PM

Reduction of Sodium Aluminosilicate Scale in Bayer Plant Heat Exchangers: A. Gerson[1]; *Jonas Addai-Mensah*[1]; R. Jones[1]; [1]University of South Australia, Ian Wark Res. Inst., Mawson Lakes, Adelaide 5095 Australia

Costly and unwanted precipitation of sodium aluminosilicate scale on steel heat transfer surfaces in contact with spent Bayer liquor occurs during alumina refining. Although our knowledge and understanding of the mechanisms by which the nucleation and growth of the scale occur are fairly advanced, complete suppression of the fouling process remains to be achieved. In this paper the results of investigations involving the use of chemical additives to control sodium aluminosilicate precipitation fouling of stainless steel surfaces under alumina refinery processing conditions are reported. It is shown that varying degrees of reduction in scale coverage may be achieved by using a variety of additives containing different functional groups. It appears that reduction in fouling largely occurred by additive mediated crystal steel substrate interfacial layer modification.

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Some Aspects of Calcium Chemistry in the Bayer Process: *Steven Philip Rosenberg*[1]; Darrel James Wilson[1]; Catherine Ann Heath[1]; [1]Worsley Alumina Pty., Ltd., Process Chem. Grp., P.O. Box 344, Collie, Western Australia 6225 Australia

Lime is used in vast quantities in alumina refineries throughout the world and is often regarded as something of a universal cure for many of the ills of the Bayer process. However, like many popular remedies, knowledge of how it works is quite sparse, and often contradictory. Fortunately, this situation is now changing, as a few recent studies have begun to reveal some of the complex solution and solid-phase chemistry that exists between calcium and the many species present in Bayer refinery liquor streams. In this paper, the findings of some studies undertaken in this area at the research laboratories of Worsley Alumina are described. These findings are discussed in terms of their relevance to the common applications of lime in the Bayer process and some explanations are offered for the sometimes confusing behaviour of calcium in these systems.

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Improved Causticisty and Lime Efficiency in Causticisation: *Gerald Ian Roach*[1]; G. Charmaine de Witt[1]; Glenn Reid[1]; [1]Alcoa World Alumina, Tech. Del. Grp., Cockburn Rd., Kwinana, Western Australia 6167 Australia

The level of causticity that can be obtained in causticising Bayer liquors to remove carbonate and regenerate caustic is limited by thermodynamics. To achieve the maximum causticity (the thermodynamic equilibrium) normally requires the addition of excess lime and a consequent lime efficiency of around 50%. An improved method of causticisation has been developed which enables higher

causticity of liquors to be obtained, together with higher lime efficiency. The causticity of the liquor can be increased to more than 0. 05 TC/TA above the thermodynamic equilibrium at a causticising TA of 110g/l, and with lime efficiencies close to 100%. Such improved lime efficiency not only reduces lime cost, but also alumina losses and residue alkalinity because of the alkaline nature of the tricalcium aluminate formed in the conventional process. The reaction chemistry, which is different to conventional causticisation, will be described. The process has been piloted and is currently undergoing full scale testing.

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Industrial Experience of Polishing Filtration Performance–Improvement and Interpretation: *Nicolas Mugnier*[1]; Phillipe Clerin[1]; Joel Sinquin[2]; [1]Aluminium Pechiney, Centre de Recherche, P.O. Box 54, Gardanne Cedex, France; [2]Aluminium Pechiney, Usine de Gardanne, P.O. Box 62-13541, Gardanne Cedex, France

In alumina refineries, the performance of the polishing filtration has been widely studied. The work presented in this paper aims at elucidating the mechanisms of the TCAS or CAIS synthesis in order to set-up an optimal set-point. Several points were found to be crucial to the synthesis of the best precoat, made from slaked lime and pregnant liquor. The crystal size distribution of the precoat and its evolution with respect to filtration time were the two parameters studied. It was found that the crystal size distribution obtained and the corresponding specific resistance are highly dependent on the chemical process, and more specifically on the batch operation as opposed to continuous operation. Moreover, some industrial data indicate that the SiO2 content of the liquor has a great impact on the filter productivity. Therefore, the SiO2 content of the precoat CAIS, synthesized in various process conditions, is analyzed with respect to time. This work was followed by industrialization of the process in Gardanne (France) and ADG (Greece) refineries. The increase in overall performance before and after start-up was monitored on traditional horizontal Kelly filters and the new self-cleaning filters Diastar(Gaudfrin). The corresponding figures are mentioned in the paper.

5:00 PM

Alumina Particle Breakage in Attrition Test: *Philippe Clerin*[1]; Veronique Laurent[2]; [1]Aluminium Pechiney, Centre de Recherche, P.O. Box 54-13541, Gardanne, Cedex, France; [2]Aluminium Pechiney, Pechiney Centre de Recherche de Voreppe, Parc Economique Centr'Alp, P.O. Box 27-38340, Voreppe, France

Smelter grade alumina particle strength has a strong influence on smelter cell operations. Commercial alumina particles exhibit a wide range of behavior when submitted to the conventional attrition test. The influence of alumina particle size distribution on the attrition index value has been first studied. A method is then proposed to determine the specific strength of each of six particle fractions constituting the alumina powder. Each particle size fraction is impregnated with aqueous solutions of different salts and heat treated. After submitting the remixed powder to attrition test, each resulting fraction is analysed by ICP spectroscopy. At this time each cation is used as a "flag" to identify the origin of the particle size fractions. We tentatively express the strength behavior in terms of distribution and grinding functions. Results obtained with 6 alumina samples issued from different plants with various modes of precipitation and calcination are discussed.

Aluminum Reduction Technology: New Processes and Refractory Materials

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

Monday PM — Room: 206-207
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Jomar Thonstad, Norwegian University of Science and Technology, Dept. of Matls. Tech. and Electrochem., Trondheim N-7491 Norway

2:00 PM

AP50: The Pechiney 500 kA Cell: *Claude Vanvoren*[1]; Jean Luc Basquin[1]; Pierre Homsi[2]; Thierry Beheregaray[3]; [1]Aluminium Pechiney, LRF Tech. Rsch. Ctr., BP 114, Saint Jean de Maurienne 73300 France; [2]Aluminium Pechiney, Ctr. de Tech. Aluval, Zi de Voreppe Moirans, BP 7, Voreppe 38340 France; [3]Pechiney Nederland NV, PNL, BP 49, AA Vlissingen, Oost, Nederland 4380 France

As a result of ten years of extensive tests carried out on three prototypes, Pechiney has developed a new generation of reduction cell. Designed with the help of advanced computer models to achieve magnetic stability and optimize potroom ventilation and temperatures, these prototype cells were progressively boosted with forced shell ventilation and modified anode assemblies allowing operation at 500kA with a current efficiency of 95%. The AP50 cell process control features fully integrated automatic measurement of bath temperature and height for enhanced thermal stability. Improved alumina feeding almost totally eliminates anode effects, ensuring a CO2 equivalent emission level close to the anode consumption CO2 emission. The AP50 technology with a single potline of 336 cells having an annual output of 460 kt, will increase productivity per employee by 35% and reduce investment cost by 15% as compared to a 2 potline AP30 smelter.

2:30 PM

Modeling Vapor Recovery in a Carbothermic Aluminum Process: *Vianey Garcia-Osorio*[1]; Tor Lindstad[1]; B. Erik Ydstie[1]; [1]Carnegie Mellon University, Dept. of Chem. Eng., Pittsburgh, PA 15213-3890 USA

One of the problems in the carbothermic aluminum process is the substantial loss of aluminum compounds as vapors due to the high temperatures in the process. In order to solve this problem a Vapor Recovery Unit (VRU) is proposed. In this unit operation the aluminum vapor undergoes a series of heterogeneous non-catalytic reactions forming solid products. In this work a model for the simulation of this unit was developed which includes heat and material balances as well as heterogeneous non-catalytic reactions. The model leads to a system of Differential Algebraic equations; its solution was implemented in Matlab giving as a result a stage-by-stage solid-fluid reaction model.

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Aluminum Reduction via Near Room Temperature Electrolysis in Ionic Liquids: *Banqiu Wu*[1]; Ramana G. Reddy[2]; Robin D. Rogers[1]; [1]The University of Alabama, Ctr. for Green Manufact., Tuscaloosa, AL 35487 USA; [2]The University of Alabama, Dept. of Metall. and Matls. Eng., Tuscaloosa, AL 35487 USA

Experimental studies on aluminum reduction via electrolysis in ionic liquids at near room temperature were carried out. Anhydrous aluminum chloride was used as raw material. The electrolyte was made from $AlCl_3$ and butyl-methylimidazolium chloride. X-ray diffraction and micro-image analyzer were used for the product characterization. Aluminum was electrowinned at copper cathode and chlorine gas was released at graphite anode. Experimental temperature was in the range of 100-150°C. Cathode current density was

about 400 A/m^2. Aluminum deposition with thickness of 0. 1-0. 2 mm was obtained.

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Basic Investigations on the Optimization of the Refractory Barriers in Aluminium Electrolytic Cells: *Wolfgang Walz*[1]; [1]BURTON GmbH, Barkhausener StraBe 55, Melle, Buer 49328 Germany

The primary aim of the present work is to indicate the principles of operation of refractory materials of widely varying composition in their application as barrier blocks in aluminium electrolytic cells. This will take into consideration both refractory products which have already been used as barrier materials or derive from other fields of application in metallurgy or the chemical industry, and also those which have recently been developed especially to meet the present requirements. In particular the relationship of the parameters relevant to their use and quality-such as resistance to cryolite and aluminium melts, thermal conductivity-of the chemical-mineralogical composition and the structural nature of the refractory materials are demonstrated and discussed with reference to measured data from laboratory tests. On the basis of these experimental results, conclusions are drawn on their use in practice with the aim of guaranteeing maximum periods of service life under largely unchanged refractory material properties. In consideration of these criteria suggestions are made for the optimization of the barrier systems at the material and structural design levels with allowance for the costs incurred.

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Refractory Requirements for the Lining of Aluminum Reduction Cells: *Claude Allaire*[1]; Roger Pelletier[1]; Ole-Jacob Siljan[2]; Alton Tabereaux[3]; [1]Ecole Polytechnique of Montreal, Phys. and Matls. Sci. Dept., 8475 Christophe-Colomb Rd., Montreal, Quebec H2M 2N9 Canada; [2]Norsk Hydro ASA, Rsch. Ctr. Porsgrunn, P.O. Box 2560 Rsch. Ctr. HPI, Porsgrunn 3907 Norway; [3]Alcoa, Rsch. Dept., 4276 Second St., Muscle Shoals, AL 35661 USA

Aluminum reduction cell potlinings are deteriorated in service by the action of the cell chemicals, which include mostly molten bath and sodium vapor. Two different types of corrosion test were introduced in the past to evaluate refractories for this application: crucible tests and electrolysis bath tests. While the former do not incoporate the action of sodium vapor during the test, the latter do not adequately reproduce the action of the molten bath. From these two types of test, contreverse conclusions regarding the effect of the alumina:silica ratio of aluminosilicate refractories were published in the litterature. This paper presents a noval testing procedure which permits to expose refractories to controlled amounts of sodium relative to molten bath during the test. Corrosion test results from this new procedure are presented and the effect of the refractories alumina:silica ratio is discussed.

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Silicon Nitride Bonded Silicon Carbide Refractories in Aluminum Reduction Cells: *Eric Jorge*[2]; *Stephen M. Kubiak*[1]; *Jacques Schoennahl*[2]; *Olivier Marguin*[2]; [1]Saint Gobain Industrial Ceramics, High Performance Refractories, P.O. Box 15136, Worcester, MA 01615-0136 USA; [2]Saint Gobain Industrial Ceramics, High Perf. Refract., Rue Jean Monnet, Z. A. C. DUM. I. N. 84306, Cavaillon Cedex, France

Si3N4 bonded SiC refractories have become the state of art for aluminum reduction cell sidewall and endwall applications. More than 10 years of practical experience within about 50 different smelters and thousands of reduction cells worldwide show that an attractive technical data sheet does not automatically mean good in service performance. Examined and evaluated samples help explain that a long and consistent service is primarily related to good oxidation resistance and a very consistent quality product. We have now developed a specific set of laboratory tests that model(s) the wear mechanisms in order to better predict the service performance of the SiC sidewall and endwall. REFRAX® ARC has been developed to optimize the key properties and attributes necessary for longer service life. Refrax® ARC is now available on a worldwide basis from our key Saint Gobain source plants with production control based upon calibrated sampling and analysis methods. Lining design features, construction, and careful assembly with appropriate joining mortars, castable cements, coatings, which allow for tight joints and the avoidence of air infiltration are also discussed.

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Chemical Stability of Ceramic Sidelinings in Hall-Héroult Cells: *Eirik Hagen*[1]; Tor Grande[1]; Mari-Ann Einarsrud[1]; [1]Norwegian University of Science and Technology, Dept. of Chem., Sem Saelands vei 12, Trondheim 7491 Norway

Candidate sidelining materials based on AlN, Si_3N_4 and SiC with low oxide content have been tested in environments simulating the conditions in an electrolysis cell. In a polarised laboratory test cell previously described by Skybakmoen, the materials are simultaneously exposed to molten Al, electrolyte and the gas above the bath. The oxidation of the materials has also been investigated in cryolite saturated with CO_2 and in air. Reduction of the materials was studied during exposure to mixtures of molten Al and the electrolyte. The chemical stability and degradation mechanisms are discussed in terms of thermodynamics and the present experimental findings.

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Preparation and Evaluation of AlN/Al-Composites as Drained Cathode and Side Lining Materials in Hall-Héroult Cells: *Arve Solheim*[1]; Tor Grande[2]; Ove Paulsen[1]; Mari-Ann Einarsrud[2]; [1]SINTEF Materials Technology, Dept. of Process Metall. and Cer., Sem Saelands vei 12, Trondheim N-7465 Norway; [2]Norwegian University of Science and Technology, Dept. of Chem., Sem Saelands vei 12, Trondheim N-7491 Norway

AlN/Al-composites are lightweight electrical conducting materials with moderate mechanical strength, and thus represent candidate materials for drained cathode and side linings in aluminium electrolysis cells. AlN/Al-composites have been prepared by immersion of Si/Si3N4-preforms in molten Al. The AlN/Al-ratio in the composites has been controlled by variation of the nitrogen content in the Si/Si3N4-preforms. The AlN/Al-composites have been evaluated as candidate materials for drained cathode and side linings in aluminium electrolysis cells. A polarised laboratory test cell simulating the environments in an industrial electrolysis cell have been used for evaluation of the chemical stability of the materials towards molten Al, electrolyte and the corrosive gases evolved during electrolysis. The chemical stability and degradation mechanisms are discussed in terms of thermodynamics and the present experimental findings.

Automotive Alloys 2001: Session I

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizer: Subodh K. Das, University of Kentucky, College of Eng., Center for Aluminum Technology, Lexington, KY 40506-0043 USA

Monday PM — Room: 214
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Subodh K. Das, University of Kentucky, Cen. for Alum. Techn., Lexington, KY 40506-0043 USA

2:00 PM

Bendability of Al-Mg-Si Sheet Alloys for Automotive Closure Applications: *P. A. Friedman*[1]; S. G. Luckey[1]; [1]Ford Research Laboratory, Manuf. Sys. Dept., P.O. Box 2053, MD3135/SRL, 2101 Village Rd., Dearborn, MI 48121-2053 USA

In an effort to reduce the weight of vehicle body structures, the automotive industry has looked to alternative materials other than steel for vehicle body structures. One such material that can offer weight savings and is consistent with existing manufacturing processes is aluminum. To date, the largest application of aluminum sheet in high volume is horizontal and vertical outer body panels. The current trend for these applications has been the use of a heat-

treatable Al-Mg-Si alloy (AA6xxx) which benefits from artificial aging during the paint bake cycle. One important requirement of these materials is their ability to resist fracture in severe bending operations, such as hemming. The hemming process entails bending the outer panel around an inner panel to create the closure system. While in steel this process can result in a clean flat hem with good craftsmanship, aluminum often cannot be flat hemmed and a larger radius of curvature must be used to produce the hem. While this type of hem, often referred to as a rope hem, helps to alleviate some of the severe bending, it is not the preferred method because it results in a less robust process than the flat hem and can lead to surface defects. In this study several factors that affect the bendability of AA6xxx alloys are quantified. Critical elements regarding natural aging, artificial aging, and composition will be discussed. A parameter of particular interest is the inclusion of copper in these alloys. Copper has been added to some of these alloys in an effort to increase the kinetics during the paint bake response, which in turn increases the post-painted yield stress of the panels. While this increase in yield stress can lead to further weight and cost savings through down-gauging, it is believed by some automotive manufacturers that the inclusion of copper can have a negative effect on formability and bendability. In this paper, the effect of copper on the bending performance of aluminum will be discussed.

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Effect of Fe Content on Ductility and Bendability of AA5754 and AA6111: Jaydeep Sarkar[1]; *David S. Wilkinson*[1]; David Embury[1]; David Lloyd[2]; [1]McMaster University, Mater. Sci. & Engin., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [2]Alcan International, Kingston Res. & Dev. Centre, Kingston, ON Canada

The fracture mode and ductility of aluminum sheet alloys are quite sensitive to the level of residual iron in the alloy. This results directly from the intermetallic particles which form during solidification of these alloys and subsequently develop as particle stringers during rolling. The mode of failure appears to be controlled by the interaction of shear bands and particles. We will present data on both AA5754 and AA6111 alloys in which heats containing a wide range of Fe levels have been studied.

2:50 PM

Modelling of the Recovery and Recrystallization Behavior of Aluminum Alloy AA5754 After Industrial Cold Rolling: *Johnson Go*[1]; Mary Wells[1]; Warren J. Poole[1]; Matthias Militzer[1]; David J. LLoyd[2]; [1]The University of British Columbia, Dept. of Mets. and Mats. Eng., 309-6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada; [2]Alcan International Limited, Kingston Res. and Dev. Centre, Kingston, Ontario K7L 5L9 Canada

Aluminum is becoming the material of choice in the automotive industry because it will provide the potential for substantial fuel savings. One of the aluminum alloys slated to be used for structural applications for the automotive industry is AA5754, due to its excellent formability and high strength. In an effort to understand the microstructural changes that occur during industrial continuous annealing, the recovery and recrystallization behavior of industrial cold-rolled AA5754 was studied. Specifically, a series of annealing tests were conducted in salt and oil baths at temperatures ranging from 175 to 400°C and for various lengths of times. Recovery and recrystallization kinetics were followed by measuring the change in the mechanical properties of the material as a function of time and temperature. The results were confirmed using optical metallography techniques and the data has been implemented into a preliminary mathematical model which can predict the mechanical properties and microstructure in the sheet.

3:15 PM

Study of the Effect of Pre-Aging Heat Treatments on the Precipitation Hardening Behaviour of AA6111: *Shahrzad Esmaeili*[1]; David J. Lloyd[2]; Warren J. Poole[1]; [1]University of British Columbia, Dept. of Mets. and Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Alcan International, Kingston Rsch. and Dev. Ctr., P.O. Box 8400, Kingston, Ontario K7L 5L9 Canada

It has been shown that pre-aging heat treatments immediately after solution treatment can significantly improve the subsequent artificial aging response of Al-Mg-Si(-Cu) alloys. In this work, the effect of different pre-aging treatments on the behaviour of AA6111 during artificial aging process was investigated. It was found that the changes in the quenching process following the solution treatment and preceding the pre-aging treatment has no effect on the strength of the alloy artificially aged at 180°C. However, different quenching temperatures and cooling rates can lead to differences in the fracture behaviour of the as-pre-aged and artificially aged materials. The effect of the other main pre-aging parameters, namely temperature and time, was also studied in a broad range. Electrical resistivity measurements gave useful information on the microstructural state during the aging processes. The information could be utilized for the prediction of the artificial aging response of the pre-aged alloy.

3:40 PM Break

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Effect of Superimposed Pressure on Fracture Behaviour of Automotive Al Alloys: *Jeffrey Gimple*[1]; David S. Wilkinson[1]; David Embury[1]; John J. Lewandowski[2]; [1]McMaster University, Mat. Sci. & Eng., 1280 Main St. W., Hamilton, ON L8S 4L7 Canada; [2]Case Western Reserve University, Mat. Sci. & Eng., Cleveland, OH USA

The fracture behaviour in both AA5754 and AA6111 alloys depends on the level of Fe-based constituent particles in the alloy. We have examined this on a range of heats which differ only in the Fe content. In this paper we report on the tensile behaviour of these material tested under a superimposed pressure. As the pressure is increased the fracture mode changes in both alloy systems. This is accompanied by an increase in ductility.

4:15 PM

Initial Assessment of the Thixomolding™ Process on the Microstructure and Properties of a Magnesium Alloy: Al Ridilla[2]; Jim Williams[1]; *Sudhakar Mahajanam*[1]; [1]The Ohio State University, Dept. of Mats. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Armetal, Pittsburgh, PA USA

The microstructure and properties of the magnesium alloy AZ91D cast with the Thixomolding™ process have been studied. Light metallography, transmission electron microscopy and x-ray diffraction have been used to characterize the constitution and microstructure. This has been compared to material taken from the (non-Thixomolded™) sprue and from conventionally cast plates. Mechanical properties also will be discussed and some suggestions will be made regarding the effect of this process on properties. Honda of America has sponsored this project.

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A Review of Forming Induced Surface Defects in Aluminum Alloy Sheets for Automotive Applications: *Henry R. Piehler*[1]; Yoon Suk Choi[1]; [1]Carnegie Mellon University, Matls. Sci. and Eng. Dept., Pittsburgh, PA 15213-3890 USA

Forming induced surface defects in aluminum alloy sheets have created significant problems with automotive outer panels, which sometimes need to be hand finished to obtain an acceptable surface quality. A variety of terms have been used to describe different types of forming induced surface defects, including roping, ridging, and diamonding. The first two surface defects are lineal in configuration while the latter is areal, though generally elongated in the rolling direction. A central issue in understanding the development (and potential elimination) of these surface defects is whether they are controlled only by surface or near surface grains or are influenced or perhaps even controlled by the interior microstructure. Another issue involved in the cases where the interior microstructure plays a role in surface defect formation is whether the resulting defects are symmetric (ribbing, where high spots on one surface correspond to high spots on the other and low spots to low) or antisymmetric (corrugating, where high and low spots correspond on the two surfaces). Various crystallographically based models based on these various behaviors are reviewed and compared to experimental observations. These models include classical Taylor-type plasticity modeling as well finite element modeling which incorporates crystal plasticity, typically using data obtained using orientation imaging microspcopy. Solutions to the diamonding and other surface defect problems described in the patent literature are reviewed as well.

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Separation of Wrought Fraction of Aluminum Recovered from Automobile Shredder Scrap: *Adam J. Gesing*[1]; Christopher Stewart[1]; George Hopson[1]; Tim Good[1]; Larry Berry[1]; Richard Wolanski[1]; [1]Huron Valley Steel, Rsch. and Dev. Div., 41000 Huron River Dr., Belleville, MI 48111 USA

After more than decade of gestation use of sheet and extruded (wrought) aluminum is finally becoming wide-spread in automobiles. While it is still a decade before the wrought scrap from these new autos will show up in the recycle streams in a large quantity, efficient and cost effective technologies are being developed to recycle these alloys. The composition of cast aluminum alloys is incompatible with that of their wrought cousins. Methods of separating these fractions are being developed to permit the increase in the recycled content of the wrought alloys. The purer wrought scrap can also be used to batch newer grades of cast alloys that now utilize significant quantities of prime smelter metal for dilution. As a part of the Automotive Aluminum Alliance program designed to promote the development of the recycling system for the aluminum scrap from end-of-life vehicles we report on a new wrought aluminum scrap product recovered commercially from aluminum mix separated from non-magnetic shredder scrap. We give the size distribution, hand sort composition plus spectroscopic analysis and melt recovery of 20,000 lb batches of the wrought material melted in an industrial reverberatory side-well melter.

Carbon Technology: Plant Process Studies and Improvments

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

Monday PM — Room: 215-216
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Barry A. Sadler, Comalco Aluminium, Ltd., Brisbane, Queensland 4001 Australia

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Alcan VS Söderberg Anode Quality System: *Nathalie Bouchard*[1]; *Steward Young*[2]; [1]Alcan International, Ltd., 1955 Blvd. Mellon, C. P. 1250, Jonquière, Québec G7S 4K8 Canada; [2]Alcan Primary Metal, Kitimat Smelter, P.O. Box 1800, Kitimat, BC V8C 2H2 Canada

The paper describes the approach used by Alcan at Kitimat, its VS Söderberg smelter, to determine its anode quality. A coring technique was developed to core an anode sample from a VS anode. The characterisation of the properties of the anode cores is then linked to anode performance in the VS paste manufacturing process and the VS electrolytic cells. The approach has allowed Alcan to identify some key operating parameters and improve the overall anode performance of its Kitimat smelter.

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Interaction Study of the Paste Plant Production Parameters on VS Söderberg Anode Paste Fluidity and Baked Anode Performance: *Amir A. Mirtchi*[1]; Marcel Collard[1]; Gaby Savard[1]; Steward Young[2]; Dave Shannon[2]; Nigel Steward[2]; [1]Alcan International Limited, Reduc. Techn. Svc., Arvida Res. and Dev. Center, P.O. Box 1250, Jonquiere, Quebec G7S 4K8 Canada; [2]Alcan Primary Metal Group, B. C. Operations, P.O. Box 1800, Kitimat, British Columbia V8C 2H2 Canada

Anode performance during cell operation depends on its intrinsic quality and the cell operation parameters. The intrinsic quality of an anode is strongly influenced by the characteristics of the raw material (coke and pitch) and by the anode fabrication process. A factorial experimental design has been used to establish the interaction between the paste plant production parameters and the effect of these parameters on the Söderberg paste and anode properties. This laboratory study determines the effect of the variability of certain operating parameters as binder level, mixing temperature, variation of coke size distribution and the effect of coke super-fine on the paste fluidity and the performance of the anodes.

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15 ton/hr Computer Controlled Ball Mill: *Mike Shouse*[1]; Mike Benton[1]; [1]NSA, Process Eng., P.O. Box 500, 1627 State Rt. 271 N., Hawesville, KY 42348 USA

In July 1999, as part of a 5th Potline expansion at NSA, a new 15 ton/hr ballmill was installed. The mill has exceeded its capacity grinding petroleum coke to 50% -200 mesh and is currently averaging 16 tons/hr (-200 mesh). All variables are computer controlled with an accuracy of plus or minus 0.2%. The old 10 ton/hr ballmill operates with a 2% accuracy and its standard deviation is almost double the new ballmill. The precision of particle size distribution has improved anode quality and simplified pitch additions tremendously.

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Indirect Measurement of Anode Bake Level in Baking Furnaces by Use of Microscopical Reflectance Techniques: *J. Anthony Ross*[1]; R. J. Roush[1]; [1]Century Aluminum of WV, P.O. Box 98, Ravenswood, WV 26164 USA

Anode bake level measurements associated with baking furnace operation, which include temperature profiles, are a critical aspect of the process due to their relationship to anode quality and the efficiency of the furnace firing systems. Methods exist and are routinely used, that indirectly correlate to the temperature andoes experience during the baking process. The paper presents an alternative method based on optical microscopy that utilizes the reflectance of light from coal tar pitch coke microstructures, which are an integral part of the anode binder matrix. Data from a typical baking furnace temperature profile are presented with correlations using various indirect measurement methods, as well as the proposed reflectance technique.

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Improvement of the Anode Baking Process by Estimation of the Anode Temperature: *Wolfgang Leisenberg*[1]; [1]University of Applied Sciences, Rosenstrasse 19, Bad Nauheim D-61231 Germany

Although anode temperature is the most important parameter for the baking process, it is not used for process control. The response time is too long for direct control of the burners and flue gas volume; the life time of anode thermocouples is short and their routine replacement would be expensive and, anode thermocouples measure only one point of the pit which is not sufficient to ensure good baking uniformity. A method is presented based on an adaptive dynamic model, which allows temperatures to be calculated at any point in the pit using only a few parameters. After a single basic identification, parameters are adapted during a test period in one or two preheat sections. At any point in time, final temperatures can be predicted and the firing profile modified accordingly. This ensures a uniform temperature distribution in the pit and a standard anode quality at the lowest energy and refractory cost.

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Development of a Mathematical Model to Treat the Albras Bake Furnaces Fumes Based on a Fluidized Bed: *Paulo Douglas Santos Vasconcelos*[1]; André Luis Amarante Mesquita[2]; [1]Albras Alumínio Brasileiro S/A, Carbon Plant, Rod. PA 483 Km 21 Vl de Murucupi, Barcarena, Pará 68447000 Brazil; [2]Federal University of Pará, Mech. Eng. Dept., Belém, Pará Brazil

Albras operates four open ring-type furnaces: Plant I has furnaces A and B and a future furnace E. Plant II has furnaces C and D and is fitted with a Procedair fume treatment plant (FTP) designed to treat 215,000 Nm^3/h of gas. Plant I does not have a fume treatment system. With the expansion of the plant, new environmental legislation and the high cost of an FTP, an internal solution was sought to treat the bake furnace fumes. This paper shows how the Carbon Plant Engineering Group in partnership with the local University developed a prototype to study and survey the phenomenon of the fluorides and tar treatment in an alumina fluidized bed.

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Rodding Room Upgrade At Dubal: A. J.M. Kalban[1]; *Abdul Binbrek*[1]; G. S. Sachan[1]; [1]Dubai Aluminum Company, Ltd., Reduction Mats., P.O. Box 3627, Dubai United Arab Emirates

During 1999 Dubal retrofitted its Rodding Room to enable it to cater for the increased anode requirements for it's six potlines producing over 536,000 tonnes of aluminum per year. The retrofit included an upgrade of all rodding equipment, the incorporation of a SCADA System and an on-line rod straightening and stub repair facility. For a comparable two shift operation a 40% increase in production coupled with a 17% lower operating cost has been achieved. In addition, a major improvement in rodded anode quality and the occupational health and work environment has also been achieved. Further, improved butt cleaning enable Dubal to recycle carbon dust from butts crushing and screening system.

Cast Shop Technology: Direct Chill Casting

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

Monday PM — Room: 208-210
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Laurens Katgerman, TU Delft; Ho Yu, Alcoa

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Advances for DC Ingot Casting: Part 1-Introduction and Metal Distribution: *Gary Patrick Grealy*[1]; J. Lee Davis[2]; Einar Kristian Jensen[3]; [1]CORUS, Res., Dev. & Techn., Ijmuiden Technology Centre 1970 CA Ijmuiden, The Netherlands; [2]Wagstaff, Res. & Dev., 3910 North Flora Rd., Spokane, WA 99216 USA; [3]Elkem Aluminium, Research, Alum. Techn., P.O. Box 8040, Vaagsbygd, Kristiansand N-4602 Norway

The continual drive for improvements and advancements in the field of DC sheet ingot casting, and the introduction of casting technologies that provide the user with greater degrees of control over a number of operational parameters, emphasises the need for more in-depth understanding of many related casting fundamentals. This paper will present a study of a number of these fundamentals in two parts. Part 1 will include the influence of metal distribution, while Part 2 will present heat transfer and the resulting solidification behaviour. The results of physical and mathematical modeling and experimental casting trials will be discussed.

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Advances for DC Ingot Casting: Part 2-Heat Transfer and Solidification Behaviour: *Gary Patrick Grealy*[1]; *J. Lee Davis*[2]; *Einar Kristian Jensen*[3]; [1]Corus, Res., Dev., and Techn., Ijmuiden Technology Center, 1970 CA Ijmuiden, The Netherlands; [2]Wagstaff, Res. & Dev., 3910 N. Flora Rd., Spokane, WA 99216 USA; [3]Elkem Aluminium, Research, Aluminium Technology, P.O. Box 8040 Vaagsbygd, Kristiansand N-4602 Norway

The continual drive for improvements and advancements in the field of DC sheet ingot casting, and the introduction of casting technologies that provide the user with greater degrees of control over a number of operational parameters, emphasises the need for more in-depth understanding of many related casting fundamentals. This paper will present a study of a number of these fundamentals in two parts. Part 1 will include the influence of metal distribution, while Part 2 will present heat transfer and the resulting solidification behaviour. The results of physical and mathematical modeling and experimental casting trials will be discussed.

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The Influence of Metal-Entry on Macrosegregation in the DC-casting of Aluminium-Copper Alloys: *Bart Venneker*[1]; Laurens Katgerman[1]; [1]Netherlands Institute for Metals Research/Delft University of Technology, Lab. of Mats. Sci., Rotterdamseweg 137, Delft 2628 AL, The Netherlands

Modeling results are presented for the metal flow and solidification behavior of a binary Al-Cu alloy in the D. C. casting of cylindrical ingots. The results are obtained with the commercial CFD package CFX-4, with additional user subroutines for the simulation of solidification and segregation. In the model, both shrinkage and natural convection due to thermal and solutal buoyancy are taken into account. The solid phase is assumed to flow with the casting velocity. Emphasis in this paper lies on differences in the macrosegregation with different metal-entry/mould configurations, but with the same experimental conditions. The different sump flow field and mushy zone characteristics are analyzed to explain the differences in the concentration profiles.

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Mechanism of Oxides Generation and Mixing into Aluminum Ingot in Cast Start Phase: *Masanori Tsunekawa*[1]; Shinichi Tani[1]; Hajime Okazaki[2]; Norifumi Hayashi[1]; [1]Sumitomo Light Metal Industries, Ltd., Res. & Dev. Center, 1-12, 3-Chome, Chitose, Minato-Ku, Nagoya, Aichi 455-8670 Japan; [2]Sumitomo Light Metal, Ltd., Slab Casting Techn. Dept., Nagoya Works, Japan

On aluminum semi-continuous casting, many oxides are generated by the turbulence metal flow when feeding the metal to the mold in cast start phase. These oxides were entrapped on metal surface or bottom of the glass cloth filter,and mixing into the aluminum ingot through the casting period. Finally these oxides cause the sheet defects. Therefore in this works we clarified the mechanism of oxides generation and mixing into the ingot by investigating qualities of rolled sheets and ingots which were cast under such conditions as initial metal feeding speeds, distance of between the glass cloth filter and the bottom block,and inert gas atmosphere in the mold.

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Mould Surface Roughness Effects on the Microstructure and the Hot Tearing Strength for an Al-4. 5wt% Cu Alloy: *Martin Fortier*[1]; Denis J. Lahaie[1]; Michel Bouchard[1]; Joseph Langlais[2]; [1]Alcan-UQAC CSMA, 555 Universite Boul., Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International. Ltd., Arvida Res. and Dev. Centre, 1955 Mellon Boul., P.O. Box 1250, Jonquiere, Quebec G7S 4K8 Canada

The effects of D. C. casting mould roughness on the microstructure and the strength to hot tearing was investigated for an Al-4. 5wt%Cu alloy. Mould surface roughness has been found to influence the resulting grain size near the surface by the preferential nucleation and growth on surface asperities. This grain size variation was found to influence the mechanical response of the semi-solid material which may be associated to the hot tearing susceptibility. The effect of the mould roughness on the resulting microstructure was also reproduced using a simple cellular automaton model. A "surface efficiency coefficient" is proposed to characterise the cyclic effect of the mould surface roughness on the resulting grain size. This coefficient is defined as to represent the combined effects of the localised casting shrinkage and the liquid/mould contact distribution.

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Automatic Control of Vertical Direct Chill Casting: *Peter Ridley*[1]; *Phillip Baker*[2]; [1]Queensland University of Technology, Sch. of Mech., Medi. and Manuf. Eng., GPO Box 2434, Brisbane, Queensland 4001 Australia; [2]Hatch Australia Pty, Ltd., Light Metals Business Unit, P.O. Box 425, Spring Hill, 152 Wharf St., Brisbane, Queensland 4000 Australia

This paper provides a theoretical analysis of automatic control of pouring during a vertical direct chill (VDC) hot top casting process. A mathematical model of the operation is created and used to predict the steady state and transient response of process variables (eg furnace tilt, actuator loads,liquid levels),under closed loop control. Classical control (root-locus) techniques are used to predict suitable PID loop tunings which ensure stable plant operation throughout the pour. System sensitivity to plant disturbances is also examined using frequency response analysis.

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A Novel Approach to Direct Chill (DC) Solidification of Irregular Shaped Ingots for the Forged Production of Automobile Suspension Components: *Robert B. Wagstaff*[1]; *Richard S. Bruski*[2]; *Daniel J. Groszkiewicz*[3]; [1]Wagstaff, Inc, 3910 North Flora Rd., Spokane, WA 98226 USA; [2]Alcan, 37676 Enterprise Court, Farmington Hills, MI 48331-3440 USA; [3]Alcoa, P.O. Box 790, Barberton, OH 44203 USA

The Direct Chill (DC) casting of symmetrical and non symmetrical shaped ingot for forging stock presents some unique challenges in the industry today. The authors of this paper will cover the production of cast non symmetrical shaped ingot, the transformation of said ingots to forging preforms, the forging process as it relates to shaped ingot and discuss the material properties available in the final forged product. While other shapes are currently being worked on, this paper will use the Ford DEW 98 Front Lower Control Arm as a case study to quantify and present the process.

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Magnesium Direct Chill Casting: A Comparison with Aluminium: *John F. Grandfield*[1]; Philip W. Baker[2]; Paul T. McGlade[3]; [1]CSIRO, Manuf. Sci. & Techn., cnr Albert & Raglan Sts., Preston, Vic 3072 Australia; [2]Hatch Australia, Ltd., 152 Wharf St., Brisbane, Queensland 4000 Australia; [3]Fluor Australia, Level 6, Toowong Tower, 9 Sherwood Rd., Toowong, Queensland 4066 Australia

Magnesium is going through something of a renaissance due to the growth in automotive die cast components. However, wrought alloy direct chill (DC) cast products are also receiving some attention and DC casting of magnesium is being improved. It is instructive to compare the DC casting of magnesium and aluminium. We briefly examine the current state of magnesium DC castings' production, product mix, industry characteristics and costs. The basic properties of the two metals and the status of DC technology are compared. The fact that the tonnage of aluminium DC castings is about one hundred times that of magnesium accounts largely for the relatively small research and development effort in magnesium DC casting compared to aluminium. Improvement of magnesium DC casting is however, taking place, including development of hot top moulds, safety systems and horizontal direct chill casting. The good news for magnesium casters is that there is much know how being generated and published on aluminium DC casting which can be applied to magnesium. Conversely some of the technology generated for magnesium may be applicable to casting of high magnesium aluminium alloys. Mutual awareness between the two industries will be beneficial to both.

Chemistry and Electrochemistry of Corrosion and Stress Corrosion: A Symposium Honoring the Contributions of R.W. Staehle: Mechanisms and Modeling-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

Monday PM Room: 222
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Russell H. Jones, Pacific Northwest National Laboratory, Mats. Scis. Dept., Richland, WA 99352 USA; Jack H. Westbrook, Brookline Technologies, Ballston Spa, NY 12020 USA

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A Discussion of Mechanisms and Modeling of Secondary Side Corrosion Cracking in PWR Steam Generators: *P. M. Scott*[1]; [1]Framatome, Tour Framatome, 92084 Paris La Defense, France

Intergranular stress corrosion cracking (IGSCC) and intergranular attack (IGA) of mill annealed alloy 600 tubes is a widespread and growing problem for operators of Pressurized Water Reactors (PWR). In the case of recirculating steam generators (as distinct from once through steam generators), these forms of tube attack are usually associated with the hideout of impurities from the secondary feed water in superheated crevices where water flow is restricted by sludge deposits. Typical sites for IGA/IGSCC are tube support plate crevices and under sludge piles on the tube sheet. The temperature difference between the primary and secondary sides of a steam generator tube vary typically from 45°C at the base of the hot leg tubes to 22°C near the upper U bends. At these degrees of superheat, impurity concentrations of between 25 and 15M are necessary in order for such solutions to remain in the liquid state at the secondary side pressure. The evaluation and modeling of tube damage has traditionally been based on th e assumed formation of these solutions with extreme values of pH typically less than 5 or greater than 10 at temperature. It will be argued in the present paper that the formation of such concentrated solutions in superheated crevices with modern steam generator chemistry practices is unlikely. An equally valid hypothesis is that steam pockets can form in such occluded positions, albeit contaminated by impurities concentrated by the classical hideout mechanism. Even if very concentrated liquids could form, it will be argued that it is highly unlikely that pH alone is a satisfactory descriptive parameter sufficient to characterize the occluded environment.

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Insights into Environmental Degradation Mechanisms from High-Resolution Characterization of Crack Tips: *Stephen M. Bruemmer*[1]; Larry E. Thomas[1]; [1]Pacific Northwest National Laboratory, Mats. Interf. Grp., P.O. Box 999, Mail Stop P8-16, Richland, WA 99352 USA

Intergranular stress corrosion cracks and crack tips produced in high-temperature water environments have been characterized in both Ni-base (alloy 600) and Fe-base (304 and 316SS) stainless alloys. Cracks and corroded regions are examined in cross-section using high-resolution analytical transmission electron microscopy to determine the structure and composition of crack-wall and crack-tip oxides as well as the grain boundary metallurgy immediately ahead of crack tips. Fundamental differences are identified in crack-tip characteristics between the Ni-base and Fe-base stainless alloys and as a function of the water environment and the grain boundary microstructure. These unique nanometer-scale measurements indicate corrosion and deformation processes occurring during crack advance and provide insights into mechanisms controlling environmental degradation.

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Role of Grain Boundary Deformation in IGSCC of Austenitic Alloys in High Temperature Water: *Gary S. Was*[1]; [1]University of Michigan, Nuclear Eng. and Radiol. Scis., and Mats. Sci. and Eng., 2355 Bonisteel Blvd., 1921 Cooley Bldg., Ann Arbor, MI 48109-2104 USA

Grain boundary properties are known to affect the intergranular stress corrosion cracking (IGSCC) behavior of alloys in many environments. However, only recently has there been enough evidence to suggest that deformation in and near the boundary may play a key role in IGSCC in many systems. In austenitic alloys at high temperature, the deformation behavior of the grain boundary may control grain boundary cracking and intergranular crack growth in several ways. Grain boundary structure influences the absorption and production of dislocations that influence deformation in the boundary and in the neighboring grains. The inability of a grain boundary to absorb and transmit dislocations may result in increased stress on the boundary. Grain boundary carbides are known to lower susceptibility to IGSCC, either as obstacles for crack propagation or as sources for dislocation production. Solute additions can affect deformation in and near the grain boundary. Carbon present as a solute reduces the diffusivity of solvent atoms in the grain boundary by several orders of magnitude, virtually eliminating grain boundary sliding. Carbon also affects the slip mode and the dislocation structure in the matrix. This talk will focus on the role of deforma-

tion processes in and near the grain boundary and on how they may affect IGSCC in austenitic alloys at high temperature.

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Mechanism of the Cracking Behavior of Alloy 600 in Caustic Environments with Inhibiting Species: *Jesse B. Lumsden*[1]; Al McIlree[2]; [1]Rockwell Science Center, 1049 Camino Dos Rios, Thousand Oaks, CA 91360 USA; [2]EPRI, 3412 Hillview Ave., Palo Alto, CA 94304 USA

Mill Annealed Alloy 600 is susceptible to two types of cracking processes when it is exposed to high temperature caustic solutions, intergranular attack (IGA) and intergranular stress corrosion (IGSCC). The IGA crack growth rate is slow and is characterized by uniform penetration of all or most grain boundaries; whereas, the IGSCC growth rate is much faster than that of IGA and occurs as a single penetration or widely separated penetrations down grain boundaries. These two cracking modes have well-defined electrochemical potential zones within which they occur. An evaluation of results from surface analysis and from electrochemical measurements has identified the chemical processes occurring on the surface in the two potential regimes where cracking occurs. This has provided the key to understanding the cracking mechanisms and has suggested species for inhibiting caustic cracking in Alloy 600.

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Prediction of Stress Corrosion Crack Growth in Service, Based on Laboratory Data: *Markus O. Speidel*[1]; [1]Swiss Federal Institute of Technology, Inst. of Metall., Zurich CH-8092 Switzerland

Research in stress corrosion cracking is most often justified by the desire to reach one of the two following goals: either a scientific, academic understanding of the underlying physical and chemical mechanisms or the prediction and prevention of stress corrosion cracking of critical components in service. This paper shows to what extent the latter goal can be reached by fracture mechanics stress corrosion crack growth rate tests in the laboratory. Quantitative comparisons between laboratory test results and in-service stress corrosion crack growth rates are shown for critical components of nuclear power plants. These involve nickelbase alloys of the types 600 and 690, stabilized stainless steels of the types 321 and 347 as well as non-stabilized stainless steels 304 and 316. The less satisfactory situation of ferritic steels is also characterized, where both, laboratory results and in-service stress corrosion crack growth rates are known with much less precision.

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The Role of Internal/External Environment Coupling in Stress Corrosion Cracking: *Digby D. Macdonald*[1]; [1]Pennsylvania State University, Center for Electro. Sci. and Techn., 517 Deike Bldg., University Park, PA 16802 USA

Strong coupling, as required by the differential aeration hypothesis, has been observed in stress corrosion cracking in a variety of systems, including IGSCC in sensitized Type 304 SS in simulated BWR coolant environments at 288ºC and in the caustic cracking of AISI 4340 steel at ambient temperature. Examination of the coupling current, which is easily measured using a sensitive zero resistance ammeter, shows that it contains "structured" noise superimposed upon a mean. The noise in the current is found to yield a wealth of information on the fracture events that occur at the crack tip, including their frequency, temporal relationship with other events, and size. This information has provided a clear view of the fracture mechanisms, which in both cases involves brittle micro fracture events of a few micrometers in size. These data are more consistent with hydrogen-induced fracture than they are with a slip/dissolution mechanism, even though the external environment is oxidizing in nature.

Computational Thermodynamics and Materials Design: Thermodynamic Modeling 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, 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

Monday PM — Room: 201
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Gerhard Inden, Max-Planck-Institut fur Eisenforschung GmbH, Duseldorf D-40237 Germany

2:00 PM

Some Improbable Phase Diagrams Revisited: Ab Initio Predictions: *Patrice E. A. Turchi*[1]; Vaclav Drchal[2]; Josef Kudrnowsky[2]; [1]Lawrence Livermore National Laboratory, C. & M. S. Dept. (L-353), P.O. Box 808, Livermore, CA 94551 USA; [2]Institute of Physics, Acad. of Scis. of the Czech Republic, Na Slovance 2, Praha 8, CZ 182-21 Czech Republic

A large number of phase diagrams only display below the solidus-liquidus lines complete miscibility among the alloy species supposedly down to zero temperature. Examples of this type of diagrams include those of most alloys made of two bcc-based transition metals. Using a first-principles electronic structure approach to phase stability and order, possible ordering trends that may occur at low temperatures for these alloys are revealed. The methodology is based on the Generalized Perturbation Method applied to the fully relativistic Tight-Binding Linear Muffin-Tin Orbital description of the electronic structure of the chemically random configuration of the alloy, within the Atomic Sphere Approximation and the Local Density Approximation of Density Functional Theory. The underlying lattice and the atomic number of each alloy species constitute the only required input information. Finite temperature effects are treated within the generalized mean-field Cluster Variation Method. Ordering trends are then ra tionalized as functions of simple electronic parameters that are known to govern alloy stability. Work performed under the auspices of the U.S. Dept. of Energy by the University of California, Lawrence Livermore National Laboratory under Contract W-7405-ENG-48.

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Accurate First-Principles Calculations of Phase Boundaries in Al-Based Alloys: *Vidvuds Ozolins*[1]; Mark D. Asta[2]; Christopher M. Wolverton[3]; [1]Sandia National Laboratories, Thin Film and Interf. Sci. Dept., P.O. Box 969 MS 9161, Livermore, CA 94551-0969 USA; [2]Northwestern University, Dept. of Mats. Sci. and Eng., Evanston, IL 60208-3108 USA; [3]Ford Research Laboratory, MD3028/SRL, Dearborn, MI 48121-2053 USA

Although first-principles calculations usually yield phase diagrams that are qualitatively correct, their practical applications are somewhat limited since the remaining quantitative errors in transition temperatures are still unacceptably large. We study phase stabilities and solvus boundaries in Al-Ti, Al-Cu, and Al-Sc alloys and show that the accuracy of first-principles calculated phase diagrams can be drastically improved by including contributions to entropy from ionic vibrations. For instance, vibrational entropy increases

the solid solubility of Sc in Al matrix by a factor of thirty, leading to an excellent agreement between the calculated and experimentally measured solvus boundaries. Similar large effects are found in other Al-based alloys. These results demonstrate that state of the art first-principles calculations have evolved to the point where they are capable of producing quantitatively accurate alloy phase diagrams.

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Application of Statistical Moment Method to Thermodynamic Quantities of Metals and Alloys: *K. Masuda-Jindo*[1]; Vu Van Hung[2]; Pham Dinh Tam[2]; [1]Tokyo Institute of Technology, Dept. of Mats. Sci. and Eng., Nagatsuta 425, Midori-ku, Yokohama 226-8503 Japan; [2]Hanoi National Pedagogic University, km8 Hanoi-Sontay Hwy., Hanoi, Vietnam

The thermodynamic quantities of metals and alloys are studied using the moment method in the statistical dynamics [1,2]ÅC which allows us to take into account the effects of thermal lattice vibrations and size-mismatch of constituent atomsÅC going beyond the harmonic approximation. Within the fourth order moment approximation, the free energy, specific heats C subscript V and C subscript p, the mean square relative displacements and equilibrium lattice spacing of the binary a11oys are given explicitly in terms of the effective pair potentials and the second and fourth order vibrational constants. We calculate the long range order (LRO) parameter and order-disorder transition temperature of the ordered alloys using the electronic many body potentials. The numerical calculations of LRO parameters for Beta-CuZn and Cu subscript 3Au alloys are in good agreement with the experimental results. We also discuss the vibrational properties of ordered alloys like Fe subscript 3 Al, in conjunction with their large differen ces in vibrational entropy and mean square relative displacements between ordered and disordered states.

3:20 PM

Chemical Potentials in Tin-Lead Solid Solutions: *Manuel Alvarado*[1]; John T. Farraro[1]; Roy Arrowood[1]; [1]University of Texas at El Paso, Metall. and Mats. Eng., M201 Eng. Scis., El Paso, TX 79968-0520 USA

As part of a computational investigation of diffusion phenomena in tin-lead alloys, we are developing a model for the chemical potentials of tin-lead-vacancy solid solutions. The model is based on the quasichemical theory. To estimate input parameters such as bond energies and vacancy formation energies, we are using computational methods (empirical potentials, density functional theory) as implemented in the Cerius$^{2(TM)}$ molecular/crystal simulation software. In addition, we use published physical and thermochemical data as inputs, where available. To validate the model, we compare its predictions with published phase diagram and diffusion data.

3:40 PM Break

3:50 PM

Incorporating First-Principles Energetics in Computational Thermodynamics: *Chris Wolverton*[1]; Ravi Vijayaraghavan[1]; Xin-Yan Yan[1]; Vidvuds Ozolins[2]; [1]Ford Motor Company, MD 3028/ SRL, P.O. Box 2053, Dearborn, MI 48176 USA; [2]Sandia National Laboratories, Livermore, CA USA

Computational thermodynamic approaches, such as those pioneered by Dr. Kaufmann, have become a valuable tool in the calculation of complex, multicomponent phase equilibria often found in industrial alloys. These methods rely on databases of free energies, which are often obtained from an optimization process involving experimental thermodynamic data and phase diagrams. However, many phases of practical interest, such a precipitate phases, are metastable, and the metastable phase boundaries are often not well characterized. Consequently, these important phases are often absent from computational thermodynamics databases. We demonstrate that first-principles, quantum-mechanical calculations provide a means to obtain thermodynamic functions of phases absent from present databases. We illustrate this approach with the famous metastable Cu-containing precipitate phases (GP zones and Al_2Cu-Θ') often found in age-hardened aluminum alloys. We discuss issues of the accuracy of first-principles energetics and the incorporation of absolute vs. relative energies into thermodynamics databases.

4:20 PM

Predicting Thermodynamic Properties of Materials: *Marius Stan*[1]; Michael I. Baskes[1]; Steven M. Valone[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Techn. Div., P.O. Box 1663, MS-G755, Los Alamos, NM 87545 USA

Microscopic and macroscopic scale models are combined to predict thermodynamic properties of materials. Many-body interactions, as well as vacancies, defects and non-stoichiometry are included in the modeling process and the structural stability of hypothetical phases is evaluated. The process of relating microscopic modeling results to the macroscopic phase equilibrium calculations is detailed to emphasize the self-consistency of the approach and to identify the potential sources of errors. The sequence: data acquisition, modeling, prediction, experimental validation, is illustrated for several recent results in the Mo-Si and Ce-Ga-O systems.

4:50 PM

CVM Calculations of BCC Fe-Mo-Al Phase Diagram: *Silvana Zacarelli*[1]; Jorge Alberto Soares Tenório[1]; Cláudio Geraldo Schön[1]; [1]Escola Politecnica Da Universidade De Sao Paulo, Dept. Metall. Matls., Av. Prof. Mello Moraes, 2463, Cidade Universitaria, Sao Paulo, SP 05508-900 Brazil

Iron aluminides are characterised high strength/density ratios and good corrosion/oxidation resistence. This combination of properties makes these alloys good candidates for structural applications involving moderate to high temperatures. Binary iron aluminides, however, are also characterised by low room termperature ductility, specially in deformation under air. Several strategies have been proposed to improve the ductility of these materials, among them, alloying with a ternary component. The first step to such investigation involves the knowledge of the phase diagram, and for the case of the iron aluminides, specially of the ordering phase equilibria. In this context phase diagram calculations play an essential role, helping to set experiments and in the interpretation of experimental data. The present contribution focuses on the BCC Fe-Al-Mo system, with emphasis in the iron-rich corner, for which only limited experimntal information is available in the literature. Phase diagram calculations for the BCC phase equilibria have been performed within the Cluster Variation Method (CVM) formalism, using the irregular tetrahedron approximation. First and second nearest pairs interaction as well as tetrahedron interactions have been taken into account. In a first step the critical surfaces for ordering of the B2/A2 and D03/B2 second-order phase transitions have been calculated using preliminary thermodynamic data taken from the literature. With reference to these calculated surfaces, compositions of samples for differential thermal analysis (DTA) and differential scanning calorimetry (DSC) experiments were chosen and the experiments have been carried out. With the new experimntal information, the interaction parameters for the CVM calculation have been reassessed and isothermal sections of the phase diagram in the temperature range between T= 623K and T=1200K have been calculated. Good agreement between calculations and experimental data has been obtained.

5:10 PM

Molecular Dynamics Simulation and In Situ TEM Study: *Wuyang Chu*[1]; Shen Li[1]; Kewei Gao[1]; Lijie Qiao[1]; [1]University of Science and Technology Beijing, Dept. of Matls. Phys., 30 Xueyuan Lu, Beijing 100083 China

The molecular dynamics method is used to simulate microcrack healing during heating or under compressive stress. A center microcrack in Al and Cu crystals can be sealed under a critical compressive stress or by heating it over a critical temperature. During microcrack healing, dislocation and vacancy are generated and moved, and sometimes twin appears. The critical temperature necessary for microcrack healing depends upon the orientation of the crack plane. For example, the critical temperature of the crack along the (111) plane is the lowest. When there are pre-existing dislocations around the microcrack, the critical temperature necessary for microcrack healing well decrease. In situ TEM study shows that microcrack

with size of 0.2 micron to 2 micron nearby a main crack for alpha-Fe single crystal can be gradually sealed by heating in TEM over 973K.

Cyanide: Social, Industrial, and Economic Aspects: Politics and Spills 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

Monday PM Room: 225
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Corby Anderson, Montana Tech, CAMP, Butte, MT 59701-8997 USA

2:00 PM Invited
Unresolved Problems With the Use of Cyanide in Precious Metals Mining: *Glenn C. Miller*[1]; Chris A. Pritsos[2]; [1]University of Nevada, Center for Environmental Sciences and Engineering, Mail Stop 199, Reno, NV 89557 USA; [2]University of Nevada, Dept. of Nutrition, Mail Stop 199, Reno, NV 89557 USA

Cyanide is presently the most commonly used lixiviant in gold mining. The efficiency and cost-effectiveness of cyanide has made possible the development of large open pit mines in the western United States and is a factor associated with the environmental impacts and political opposition to many of these large mines. Although cyanide is generally preferred over other lixiviants, the use of cyanide in the past two decades has revealed at least three environmental issues that are not yet well understood. These include: sublethal impact of cyanide on wildlife, release of transformation products of cyanide including metal cyanide complexes (i. e. mercury cyanide), and closure of precious metals heaps. The first two topics relate directly to cyanide use, while the third is a result of use of cyanide in the recently developed heap leach methods for extraction of low grade gold ore. Particularly in moderately arid areas with annual precipitation of 15-40 mm/year, contaminated drainage from many closed heaps con taining elevated concentrations of salts, selenium, arsenic, cobalt and antimony will be a management concern for several decades into the future.

2:25 PM Invited
The Attack on Open-Pit Mining through the Ban of Use of Cyanide, McDonald Gold Project, Montana: *Richard H. DeVoto, President*[1]; [1]Canyon Resources Corporation, 14142 Denver West Parkway, Suite 250, Golden, CO 80401 USA

The McDonald Gold project and two satellite gold deposits near Lincoln, Montana, were discovered in the early 1990's and contain at least 9. 9 million ounces of gold and 30 million ounces of silver. The McDonald gold deposit contains 7. 2 million ounces of gold reserves that can be open-pit mined and processed by cyanide heap leaching at a projected cash operating cost of $180 per ounce. The deposit is predominantly oxidized with no chance of acid production, and can be mined, operated, and reclaimed with minimal environmental disturbance other than the creation of a fresh-water lake which would support trout. The Seven-Up Pete Joint Venture, the owner of the McDonald Gold Project, commenced permitting of an open-pit, heap-leach operation in November 1994. In 1998, environmental activists proposed and Montana citizens passed an anti-cyanide, open-pit mining initiative, I-137. The campaign rhetoric focused on the hazards of cyanide in mining, but the unstated, now revealed, objective was to ban any future open-pit mining in the State, particularly the McDonald deposit. The Seven-Up Pete Joint Venture has filed two lawsuits against the State of Montana seeking to overturn I-137 or, alternatively, to obtain a takings damage award, which could be more than $500 million. The Venture has conducted exhaustive studies, including metallurgical testing of the leachability of McDonald ores with alternative lixiviants, and has determined that all other mining and known metallurgical treatment systems are vastly uneconomic in the recovery of gold and silver from the McDonald deposit. By misleading the public about the risks of cyanide usage in mining operations and passage of the anti-cyanide, open pit mining initiative, the environmental activists have been successful in prohibiting new open-pit gold/silver mines in Montana.

2:50 PM Break

3:00 PM Plenary
Cyanide: Just the Facts: *Courtney A. Young*[1]; [1]Montana Tech, Metallurgical and Materials Engineering, ELC Bldg. 215, 1300 West Park St., Butte, MT 59701 USA

This presentation discusses the facts about cyanide beginning with a definition and ending with a discussion on cyanide alternatives. Other topics include chemistry, toxicity, cyanogenics, cyanide cycle, handling, destruction, removal, and markets and uses (not just in the mining industry). Emphasis, of course, is placed on gold production by leaching and how it is influenced by ore type, mineral characteristics, and economics.

3:30 PM Invited
Panel Disussion-Cyanide, Where To From Here?: *Corby Anderson-Panel Moderator*[1]; [1]Montana Tech, CAMP & Metallurgical and Materials Engineering, 1300 W. Park St., Butte, MT 59701 USA

This panel discussion will be arranged later but will provide a forum for technical and non-technical people to get together and discuss cyanide issues and their impacts on society and industry.

Defect Properties and Mechanical Behavior of HCP Metals and Alloys: Defects, Interfaces, and Diffusion

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, Ames Laboratory, Iowa State University, Ames, IA 50011-3020 USA; Carlos N. Tome, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Monday PM Room: 211
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Vasek Vitek, University of Pennsylvania, Dept. of Mats. Sci. and Eng., Philadelphia, PA 19104-6272 USA; Alexander H. King. Purdue University, School of Mats. Eng., West Lafayette, IN 47907-1289 USA

2:00 PM Invited
Diffusion in Hexagonal Metals and Intermetallics: *Chr. Herzig*[1]; Y. Mishin[2]; [1]University of Muenster, Instit. fuer Materialphysik, Wilhelm-Klemm-Strasse 10, Muenster D-48149 Germany; [2]George Mason University, Sch. of Compu. Sci., 4400 University Dr., Fairfax, VA 22030-4444 USA

Diffusion in hexagonal structured materials is important for many applications. This paper reviews the recent work on diffusion in such materials, with emphasis on group IV B hcp metals Ti, Zr and Hf, and the intermetallic compound Ti_3Al with a hexagonal structure $D0_{19}$. Recent radiotracer diffusion measurements performed on high-purity hexagonal materials reveal that their self-diffusion is "normal" and can be understood through vacancy-related mechanisms. Some impurities, such as Fe, Ni and Co, dissolve in group IV B hcp metals interstitially and diffuse anomalously fast by intersti-

tial-related mechanisms. They accelerate diffusion of host atoms and result in "anomalous" behaviour of self-diffusion. This extrinsic effect has dominated previous self-diffusion measurements. Other impurities (e.g. Al, Ga and In) dissolve substitutionally and diffuse by the same mechanism as host atoms. Diffusion in Ti_3Al follows basically the same pattern. Grain boundary diffusion in hexagonal materials is briefly reviewed. The recent results suggest that grain boundary diffusion follows a similar behaviour with respect to "fast" and "normal" impurities as bulk diffusion.

2:30 PM

Point Defect Diffusion in Alpha Zirconium: *Yuri N. Osetsky*[1]; David J. Bacon[1]; Nieves de Diego[2]; [1]The University of Liverpool, Mats. Sci. and Eng., Brownlow Hill, Liverpool, UK; [2]Universidad Complutense, Dpto. de Fisica de Materiales, Ciudad Universitaria, Madrid, Spain

Two types of intrinsic defect, i.e. vacancy and self-interstitial atom, are formed in metals during irradiation with energetic particles. The evolution of defect population leads to significant changes in microstructure and causes a number of radiation-induced property changes. Some phenomena, e.g. radiation growth of anisotropic materials, are due to anisotropy of atomic mass transport of point defects. Detailed information on atomic-scale mechanisms is therefore necessary to understand such phenomena. In the present paper we present results of a computer simulation study of mass transport via point defects in hcp zirconium. The matrix of diffusion coefficients, activation energies and correlation factors have been obtained and different methods of treatment of diffusion have been tested. MD has shown that vacancy diffusion is almost isotropic at all studied temperatures. The mechanism of interstitial diffusion changes from one-dimensional (1-D) diffusion at low temperature (< 300K) to planar and three-dimensional (3-D) motion at higher temperatures.

2:50 PM

Point Defects and Diffusion Mechanisms in the Hexagonal $D0_{19}$-Ti_3Al: *Y. Mishin*[1]; Chr. Herzig[2]; [1]George Mason University, Sch. of Comp. Scis., 4400 University Dr., Fairfax, VA 22030-4444 USA; [2]University of Muenster, Institut. fuer Materialphysik, Wilhelm-Klemm-Strasse 10, Muenster D-48149 Germany

Many high-temperature properties of the intermetallic compound Ti_3Al depend on the atomic mobility of Ti and Al in the lattice. Although it is well understood that diffusion in Ti_3Al is dominated by vacancy exchanges with nearest-neighbor atoms, the exact mechanisms by which the vacancy jumps preserve the average degree of atomic order are not well known. This talk discusses the recent advances in the understanding of such mechanisms through atomistic computer simulations combined with concurrent experimental diffusion measurements. A statistical model of point-defect disorder in Ti_3Al is presented along with calculations based on the embedded-atom method. Possible order-preserving mechanisms of vacancy diffusion are evaluated by calculating the relevant activation energies. Predictions are made regarding the dominant diffusion mechanism depending the temperature and degree of off-stoichiometry. The simulation results are compared with the diffusion behavior of Ti_3Al observed experimentally.

3:10 PM Break

3:30 PM Invited

On the Mechanisms of Grain Boundary Processes in HCP Metals: *R. C. Pond*[1]; [1]University of Liverpool, Dept. of Mats. Sci. and Eng., Brownlow Hill, Liverpool L69 3GH UK

The kinetics of grain boundary processes, such as sliding, migration and source/sink action, are determined by the atomic mechanisms which operate. Experimental observations show that, in some circumstances, interfacial defects are the active agents, and hence process kinetics are determined by the mobility of individual defects and their interactions. In the present paper these factors are investigated using atomic-scale computer simulation of defect mechanisms induced in hcp bicrystals by applied stresses. It is shown that the mobility of interfacial defects depends on their topological character, i.e. their Burgers vector, b, and step heights, h, and also the extent to which defects are localised. In some respects interactions between interfacial defects resemble those between dislocations in single crystals, but distinct differences also arise. For instance, defects which pass in an interface must proceed up/down each other steps, and this may involve climb. This factor leads to novel defect mechani sms, and examples involving zero overall difffusive fluxes will be illustrated by computer simulation.

4:00 PM

Mobility of Interstitial Clusters in HCP Zirconium: *Nieves de Diego*[1]; *Yuri N. Osetsky*[2]; *David J. Bacon*[2]; [1]Universidad Complutense, Dpto. de Fisica de Materiales, Facultad de Ciencias Fisicas, Ciudad Universitaria, Madrid, Spain; [2]The University of Liverpool, Dept. of Mats. Sci. and Eng., Brownlow Hill, Liverpool UK

Significant attention has been given to the finding that clusters of self-interstitial atoms (SIAs) formed directly in displacement cascades in irradiated metals and play an important role in microstructure evolution. The successful application of the production bias model in explaining many features of radiation-induced microstructures such as inhomogeneous damage near grain boundaries, decoration of dislocations by interstitial dislocation loops, rafts of dislocation loops, void lattice formation, etc., has initiated extensive studies of the properties of SIA clusters. So far these studies were concentrated on cubic metals, e.g. fcc and bcc. In this work we present the first results of an atomistic study of SIA cluster mobility in an hcp crystal. It was obtained that all clusters larger than 4 SIAs exhibit thermally-activated one-dimensional glide in <11-20> directions. The cluster jump frequency, activation energy and correlation factors have been estimated, and comparisons drawn between the behaviour of SIA clusters in different structures.

4:20 PM

Grain Boundary Diffusion by Vacancy Mechanism in α-Ti and α-Zr: *Julián Roberto Fernández*[1]; Ana María Monti[1]; Roberto C. Pasianot[1]; [1]Comisión Nacional de Energía Atómica, Departamento de Materiales, Avenida Libertador 8250, Capital Federal, Buenos Aires 1429 Argentina

Grain boundary diffusion influence many metallurgical phenomena such as creep, segregation, corrosion, etc. The study of the mechanisms operating on the grain boundary core can shed some light on the relationship between structure and macroscopic properties. On the other hand, despite the technological importance of Zr and Ti few studies relating to grain boundaries can be found in the literature. In this work we perform atomistic studies of the grain boundary self-diffusion by the vacancy mechanism in the hcp structures of α-Ti and α-Zr using computer simulation techniques. Semiempirical interatomic potentials of the EAM type are used to obtain the relaxed structures of the (1-211) and (1-212) tilt grain boundaries. Then, the necessary vacancy properties are calculated in order to evaluate the diffusivity. It is found that the activation energy is less than that corresponding to the bulk. Results indicate that diffusion is faster on the tilt axis than perpendicularly.

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Diffusion Along Grain and Interphase Boundaries in Alpha Zr and Zr-2. 5%Nb Alloy: *Manuel Jose Iribarren*[1]; *Fanny Dyment*[1]; [1]Comision Nacional de Energia Atomica, Materiales, Av del Libertador 8250, Buenos Aires, 1429 Argentina

A critical scope of several works concerning grain and interphase boundaries in α-Zr and Zr-based alloys is presented. Diffusion parameters of self and solute diffusion along grain and interphase short circuit paths are summarized: Zr, Nb, Ni, Fe and Co. Most of them show unusual diffusion behaviour in bulk hcp α-Zr. New data of Cr diffusion along the α/β interphase boundaries (IB) in a Zr-2. 5%Nb alloy are presented. A wide range of temperatures has been studied. The conventional radiotracer technique combined with serial sectioning of the samples was applied in order to measure both grain and interphase boundaries diffusion. Fisher, Bondy-Levy, Martin, Whipple and Suzuoka solutions were employeed for the evaluation of the experimental data. Different mechanisms are proposed and the influence of segregation is discussed in the work. Basic and applied conclusions are presented.

General Abstract Sessions: Adhesion

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

Monday PM Room: 212
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Thomas P. Battle, E. I. DuPont de Nemours & Co., Inc., White Pigments and Mineral Products, Edgemoor, DE 19809 USA

2:00 PM
Indentation Techniques to Measure the Adhesion of Hard Films on Soft Substrates: *David F. Bahr*[1]; Adam L. Olson[1]; [1]Washington State University, Mech. and Matls. Eng. Dept., P.O. Box 642920, Pullman, WA 99164-2920 USA

Indentations can be used to measure interfacial failure in thin films on hard substrates via compressive stresses in the film from the indentation leading to buckling in the film. However, these indentation methods may not be appropriate for hard films on softer substrates. In the current study, various systems of substrates and films are examined; including chromium electroplated onto aluminum and steel, lacquer on brass, and tungsten on a polymer substrate. It is shown through cross sections of the resulting morphology of the indentations that buckle driven delamination can occur when large radial compressive stresses are reached in the substrate. The practical work of adhesion is estimated from buckling theory, and compared to qualitative measurements of adhesion. The effects of out of plane deformation is examined, and it is shown that increased pile up around the indentation can alter the buckling conditions.

2:25 PM
Adhesion and Fracture Testing of Multilayer Films in Hybrid Microcircuits: *Neville R. Moody*[1]; David P. Adams[2]; Alex A. Volinsky[3]; William W. Gerberich[3]; [1]Sandia National Laboratories, Livermore, CA 94551-0969 USA; [2]Sandia National Laboratories, Albuquerque, NM 87185 USA; [3]University of Minnesota, Minneapolis, MN 55455 USA

Interface adhesion is one of the primary factors controlling the reliability of thin films. It is particulary important in hybrid microcircuits with multilayer films and dissimilar metal interconnects. However, the effect of adhesion on reliability is not well known due to difficulties in testing thin metal films. We have therefore begun a study to determine adhesion of these films by measuring interfacial fracture energies with deposition of stressed overlayers and nanoindentation. In some cases, blisters formed spontaneously after stressed overlayer deposition while in others nanoindentation was required to trigger delamination and blister formation. Fracture energies and interfacial toughness values were then obtained from the blisters and spalls using mechanics-based models. In this presentation, we will describe the techniques used to measure interfacial fracture properties of thin films and the results we obtained from testing films in hybrid microcircuits using these techniques. This work supported by U.S. DOE Contract DE-AC04-94AL85000.

2:50 PM
The Evaluation of Elastic Modulus and Adhesion Energy of Diamond-Like Carbon Film with Adhesion Method: Kyu Hwan Oh[1]; Kwang-Ryeol Lee[2]; *Myoung-woon Moon*[1]; [1]Seoul National University, Sch. of Matl. Sci. and Eng./Coll. of Eng., Kwanak-gu, San 56-1, Seoul 151-742 South Korea; [2]Korea Institute of Science and Technology, Thin Film Tech. Rsch. Ctr., P.O. Box 131, Cheongryang, Seoul, South Korea

This study presents a new evaluation method of fundamental adhesion energy of thin films of high residual compressive stress. An elastic instability of the highly compressed film results in a chracteristic buckling from a substrate. Because no external energy is supplied to this system, energy before the buckling should be balanced with that of the buckled system. From the energy balance criteria, we could obtain the fundamental adhesion energy between the film and the substrate which can be defined by the change in total interfacial energy. And the unknown properties, Young's modulus of the film can be independently estimated from the freehang method which could be developed with artificial technique of etching the substrate out at the side of delaminated film. The technique was applied to diamond-like carbon films deposited on glass substrate.

3:15 PM
Production and Studies of AL-SN Bearings with Reticlar Structures: *Mohsen Sadrossadat*[1]; [1]506 Koye Ostadane, Golestan Hwy., Ahwaz, Iran

The practical activities in this investigations can be brief as follows: first different selected alloys cast in permanent molds. Then the cross section of the samples reduced with the rolling machine. In the process of reduction of area of the samples these went throught annealing process enabling them for mechanical work then by suitable heat treatment microstructure modified to reticular. The next step was cleaning and polishing of these items as well as steel backing of bearings. Then on the backing of bearings covered by alluminium foil. Next step was roll cladding (Adhesion between solid surfaces) of the backing and the strip of AL-SN alloys. Tight connections was the result of the process then the prepared set was put in the pre prepared fixture and with the force of heavy press was formed as a paykan vehicle bearings finally prepared bearings was examined by hardness testing,chemical analyses,ultrasonic waves and electromicroscope. The main results of current research can be brief as follows: successfully cladding between backing and the strip of AL-SN alloys, to gain access to reticular structures and to obtain optimum reduction of area in roll cladding.

General Abstract Sessions: Mechanical Properties B

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

Monday PM Room: 202
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Janice Klansky, Buehler Ltd., 41 Waukegan Rd., Lake Bluff, IL 60044 USA; Joachim H. Schneibel, Oak Ridge National Laboratory, Met. and Ceram. Div., Oak Ridge, TN 37831 USA

2:00 PM
The Effect of Thermal-Mechanical Processes on the High Temperature Mechanical Behavior of PM U720 Forged Alloy (-270 Mesh Powder): *Chih-An Yin*[1]; Kenneth A. Green[1]; [1]Rolls-Royce Corporation, Dept. of Mats. and Proc., Speed Code S-52, 2001 South Tibbs Ave., Indianapolis, IN 46241 USA

Gas turbine disks for aircraft engines made from nickel-base superalloys can be fabricated by several processing methods. The choice of a specific processing route depends on a variety of interrelated factors which include design property requirements, material selection and processing costs. It has been shown that the powder metal(PM) superalloys offfer the potential for increasing performance and reducing the weight of gas turbine aircraft engines due to advance in strength. However, a manufacturing route such as PM must yield a product compatible with the design property requirements. For this reason, material characterization testing for tensile, creep-rupture, low cycle fatigue and fatigue crack growth rate properties was performed on PM U720 forged alloy subjected to a specified thermal-mechanical process. The purpose of this study was to examine the effects of thermal-mechanical processes on the high

temperature mechanical behavior of this material with special attention to creep-rupture, low cycle fatigue and fatigue crack growth rate. The analyses are based mainly on the phenomenological approach and predicted methods so that a quantitative comparison between the PM process and the conventional cast-wrought process of U720 forged alloys can be presented. In addition, the fracture morphologies of the materials at 538ºC & 649ºC were also investigated by SEM and optical metallography to determine the relationships of failure mades, relative fatigue and creep-rupture life to the grain sizes and local microstructure resulting from different thermal-mechanical processes.

2:25 PM

Low-Cycle Fatigue and Creep-Fatigue Crack Growth Behavior of Hastelloy X: *Lijia Chen*[1]; Peter K. Liaw[1]; James W. Blust[2]; Paul F. Browning[2]; Rodger R. Seeley[3]; Dwaine L. Klarstrom[3]; [1]The University of Tennessee, Dept. of Mats. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Solar Turbines, Inc., 2200 Pacific Highway, P.O. Box 85376, MZ R-1, San Diego, CA 92186-5376 USA; [3]Haynes International, Inc., 1020 West Park Ave., P.O. Box 9013, Kokomo, IN 46904-9013 USA

The fully-reversed total strain-controlled low-cycle fatigue tests with and without hold times, as well as the constant delta K-controlled creep-fatigue crack growth tests, were conducted at 816ºC and 927ºC in laboratory air on a nickel-based superalloy, HASTELLOY X. It was noted that the low-cycle fatigue life of the alloy considerably decreased due to the introduction of strain hold times, and the longer hold time usually resulted in a larger decrease in fatigue life. However, the test temperature seems to have little influence on the fatigue life at the temperatures used in this investigation. It was found that the crack growth rate per cycle (da/dN) increases with increasing temperature and prolonging hold time. The crack growth rate per unit time (da/dt) was also observed to increase with temperature. With prolonging the hold time, the crack growth rate per unit time (da/dt) was found to decrease at 816ºC, while increase at 927ºC. In addition, the Tensile Hysteresis Energy method was used to predict the fatigue life, which was found to be in agreement with the experimental results. Keywords: fatigue, high temperature alloy, modeling. Acknowledgements: This work is supported by the Solar Turbines Inc., Haynes International, Inc., the University of Tennessee, the U.S. Dept. of Energy's Advanced Turbine Systems Program, the National Science Foundation (NSF), under Grant No. DMI-9724476, and the NSF Combined Research-Curriculum Development Program, under EEC-9527527, with Dr. D. Durham and Ms. M. Poats as contract monitors, respectively.

2:50 PM

Influence of Aging on the Compressive Behavior of Inconel 718: *Christopher L. Hale*[1]; Mark L. Weaver[1]; [1]The University of Alabama, Metall. & Mats. Eng., Box 870202, A129 Bevill, Tuscaloosa, AL 35487-0202 USA

The kinetics of dynamic strain aging has been investigated in aged Inconel 718 over the temperature range 25°C to 800°C using a combination of uniaxial compression tests and metallographic characterization. At intermediate temperatures, the solution-annealed material exhibited dynamic strain ageing over a wide temperature range, which manifested itself in the form of discontinuous yielding (i.e., serrated flow) and negative strain rate sensitivity. In specimens aged at 760°C, the regime associated with strain aging was significantly reduced in size. The species responsible for this behavior are discussed on the basis of microstructural studies by optical, scanning electron, and transmission electron microscopy.

3:15 PM

Chemical Reactions and Mechanism for Oxygen Enhanced Crack Growth in Nickel-Based Superalloys: Christopher F. Miller[2]; Gary W. Simmons[2]; *Robert P. Wei*[1]; [1]Lehigh University, Dept. of Mech. Eng. & Mechs., 327 Sinclair Lab., 7 Asa Dr., Bethlehem, PA 18015 USA; [2]Lehigh University, Dept. of Chem., 305 Sinclair Lab., 7 Asa Dr., Bethlehem, PA 18015 USA

A mechanism involving the formation of a brittle film along grain boundaries ahead of the crack tip has been suggested as the mechanism for oxygen enhanced crack growth in g²-strengthened nickel-based superalloys, such as Inconel 718. To provide support for this mechanism, a surface chemistry study was carried out to determine the reactivity of the alloy matrix, Nb, NbC and Ni_3Nb with oxygen by x-ray photoelectron spectroscopy (XPS). XPS analyses were also made of the fracture surfaces of specimens that had experienced crack growth in oxygen at 973K and subsequently charged with hydrogen and fractured in vacuum. In this presentation, the surface chemistry results are summarized. Their implication respect to the niobium-based mechanism for oxygen enhanced crack growth and the previously proposed mechanisms are discussed. Research supported by NSF, Division of Materials Research, under Grant DMR-9632994.

3:40 PM Break

3:50 PM

Creep Properties of Mo-Mo_3Si-Mo_5SiB_2 Alloys: *Joachim H. Schneibel*[1]; Hua-Tay Lin[1]; [1]Oak Ridge National Laboratory, Met. and Ceram. Div., P.O. Box 2008, Oak Ridge, TN 37831 USA

Cast and annealed Mo-Si-B alloys with the composition Mo-12Si-8. 5B (at. %) consist of a Mo_3Si/Mo_5SiB_2 matrix containing Mo solid solution inclusions. Their melting point is on the order of 2000°C. Creep properties were determined by a combination of tensile creep tests and constant crosshead speed compression tests at temperatures ranging from 1200 to 1400°C. The stress exponent was approximately 3 suggesting viscous dislocation glide. After partial substitution of Mo by 19. 5 at. % W or Nb, respectively, the same crystal structures as in Mo-12Si-8. 5B were found. Due to the larger atomic radii of W and Nb as compared to Mo, the lattice parameters of the different phases increased by up to 2%. Nb, which has a much larger atomic size than W, was much more effective than W in increasing the creep strength. The creep properties of these alloys will be compared to those of other high-temperature alloys. This research was sponsored by the Fossil Energy Advanced Research Materials Program, U. S. Dept. of Energy, under contract DE-AC05-00OR22725 with UT-BATTELLE, LLC.

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Extrinsic Stacking Faults and Twinning in Hadfield Manganese Steel Single Crystals: *Ibrahim Karaman*[1]; Huseyin Sehitoglu[1]; Yuriy I. Chumlyakov[2]; Hans J. Maier[3]; Irina V. Kireeva[2]; [1]University of Illinois, Mech. and Indust. Eng., 1206 W. Green St., Urbana, IL 61801 USA; [2]Siberian Physical-Technical Institute, Revolution Sq. 1, Tomsk 634050 Russia; [3]University of Paderborn, Lehrstuhl f. Werkstoffkunde, Paderborn 33095 Germany

The stress-strain behavior and the microstructure of single crystals of Hadfield manganese steel (12. 3 Mn, 1. 1 C in wt%) were studied at room temperature. TEM observations have demonstrated extrinsic stacking faults (SFs) and twinning in unexpected conditions, the [001] and [15 10] orientations under tension and the [111] orientation under compression. These unexpected formations were utilized to rationale the high strain-hardening behavior of the cases studied. The driving force to overcome the kinematic barrier for the nucleation of extrinsic stacking faults and twinning is facilitated due to the following factors: (i) the effect of the applied stress on the decreasing effective extrinsic stacking fault energy and the mobility of partials, (ii) the stress concentration of pile up dislocations at the dislocation lock (iii) trapping of carbon atoms in a transient structure with the passage of leading partial (Suzuki effect), (iv) local stress field from Mn-C couples. A unique dislocation model was introduced for the extrinsic stacking fault formation and for the nucleation of twinning. These results are critical to better understand the very high strain hardening rates of Hadfield steel polycrystals such that every grain can twin and form additional barriers to dislocation motion. Moreover, these findings point to methods for designing low stacking fault energy materials with high strain hardening capability. This work was supported by the National Science Foundation contract CMS 99-00090, Mechanics and Materials Program, Directorate of Eng., Arlington, Virginia.

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The Effect of Twinning and Slip on the Bauschinger Effect of Hadfield Steel Single Crystals: *Ibrahim Karaman*[1]; Huseyin Sehitoglu[1]; Yuriy I. Chumlyakov[2]; Hans J. Maier[3]; Irina V. Kireeva[2]; [1]University of Illinois, Mech. and Indust. Eng., 1206 W. Green St.,

Urbana, IL 61801 USA; [2]Siberian Physical-Technical Institute, Revolution Sq.1, Tomsk 634050 Russia; [3]University of Paderborn, Lehrstuhl f. Werkstoffkunde, Paderborn 33095 Germany

The Bauschinger effect (BE) in single crystals of Hadfield manganese steel (12. 3 Mn, 1. 1 C in wt%) were studied for three crystallographic orientations, [111], [123], and [001]. Both forward tension-reverse compression (FT/RC) and forward compression-reverse tension (FC/RT) loading schemes were utilized to investigate the role of deformation history on the BE. The evolution of stress-strain response and the dimensionless Bauschinger parameter were utilized to study the BE. The BE stems from long range back stress generated by the dislocation pile-ups at the twin and localized slip boundaries. Twinning boundaries present a strong obstacle and leads to a strong BE. If localized slip follows twinning, permanent softening was evident such as in the case of the [111] FT/RC scheme. Localized slip and multiple slip in the forward loading provided a transient effect in the stress-strain response without a significant permanent softening. Hadfield steel single crystals have demonstrated high BE for orientations cond ucive to combined twinning/slip deformation. The BE increased with increasing prestrain, then saturated and started to decrease in contrast with precipitation hardened alloys. A unique strain hardening approach along with the back stress calculation was introduced into a Viscoplastic Self-Consistent formulation. The strain hardening formulation incorporates length scales associated with spacing between twin lamellae. The simulations correctly predicted the BP and the stress-strain response for both forward and reverse loading. This work was supported by the National Science Foundation contract CMS 99-00090, Mechanics and Materials Program, Directorate of Engineering, Arlington, Virginia.

General Abstract Sessions: Waste Minimization/Sensors and Control

Sponsored by: TMS

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

Monday PM — Room: 230
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Denis Poliquin, Buehler Ltd., 41 Waukegan Rd., Lake Bluff, IL 60044 USA; Brajendra Mishra, Colorado School of Mines, CO USA

2:00 PM

Electrothermal Processing of Converter Slags from Copper Smelters with an Elevated Content of Non-Ferrous Metals: *A. V. Tarasov*[1]; A. D. Besser[1]; [1]State Research Center of Russian Federation, State Rsch. Instit. of Non-Ferrous Mets., 13 Acad. Korolyov St., Moscow 129515 Russia

Technology has been developed for electrothermal processing of converter slags from copper smelters containing more than 3-4% copper, lead and zinc with the use of sulfiding-reducing complex reagent or low-grade coke as reductant. The products of this process were discard slag containing 0. 3-0. 4% Cu, 0. 2-0. 3% Pb and 0. 8-1. 2% Zn; recyclable matte and commercial-grade lead-zinc fumes suitable for subsequent hydrometallurgical treatment. Processing of converter slag using a separate treatment process makes it possible to produce in the main smelting process also discard slag with low nonferrous metals contents. The electric furnace can be fed either with AC or DC electric current, the latter being more preferable.

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Suppression of Surface Hot Shortness in Recycled Steels Containing Residual Copper by Physical Metallurgy: *Seokjong Seo*[1]; *Koji Shibata*[1]; [1]The University of Tokyo, Dept. of Metall., 7-3-1, Hongo, Bunkyo-ku, Tokyo 113-8656 Japan

The most serious problem in recycling steel is surface hot shortness due to residual Cu. That is to say, Cu tends to be enriched at steel/scale interface by the preferential oxidation of Fe and causes liquid embrittlement at the steel surface during hot working. Therefore, it is thought that the most effective method to suppress surface hot shortness is to restrain the amount of Cu-enriched phase at steel/scale interface. In the present research, how to suppress the surface hot shortness due to Cu was investigated by physical metallurgy. Susceptibility to surface hot shortness decreases with an increase in Si and P contents, but the effect of P is saturated at about 0. 02 mass % P. By adding 0. 4 mass % Si with Ni, a smaller content of Ni can be used to reduce susceptibility to the surface hot shortness compared with the case of the single addition of Ni.

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Characterization of Corrosion Products Developed on Immersion-Tested Stainless Steel-Zirconium Alloys That Contain U and Tc: *Dennis D. Keiser*[1]; Nancy L. Dietz[1]; Stephen G. Johnson[1]; [1]Argonne National Laboratory, Nuclear Techn., P.O. Box 2528, Idaho Falls, ID 83403-2528 USA

Argonne National Laboratory is developing an electrometallurgical treatment for spent nuclear fuel. The demonstration of this process has been conducted on U-Zr fuel elements irradiated in the Experimental Breeder Reactor II (EBR-II). This process extracts the usable U from the fuel and places the waste products into stable waste forms. One of these waste forms is a metal alloy comprised of stainless steel (SS), Zr, noble metal fission products (Tc, Ru, Rh, Pd, Nb, etc.), and minor amounts of actinides. Its baseline composition is SS-15 wt. % Zr. To test the performance of the waste form under repository conditions, SS-15Zr alloys have been doped with U (the most prevalent actinide) and Tc (a long-lived fission product that is mobile in aqueous environments). These alloys were immersion-tested in water that simulated the composition of water from Yucca Mountain, a possible location for a geologic repository. The release of the U and Tc into the water was measured and a normalized mass loss and normalized release rate were calculated. In addition, the post-test samples were characterized using scanning electron microscopy and transmission electron microscopy to identify the corrosion products and the location of U and Tc in these phases. The observed U and Tc leaching behavior will be related to the corrosion products found on the surfaces of the tested alloy samples.

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How to Attain Greater Efficiencies for an Aluminium Smelter with a Thorough Plant Information System Implementation Methodology: *Denis Poliquin*[1]; [1]Keops Technologies, Inc., 1155 University St., Ste. 1100, Montreal, Quebec H3B 3A7 Canada

Efficiency is becoming a key element for the long-term survival of every smelter in the world. Plant Information Systems can efficiently process information from SCADA or DCS supervisory computer systems and can become a major tool for improving the efficiency and yield of the plant. The presentation will be based on a methodology KEOPS has developed over the past 15 years that is used during the feasibility study, the detailed project and the construction phases of an aluminium smelter project for all activities related to industrial software and business management system implementation. The methodology presented can be adapted to current smelters who are looking to improve productivity through the use of an integrated plant information technology environment. After many successful projects in the aluminium industry, for both primary and secondary transformation, KEOPS has been internationally recognized for its domain expertise by being awarded contracts for design and implementation of Plant Information Systems around the world.

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Development of the Sensors and Algorithms for Determination of Liquidus Temperature of the Cryolite Melts in Aluminium: *V. N. Putinsev*[1]; A. M. Trufanov[1]; V. G. Kirsanov[2]; O. O. Rodnov[3]; [1]AVTEK, Ltd., Krasnoyarsk, Russia; [2]OBERON-K Limited, Krasnoyarsk, Russia; [3]Krasnoyarsk Non-Ferrous Metals and Gold Academy 95, Krasnoyarsky Rabochy St., Krasnoyarsk 660025 Russia

Devices for determination of the bath and liquidus temperature of the electrolyte and overheating have been developed. Principle of its work is based on the heat transfer to the cold subject in molten media. Measurements are made on the rising part of the temperature-time curve for the sensor. Developed device includes from the sensor, commercial thermocouple and special tip (for multiple use). It has also portable part with memory and algorithm for the data treatment. Device was tested successfully on the industrial cells in Brats and Krasnoyarsk Aluminum Smelters with the accuracy of liquidus temperature determination ±2°C.

4:15 PM

Infrared and Laser-Based Sensors and Systems to Accurately Monitor the Temperature, Level, and Dimension of Molten and Solid Metals: *Francois Reizine*[1]; [1]American Sensors Corporation, 557 Long Rd., Pittsburgh, PA 15235 USA

Infrared sensors will include scanning detectors and positioning sensors. The focus will be on 1, 2, and 4-color wavelength pyrometer systems which allow the accurate measurement of emissivity and, consequently, of the true temperature even in the presence of scale, slag, and fumes. Such sensors are being used in blast furnaces, BOF's and galvanizing lines. Laser sensors will be presented using different principles of physics, mainly, time-of-flight, pulsed infrared lasers for level measurement and dimensional measurement; triangulation lasers for width and thickness measurements; and laser Doppler velocimeters for velocity and length measurements, including mass flow, elongation, and tension control, cut-to-length applications. These sensors and systems are based on state-of-the-art technical developments to improve productivity and quality and reduce maintenance and downtime.

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Development of the Control System for Aluminium Cells Using Neuronets: *O. O. Rodnov*[1]; P. V. Polykov[1]; A. I. Beresin[1]; [1]Toks-Soft-Light Metals Limited, Krasnoyarsk, Russia

Traditional control systems for aluminium cells which include stabilization of psevdoresistence and proper arranged alumina feeding can not be properly adapted to the cells characteristics. As a result target values of resistance (according to the calculated voltage) and target alumina dose should not be considered as optimal. For improvement of the quality of the control we offer to implement neuronet control systems. Neuronets should fulfill the following tasks (for the cells with point feeding systems): filtration of the voltage, alumina concentration determination, regimes of the process calculation (dose and time interval of the feeding, target voltage). Structure of the control system is as well as algorithm neuronet teaching based on the information received from the database are developed. The imitation test of the system has shown that putting of the algorithm and much higher level of regulation is provided.

General Recycling: Topics Related to Heavy Metals and Ferrous Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee

Program Organizer: Guy Fredrickson, Hazen Research, Thermal Processing Group, Golden, CO 80403 U

Monday PM — Room: 219
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Guy Lawrence Fredrickson, Hazen Research, Thermal Processing Group, Golden, CO 80403 USA

2:00 PM

Study of the Pyrolysis Reactions of Brasilian Waste Tires Using Tga and Dta: *Jefferson Caponero*[1]; *Jorge Alberto Soares Tenório*[1]; [1]Polytechnic School, University of São Paulo, Metall. and Mats. Eng., Av. Prof. Mello Moraes, 2463, São Paulo 05508-900 Brazil

The tire is an expressive component of Municipal Solid Waste, manly when its occupied volume is considered. Its treatment by pyrolysis stand for a list of advantages, such as: landfill releasing and a production of a high quality fuel. The exploitation of new natural resources is becoming more difficult each day, basically due to the environmental damages caused by this type of activity. This work presents a study on the thermal behaviour of Brazilian tires using thermogravimetric analysis and differential thermal analysis, in order to optimise the pyrolysis process of tires. Samples of 1 to 2 mm were investigated at atmospheres of nitrogen, argon. oxygen and mixtures of these gases at temperatures up to 1273K, under heating rates between 1 to 50 K/min. The results showed that the behaviour of each sample is related to both the atmosphere composition and the heating rate.

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Recycling of Solid Wastes Containing Iron from Integrated Steelmaking Plant: *Cyro Takano*[1]; Marcelo Breda Mourao[1]; Ramiro Conceicao Nascimento[1]; Guilherme Lenz Silva[1]; Dener Martins Santos[1]; [1]University of Sao Paulo, Metall. and Mats. Eng., Av. Prof. Mello Moraes, 2463, Sao Paulo, S.P. 05508-900 Brazil

The integrated iron and steelmaking generate a large types and quantities of solid dusts and sludge, at different phases of the production units. Within these are: sludge of the Blast-Furnace; coke fines; fine and coarse fractions from oxygen converters; sludge from water treatment at rolling mill unit; and others. In this paper the above dusts and sludge were physically and chemically characterized. The obtained results allowed to define self-reducing pellets using these materials. The high temperature behaviors of the pellets were tested. Good results, with no decrepitation and swelling, and high yield of reduction. show that it is technically feasible to recycle them, as self-reducing pellets, in oxygen steelmaking converter. An estimate of the benefits of such an operation show that it is also economically viable. Key-words: steelmaking dust; recycling; self-reduction.

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Pretreatment for the Recycling of Spent Li Ion Batteries: *Denise Crocce Romano Espinosa*[1]; Marcel Touma[1]; Jorge Alberto Soares Tenório[1]; [1]University of são Paulo, Dept. Metallur. and Mats. Eng., Av. Prof. Mello Moraes, 2463, São Paulo, SP 05508-900 Brasil

Rechargeable lithium batteries are one of the most promising technology in battery development. Although Li ion batteries have been used as an alternative to the more polluting and less performing Ni-Cd batteries, Li ion batteries have limited life and their disposal is uncertain. It is imperative that valuables from wastes, such as lithium, should be recovered. The main objective of the present work was to characterize secondary lithium batteries for cellular phones. This was done using unit operations of mining processing, x-ray diffraction (XRD), chemical analysis and differential thermal analysis (DTA). The unit operations were hammer mill grinding, knife mill grinding and size separation. After the hammer grinding process, the plastic cases were almost intact, however, they have released the cylindrical lithium batteries themselves and these parts correspond to 70% of the total battery weight.

3:15 PM

Vaporization of Mercury Under Vacuum Retort Conditions: *John P. Hager*[1]; Jeanette B. Berry[2]; Antonio E. Blandon[1]; [1]Colorado School of Mines, Metall. & Mats. Eng., 1500 Illinois St., Golden, CO 80401 USA; [2]Oak Ridge National Laboratory, Energy Div., P.O. Box 2008, Oak Ridge, TN 37831-6200 USA

One approach being investigated for the recovery of mercury from acid plant blow-down sludge in copper smelters is the use of a vacuum retort reactor for the vaporization of the mercury. An experimental study was undertaken to investigate the vaporization of mercury when present as a compound in the Hg-S-O system and the Hg-Se system. A transpiration reactor was modified to operate under reduced pressure. Rates of vaporization were measured at several temperatures for the compounds HgSO4, HgSO4*2HgO, Hg2SO4, HgS, HgO, and HgSe. The results were used to calculate the minimum operating temperatures for complete vaporization in a given residence time. Experiments were conducted where the entire sample was vaporized so that mass closure could be established for the condenser system. Recoveries were typically in the range of 96-

99%. Differences were observed in the extent of back reaction during the condensation of the vapor-transported mercury.

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Study of Hg Removal and Zn Recovery from Spent Dry Batteries: Denise Corrêa de Oliveira[1]; Denise Crocce Romano Espinosa[1]; *Jorge Alberto Soares Tenório*[1]; [1]Escola Politécnica University of São Paulo, Dept. of Metall. and Mats. Eng., Av. Prof. Mello Moraes, 2463, São Paulo, SP 05508-900 Brazil

Dry batteries use zinc as the negative electrode. Producers commonly use additives such as mercury to prevent corrosion and to increase the mechanical resistance of the zinc anode. Alkaline batteries also contain it, as an anti-corrosive agent. When mixed with the common municipal solid waste, heavy metals as zinc and mercury pollute soil and ground water supplies, moving to the food-chain and affecting human health. Due to these possible damages, this kind of waste must be properly disposed, or recycled. Thus, even in low amounts (ppm) in batteries, mercury has to be eliminated or, at least, its content should be reduced. This work presents the use of pyromettalurgical processes, to reduce mercury content and recover zinc. The tests involved batteries treatment at temperatures from 300ºC to 1000ºC. Mercury is eliminated up to 650ºC, and zinc is evaporated at higher temperature treatment.

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Recycling Dry and Alkaline Batteries Using Mining Operations: Denise Corrêa de Oliveira[1]; *Jorge Alberto Soares Tenório*[1]; [1]Escola Politécnica University of São Paulo, Dept. Metall. and Mats. Eng., Av. Prof. Mello Moraes, 2463, São Paulo, SP 05508-900 Brazil

Household zinc based batteries, dry and alkaline, have become the most popular sources of electrical energy, and are widely used. Because of their heavy metals content such as mercury, zinc and manganese, spent batteries cannot be directly placed in common landfills without a pretreatment. As a municipal solid waste, these batteries may cause serious health problems and environmental impacts. These damages can be reduced by recovery (which means saving natural sources and energy), recycling and adoption of cleaner technologies. Although this worry has been increasing recently, and producers are doing efforts to reduce the mercury content, batteries still need proper safe disposal conditions or recycling processes. This work presents the efforts in progress to characterize and recycle mixed-dry and alkaline-spent batteries, by using low cost unit operations. Some possible recycling stages and recovery parameters of metals as zinc and manganese are also discussed.

4:40 PM
Vaporization of Zinc and Preparation of Zn(g)-CO-CO2-Ar Mixtures: *Nianxin Fu*[1]; [1]National Institute for Resources and Environment, Mats. Proc. Dept., Onogawa 16-3, Tsukuba, Ibaraki 305-0053 Japan

The behavior of zinc vaporization and condensation in Ar-CO-CO2 mixtures was studied in order to efficiently recover metallic zinc directly from the exhaust gases of electric arc furnace. The zinc vaporization process was monitored under different conditions by continuously measuring the changes in weight of condensed zinc on a condenser with an electronic balance. The relations of zinc partial pressure, condensation rate and recovery were discussed. The features of condensed zinc were also observed. The optimum conditions including the gas compositions, temperature, flow rates of carrier gas and bubbling gas for zinc condensation were determined.

5:05 PM
The Use of Unit Operations of Mining Treatment as the First Step of Ni-Cd Batteries Recycling: *Denise Crocce Romano Espinosa*[1]; Jorge Alberto Soares Tenório[1]; [1]Escola Politécnica University of São Paulo, Dept. Metall. and Mats. Eng., Av. Prof. Mello Moraes, 2463, São Paulo, SP 05508-900 Brasil

The amount of secondary batteries are increasing about 15% by year. Among these batteries, it can be highlighted the Ni-Cd ones, due to their high consumption. In Brazil, the consumption of Ni-Cd batteries grown quickly in the last 5 years, due to the dissemination of cellular telephones and other electronic devices. The first step of a process to recycle spent Ni-Cd batteries is the pretreatment through unit operations of mining treatment. The aim of this work is to define a pretreatment to a pirometallurgical recycling process of spent Ni-Cd batteries used in cellular telephones. Batteries were disassembled by hand to release the different components. The studied batteries are basically composed (by weight) of: 78% nucleus, 18% plastic case and 2% circuits. The grinding of the batteries was performed by knife mill and also by a hammer mill. After grinding the material was screened and submitted to a magnetic separator.

International Symposium on Deformation and Microstructure in Intermetallics: Theory and Modeling

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

Monday PM — Room: 220
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Vasek Vitek, University of Pennsylvania, Dept. of Mats. Sci. and Eng., Philadelphia, PA 19104-6272 USA; K. Sadananda, Naval Research Laboratory

2:00 PM Invited
Micromechanisms of Deformation Behavior in Fe-Al System: *Man H. Yoo*[1]; K. Yoshimi[2]; J. A. Horton[1]; [1]Oak Ridge National Laboratory, Met. and Ceram. Div., Oak Ridge, TN 37831-6115 USA; [2]Tohoku UniversityW, Inst. for Matls. Res., Sendai 980-8577 Japan

Dislocation microstructures developed by {110}<111> slip in B2 FeAl alloys indicate that the mobility of edge dislocations may be as important as that of screw dislocations in understanding the deformation mechanism. Intrinsic glide resistance to screw dislocations by cross-slip pinning has been investigated, but a dislocation mechanism that explains both yield stress anomaly and vacancy hardening is not available. In this work, activation enthalpy for the formation of a jog pair on an edge dislocation is derived by incorporating the differences in elastic interaction energies, APB energies, and internal climb stresses. The critical stress for dynamic breakaway from pinning points created by vacancy-dislocation interaction are obtained for the two limiting cases of low/high temperature and high/low stress. Yielding and plastic flow behavior of Iron-rich FeAl alloys will be discussed in view of the present results.

2:30 PM Invited
Peierls Barrier for Glide Dislocations in the $MoSi_2$ Structure: *Michael I. Baskes*[1]; Richard G. Hoagland[1]; [1]Los Alamos National Laboratory, Structure/Property Relations, MS G755, Los Alamos, NM 87545 USA

Molybdenum disilicide has a body-centered tetragonal, C11b, structure with a surprising number of slip systems. Among these systems, slip on {013} planes involving 1/2 <331> dislocations displays strong asymmetry. The slip asymmetry may be the result of dissociation of screws into three 1/6<331> partials which produce different stacking faults depending upon which direction they move in the slip plane. There is speculation that other partial dislocations on the {013} planes participate in slip. An important factor that determines which of these supposed dissociation products may actually contribute to deformation is the resistance to glide. Accordingly, in this paper we present the results of atomistic simulations in which the resistance to glide was estimated for several potential glide dislocations on the {013} planes. These calculations were based on a modified embedded a tom method (MEAM) potential for $MoSi_2$. This work was supported by the Office of Basic Energy Sciences, U.S. Dept. of Energy.

3:00 PM Invited

Atomistic Simulations of the Structure, Energetics and Forward Mobility of Jogged 1/2<110] Screw Dislocations in L10 TiAl: *Satish I. Rao*[1]; [1]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Gamma TiAl exhibits a yield stress anomaly in the high temperature regime. This is attributed to jog formation on a/2<110] screw dislocations due to their easy cross-slip ability. In this manuscript, atomistic simulations using empirical embedded atom method (EAM) potentials are used to study the structure, energetics and forward mobility of a/2<110] jogged screw dislocations in L10 TiAl. It is shown that the forward motion of jogged screw dislocations is achieved by the formation of point defects. Also, their mobility is a factor of 2 higher than continuum estimates and in reasonable accord with experimental observations of yield stress in the hoigh temperature regime.

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Dislocation Dynamics in L12 Compounds: Implications of Velocity Fluctuations: *Daryl C. Chrzan*[1]; C. K. Erdonmez[1]; [1]University of California, Dept. of Matls. Sci. and Eng., Berkeley, CA 94720 USA

The anomalous yield strength increase observed in some L12 compounds has been linked to the noncompact core structure of the superdislocations which accommodate plastic deformation. It has been argued that these dislocations display a nontrivial pinning-depinning transition as the applied stress is increased. The transition is marked by a significant increase in the amplitude of the velocity fluctuations associated with the motion of a single dislocation. These velocity fluctuations, in turn, have implications for experiment as well as for modeling. Specifically, the velocity fluctuations are characterized by a decay time which should be accessible to mechanical susceptibility measurements. Further, these large scale fluctuations may hinder efforts to extract meaningful predictions for stress vs strain response from simulations employing small numbers of dislocations. This paper considers the implications of the velocity fluctuations, describes the simulations employed to study the fluctuations. This research is supported by the National Science Foundation.

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First Principles Simulation of Ordinary Screw Dislocations in Gamma-TiAl: *Christopher Woodward*[1]; Satish I. Rao[1]; Dennis M. Dimiduk[2]; [1]UES Inc., Mats. and Proc. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [2]Materials, Manufacturing Directorate, AFRL/MLLM Bldg. 655, Wright Patterson AFB, OH 45433-7817 USA

The equilibrium core structure of an isolated a/2<110] screw dislocation is calculated using a first principles pseudopotential planewave method within the Local Density Approximation of Density Functional Theory. The long range strain field of the dislocation is treated using a variation of the recently developed lattice Greens Function Boundary Condition method. This flexible boundary method allows the dislocation to be contained in a very small simulation cell without compromising the fidelity of the final core configuration. In atomistic simulations of the ordinary screw dislocation in TiAl different investigators have found a variety of equilibrium core structures. This suggests that the core is sensitive to the inter-atomic potentials used to describe the local interactions. Here the dislocation core is calculated directly using first principles methods. The equilibrium core structure of ordinary screw dislocations will be compared with previous atomistic calculations.

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Micromechanics of Yiled Strengthening in Lamellar TiAl: Key Deformation Features: *Bimal Kad*[1]; [1]University of California-San Diego, Dept. of Struct. Eng., 409 University Ctr., La Jolla, CA 92093-0085 USA

Fully lamellar TiAl alloys are strengthened primarily by three types of boundaries: grain, lamellar, domain as well as the volume constituent of the Ti3Al phase. Experimental processing efforts designed to produce high strength alloys till date are unable to vary these three grain sizes independently, though the current practices of refining lamellar spacing increase yield strength. In an effort to deconvolute their respective contributions, and to identify key features, yielding behaviour is simulated by Finite Element methods. Polycrystalline flow stress response is computed using as input parameters, a range of scale dependent stresses to activate soft and hard mode deformation. Results show that increasing the hard mode activation stress, to reflect the refined lamellar spacing, by itself does not contribute to the experimentally observed yield strengthening. Discrepancies in such experimental versus theoretical results will be discussed, along with a ranking of key microstructural and deformation features.

International Symposium on Shape Casting of Aluminum: Science and Technology: Fatigue Behavior of Cast Aluminum

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, Dept. of Mech. & Industrial Eng., Urbana, IL 61801-2906 USA; Ray D. Peterson, IMCO Recycling Incorporated, Rockwood, TN 37854 USA

Monday PM — Room: 224
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Paul N. Crepeau, General Motors Company, Powertain Group, Pontiac, MI 48340 USA; Aindrea M. Campbell, Ford Motor Company, Ford Res. Labs., Dearborn, MI 48121-2053 USA

2:00 PM Keynote

Micromechanics-Based Model for Fatigue of Cast A356-T6 Aluminum: *David L. McDowell*[1]; [1]Georgia Institute of Technology, GWW Sch. of Mech. Eng., Dept. of Mats. Sci. and Eng., Atlanta, GA 30332-0405 USA

The high cycle fatigue life of cast Al-Mg-Si alloys is particularly sensitive to the most severe microstructural inclusions such as particles, pores, or oxides. In this lecture, a high cycle fatigue model is introduced which recognizes multiple inclusion severity scales for crack formation. The model addresses the role of constrained microplasticity around debonded particles or shrinkage pores in forming and growing microstructurally small fatigue cracks. The demarcation between high cycle fatigue and low cycle fatigue is identified as the percolation limit for plasticity at debonded particles in the eutectic regions, and corresponds closely with the macroscopic cyclic yield point. Successive fatigue limits are associated with elastic shakedown of microplasticity, arrest of cracks growing from particles or pores, and the long crack threshold for propagation. Microstructurally and physically small crack propagation is based on the cyclic crack tip displacement rather than the stress intensity factor of Linear Elastic Fracture Mechanics. Novel relations to treat multisite fatigue damage are developed for the low cycle fatigue range involving distributed bulk fatigue damage and crack coalescence.

2:45 PM

Predicting Fatigue Properties of Cast Aluminum by Characterizing Propagation and Non-Propagation Behavior of Small Fatigue Cracks: *Michael J. Caton*[1]; J. Wayne Jones[1]; Herwig R. Mayer[2]; Stefanie E. Stanzl-Tschegg[2]; John E. Allison[3]; [1]University of Michigan, Dept. of Mats. Sci. and Eng., 2300 Hayward, Ann Arbor, MI 48109-2136 USA; [2]University of Agriculture, Inst. of Meteor. and Phys., 18 Turkenschanzstrasse, Vienna A-1180 Austria; [3]Ford Motor Company, Dept. of Mats. Sci. and Eng., 2101 Village Rd., Receiving R/Bldg. R, Dearborn, MI 48124 USA

The increased use of cast aluminum in structural components necessitates a deeper understanding of the mechanisms controlling fatigue properties in order to enable improved predictive capabilities. It has been established that the fatigue life of cast aluminum specimens is dominated by the propagation of small cracks that initiate almost exclusively from microshrinkage pores. Cracks have been observed to nucleate from pores after only the first few applications of load, even at stress levels below the fatigue strength. Therefore fatigue performance is determined by the behavior of small cracks existing within the material from essentially the first cycle. If all of the cracks arrest, then the specimen will not fail. Conversely, if at least one crack eventually experiences continuous growth, then the specimen will exhibit a finite fatigue life. Specimens of a 319-type Al alloy with three different solidification conditions were tested using ultrasound at a frequency range of 15 to 20 kHz. S-N results display a marked endurance behavior up to 10^9 cycles for all three solidification conditions. It is shown that the closure-free long-crack threshold, $\Delta K_{th,eff}$, provides extremely good predictions of the experimentally observed fatigue strengths. Small fatigue cracks were monitored using replication and both propagation and non-propagation behavior was characterized. Arrested fatigue cracks existing in specimens that did not fail in 10^8 cycles were examined under incrementally increased stress amplitudes until a critical threshold condition was surpassed and at least one of the cracks grew continuously. These critical threshold conditions are compared to the threshold established for long-cracks, $\Delta K_{th,eff}$. The microstructural features controlling small-crack growth and arrest will be discussed, and a fatigue property prediction approach will be presented.

3:15 PM

The Control of Porosity and Fatigue Life in Aluminum Alloy Castings: *J. Fred Major*[1]; [1]Alcan International, Ltd., Kingston Res.& Dev. Cen., P.O. Box 8400, 945 Princess St., Kingston, Ontario, Canada

Much work has been done over the years in order to develop an understanding of the metallurgical and process variables that control the amount and size of porosity that develops in a cast aluminum part. Simultaneously, work to understand the impact of porosity on mechanical properties, and in particular the fatigue life, has been carried out. What has yet to be developed is a fully quantified and validated linkage between the two efforts. In this paper the major factors which influence and ultimately control the formation and development of porosity in A356 aluminum alloy castings will be reviewed. This will be done using example results drawn from a parametric analysis of a porosity database which covers a wide range of chemical and thermal conditions. The manner in which pores interact with the microstructure to degrade the fatigue life will then be covered. Again, example results drawn from a database of fatigue life data covering a large range of both pore size and microstructural scale will be used to illustrate the complexity of the problem.

3:45 PM Break

4:00 PM

Micro-Mechanisms of Fatigue and Fracture in Al-Si Alloys: *Aindrea M. Campbell*[1]; John E. Allison[1]; [1]Ford Motor Company, Matls. Sci., P.O. Box 2053, MD 3182, SRL, Dearborn, MI 48121-2053 USA

The effect of microstructure on high-cycle fatigue, fatigue-crack growth resistance, and fracture in two cast 319-based Al-Si-Cu alloys-modified and refined by the additions of Sr and TiB2, respectively-are investigated. Particular emphasis is placed on the damage processes during monotonic and cyclic-crack growth as a function of porosity, aluminum dendrite morphology, and Al-Si eutectic distribution. The degradation of crack-tip shielding, e. g., closure, during cyclic loading and its effect on crack-growth rates are examined. The mutual competition of intrinsic microstructural damage mechanisms ahead of the crack tip (which promote crack advance) and extrinsic crack-tip shielding behind the tip (which act to impede it) is investigated.

4:30 PM Invited

Thermo-Mechanical Behavior of Cast 319 Aluminum Alloys: *Huseyin Sehitoglu*[1]; Carlos Engler[1]; Tracy Smith[1]; [1]The University of Illinois, Dept. of Mech. and Indust. Eng., 1206 W. Green St., Urbana, IL 61801 USA

Stress-strain behavior of cast 319 aluminum-copper alloys are studied at high temperatures and under thermo-mechanical deformation exposing rate sensitivity, and microstructural changes. The causes of mechanical behaviors at the macro-scale are discussed based on different precipitates and their variation with temperature and time. A state variable unified constitutive models was developed to characterize the stress-strain response for these materials. The model handles temperature and strain rate effects, and captures the microstructurally induced changes on stress-strain response. The thermo-mechanical fatigue response under in-phase (TMF IP) and out-of-phase (TMF OP) conditions are also simulated. The decrease in strength was attributed to the significant coarsening of the precipitates at high temperatures which was confirmed with electron microscopy. The role of the stress-strain response on fatigue behavior is also discussed. Finally, recent developments to incorporate the different types of precipitates due to various aging treatments into stress-strain formulations will be also presented.

5:00 PM

Aging of Casting Alloys for Cylinder Head Applications: Modeling of the Phenomenon and Life Prediction of the Component: *D. Massinon*[1]; E. Nicouleau Bourles[2]; B. Barlas[3]; G. Cailletaud[3]; [1]Montupet, 67 Rue Jean de La Fontaine, Nogent sur Oise 60181 France; [2]Renault Technocentre, 1 Avenue du Golf, Guyancourt 78288 France; [3]Ecole Nationale Superieure des Mines de Paris, Centres des Materiaux, UMR CNRS 7633, BP87, Evry 91033 France

Operating temperatures in modern, high performance engines, frequently range between 250 and 300° at the hottest location. In such conditions, the aluminum alloys used in the cylinder head experiences ageing. To undertake a reliable thermal fatigue life prediction for the component, it is essential to quantify this ageing phenomenon and to propose a model that takes into account the rate controlling parameters. We have studied the high temperature behavior of two aluminum alloys: 319 and A356. Isothermal low cycle fatigue tests have been used to identify the parameters of the model, including cyclic viscoplasticity and ageing. The model has been implemented in the Z-Set/ZEBULON code and used to derive the number of cycles to failure in thermal fatigue of a diesel cylinder head developed by RENAULT. Using an original meshing and partitioning technique it has been possible to calculate the full component and to identify both crack locations and initiation time. These results have been compared with cracking experienced by the component on a thermal fatigue bench.

Lead-Free Solder Materials and Soldering Technologies II: Microstructure, Alloy Design

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, Dept. 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

Monday PM Room: 227
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Sungho Jin, Lucent Technologies, Bell Labs., Murray Hill, NJ 07974 USA; Mark T. McCormack, Fujitsu Computer Packaging Technologies, San Jose, CA USA

2:00 PM Invited

Alloying Effects in Near-Eutectic Sn-Ag-Cu Solder Alloys for Improved Microstructural Stability and Reliability: *Iver E. Anderson*[1]; James C. Foley[1]; Bruce A. Cook[1]; Joel L. Harringa[1]; Robert L. Terpstra[1]; Ozer Unal[1]; [1]Iowa State University, Ames Lab., Metall. and Ceram., Ames, IA 50011 USA

Environmental concerns and worldwide market forces have accelerated the development of Pb-free solders for electronic assembly. In this development process, a family of near-eutectic Sn-Ag-Cu alloys, based on the Sn-4. 7Ag-1. 7Cu (wt. %) eutectic (T_e=217°C), have emerged with the greatest potential for replacement of Sn-37Pb as a general use solder. The alloy composition range has focussed on Sn-(3. 5 to 4. 0)Ag-(0. 5 to 1. 0)Cu for development, with Sn-3. 8Ag-0. 7Cu and Sn-4. 0Ag-0. 5Cu as commercial examples. This study includes a critical comparison of alloys within the Ag and Cu content ranges in terms of melting and wetting behavior and solder joint microstructure and mechanical properties to suggest the best path forward. Minor 4th element additions, e. g., Co, Bi, and Fe, to Sn-Ag-Cu also were pursued to enhance refinement and stability of the joint microstructure. The 4th element alloying mechanisms and correlation to mechanical property effects will be reviewed. Support received from USDOE-BES, Materials Science Division (contract no. W-7405-Eng-82).

2:25 PM

Studies on Eutectic Sn-3. 5Ag Solder Reinforced with Mechanically Incorporated Ni Particles: *Fu Guo*[1]; S. Choi[1]; J. P. Lucas[1]; T. R. Bieler[1]; K. N. Subramanian[1]; [1]Michigan State University, Mats. Sci. and Mech., 3536 Eng. Bldg., East Lansing, MI 48824-1226 USA

Composite solders tend to render improved properties compared to non-composite solders. A composite solder was prepared by mechanically dispersing 15 vol% of 5 micron size Ni particles into the eutectic Sn-3. 5Ag solder paste. Isothermal aging study at 150°C was performed on small realistic solder joints to study the formation and growth of the intermetallic layers at Ni reinforcement/solder and Cu substrate/solder interfaces. Effects of reflow on microstructure and solderability were also studied using Cu substrates. Nano indentation testing (NIT) was used to obtain hardness, yield strength, and stress exponent for creep from the non-reflowed and multiple reflowed composite solder. Creep tests were carried out on solder joint specimens at 25°C, 65°C and 105°C. The results of these studies are compared with the results previously reported for eutectic Sn-3. 5Ag solder, Sn-4. 0Ag-0. 5Cu solder as well as eutectic Sn-3. 5Ag solder reinforced with Cu or Ag particle reinforcements.

2:45 PM

Observations of Microstructural Coarsening in Micro Flip-Chip Solder Joints: *Monica M. Barney*[1]; J. W. Morris[1]; [1]Ernest Orlando Lawrence Berkeley National Laboratory, Matls. Sci. Div., One Cyclotron Rd., Bldg. 66 Rm 336, Berkeley, CA 94702 USA

To keep up with the increasing demand for size reduction, flip-chip designs are being employed with smaller solder joints. This paper presents a study of microstructural evolution due to thermal cycling and aging in micro solder joints. The lead-tin solder joints in this study have a height of 55±5μm and a tin content of 65-70 wt %. An inhomogeneous, degenerate eutectic microstructure is formed during reflow, with no lamellar microstructure observed. Joint microstructure coarsens initially more rapidly during aging at 160°C than cycling from 0°C-160°C, but tapers off quickly. Coarsening during cycling is linear with time, while coarsening during aging roughly fits a power-law function. Due to the thermal expansion mismatch of the substrates, joints experience 2.8% strain with cycling and fail by 1000 cycles. No coarsened bands are observed, so it appears cracks initiate at regions of greatest stress and propagate toward voids.

3:05 PM Invited

Alloy Modifications to the Pb-Sb-Sn Ternary Eutectic System: *Mark Thomas McCormack*[1]; [1]Fujitsu Computer Packaging Technologies, 3811 Zanker Rd., San Jose, CA 95134 USA

The Pb-Sb-Sn ternary alloy system is reported to contain a ternary eutectic compostion at 85Pb-11.5Sb-3.5Sn which melts at 240ºC and a psuedobinary eutectic composition at 85Pb-10Sb-10Sn which melts at 245ºC. Alloy compositional modifications, as well as the effects of relevant quaternary and quintiary alloy additions will be discussed in terms of melting behavior, wettability during relatively low temperature processing, resultant mechanical properties, and microstructures.

3:25 PM

Investigation of Ag-Bi-Cu-Sn and Bi-Cu-Sn Solder Alloys: *Goran Matijasevic*[1]; [1]Ormet Corporation, 2236 Rutherford Rd., Ste. 109, Carlsbad, CA 92008 USA

Lead-free Ag-Bi-Cu-Sn and Bi-Cu-Sn solder alloy systems are of interest because of their capability of lowering the soldering temperature of tin-based alloys closer to the melting region of Pb-Sn solder. In this work, a number of combinations of these alloy systems are explored. Some of the solders are formed by atomizing solder powder of a given melt composition. The powders are introduced in a flux vehicle and analyzed for their properties. Other solder paste materials are created through combining two or more of constituent alloy powders. The solder paste formulated with these combinations sometimes have multiple melt points on first reflow, followed by a new melt temperature. Differential scanning calorimetry (DSC) analysis of these alloys and their combinations will be presented. Additions of other elements such as In and Sb to these basic systems have also been studied.

3:45 PM Break

4:00 PM Invited

Reactive Solders for Electronic and Optical Packaging: *Sungho Jin*[1]; Hareesh Mavoori[1]; Ainissa G. Ramirez[1]; [1]Bell Laboratories, Lucent Technologies, Appl. Mats. and Metall. Res., 700 Mountain Ave, Murray Hill, NJ 07974 USA

In electronic, optoelectronic and optical devices, there is a need to bond a wide variety of inorganic materials such as semiconductors, nitrides, carbides, oxides, fluorides, and diamond, which are employed as active components, dielectric layers, diffusion barriers, wave guides, and heat sinks. These materials are known to be very difficult to wet and bond with low melting point solders. Conventional bonding techniques for these materials often incorporate additional metallization layers for the ease of soldering. For convenience and simplicity of device assembly as well as for enhanced reliability and broadened design capability, it is desirable to perform a direct solder bonding without the use of metallization layers. We have developed new, Pb-free solders containing some reactive elements which allow direct bonding onto various inorganic surfaces. The solder alloy fabrication, microstructural evolution, interface properties, and mechanical behavior of the solder bonds will be described, and the potential applications of these new solder materials for electronic and optical device packaging will be discussed.

4:25 PM Invited

Interfacial Reactions in the Ag-Sn/Au Couples: *Sinn-wen Chen*[1]; Yee-wen Yen[1]; [1]National Tsing-Hua University, Dept. of Chem. Eng., #101 Kuang-Fu Rd., Sec. 2, Hsin-Chu, Taiwan 300 Taiwan

Ag-Sn alloys are the most promising lead-free solders. Their reactions with Au substrates have been examined by using reaction couple techniques. Sn-3. 5wt%Ag/Au and Sn-25wt%Ag/Au couples have been prepared and reacted at 120°C, 150°C, 180°C and 200°C for various lengths of time. Three phases, δ_2-AuSn, ε_3-$AuSn_2$, and η_2-$AuSn_4$, were found at the interface in all the couples. The thickness of the reaction layers increased with higher temperatures and longer reaction time. Their growth rates in all the couples followed the parabolic law. The average activation energies of the layer growth of the two kinds of couples are 76.7 KJ/mole and 87.7 KJ/mole, respectively. Based on the reaction paths and interfacial morphology, it is concluded that Sn is the fastest diffusion species in the couples.

4:50 PM

Pulsed Electrodeposition of the Eutectic Au/Sn Solder for Optoelectronic Packaging Applications: *B. Djurfors*[1]; D. G. Ivey[1]; [1]University of Alberta, Dept. of Chem. and Matls. Eng., 536 Chem. and Mats. Eng. Bldg., Edmonton, Alberta T6G 2G6 Canada

One of the more promising lead-free solders currently being used in optoelectronic packaging applications is the eutectic Au/Sn alloy (20 at%Sn). The combination of excellent thermal and mechanical properties makes this hard solder well suited for packaging applications in which long-term device reliability is important. As an alternative to the time-consuming solder pastes and preforms currently being used, a method of electroplating the eutectic Au/Sn alloy has been developed. Using a pulsed co-deposition process, it is possible to plate the solder directly onto a wafer in the eutectic composition. Currently, the effects of the various plating parameters on the composition and quality of the film are being studied. The goal is to develop an empirical model linking all plating parameters with the composition and quality of the final film. In addition, the bath chemistry is being studied in order to improve its long-term stability, a crucial step for commercializing the process.

5:10 PM

Microstructral Evolution of Eutectic Au-Sn Solder on Cu/Electroless Ni/Au Substrate: *Ho Geon Song*[1]; John W. Morris[1]; [1]University of California, Berkeley/Lawrence Berkeley National Lab., Dept. of Mats. Sci. and Eng., Mailstop 66-200, One Cyclotron Rd., Berkeley, CA 94720 USA

Both the initial microstructure and microstructural evolution of eutectic Au-Sn solder bumps on Cu/electroless Ni/Au were studied. The solder bumps studied were 150-160 μm in diameter and 45-50 μm tall, reflowed on Cu/electroless Ni/Au, and then aged at temperatures ranging from 125°C to 225°C for up to 350 days. Additionally, Au-Ni-Sn-alloys were made and analyzed to verify the phase change at the interface during aging. The results show that the bulk microstructure is strongly affected by the introduction of Au from the substrate during reflow. Although the Ni3Sn2 intermetallic phase is the primary constituent of the interface, another thin reaction layer was observed. It appears that after aging the evolution of interfacial microstructure is complicated by the P from the electroless Ni layer. The evolution of both the bulk and the interfacial microstructure will be discussed.

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Microstructural Evolution in the Sn-Cu-Ni and Pb-Sn Solder Joints with Cu and Pt-Ag Metallized Al2O3 Substrates: *J. G. Duh*[1]; C. C. Young[1]; [1]National Tsing Hua University, Dept. of Mats. Sci. and Eng., Hsinchu City, Taiwan

The growth mechanism of intermetallics between solders and metallized substrates after thermal aging are investigated. The solders used in this study are unleaded Sn-Cu-Ni solder and eutectic Pb-Sn solder. The Pt-Ag/Al2O3 are employed as the metallized substrates. Microstructure evolution of the interfacial morphology, elemental and phase distribution in the solder joint are probed with the aid of EPMA and X-ray diffraction. Two kinds of intermetallics, Cu3Sn and Cu6Sn5, are formed at the solder/Cu interface. For the solder/Pt-Ag system, only Ag3Sn is observed at the interface. The thickness of Cu3Sn, Cu6Sn5 and Ag3Sn compound layers for all solder metallized substrate systems shows at 0.5 dependence at 100, 125, 150 and 175ºC. According to the calculated activation energy and diffusion constant, it indicates that the growth rate of Cu3Sn and Cu6Sn5 intermetallics in the electroless Cu metallized substrate is relatively higher than that in the Cu block one at the range of 100 to 175°C. The growth rate of Cu6Sn5 and Ag3Sn is reuced in the Sn-Cu-Ni solder as compared to the eutectic Pb-Sn solder. On the other hand, the Cu-Sn-Ni joint exhibits a thicker Cu3Sn intermetallic layer than the eutectic Pn-Sn solder after various aging time at 100°C. However, the thickness of Cu3Sn in the eutectic Pb-Sn solder is thicker than that in the Cu-Sn-Ni solder at 170°C.

Lightweight Alloys for Aerospace Applications: Phase Transformations and Microstructure

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

Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturate Directorate, 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

Monday PM — Room: 213
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Kumar V. Jata, Air Force Research Laboratory, Mats. & Manuf. Directorate, WPAFB, OH 45433 USA

2:00 PM

Precipitation Hardening-The Oldest* Nanotechnology: *Erhard Hornbogen*[1]; [1]Ruhr Universitat Bochum, Bochum DE-44801 Germany

Using mainly aluminum alloys as example a survey is given on mechanism and limits of precipitation hardening. It is discussed how hard, nanometer-size particles can form as an ultra fine dispersoid. A simple example for optimum conditions is provided by diamond cubic particles (Si, Ge) in the f. c. c. Al-Matrix. The role of a sequence of more to less metastable phases is discussed as well as the effects of additional (and trace) alloying elements. From combinations of precipitation hardening with other hardening mechanisms the conditions and limits for ultra high strengths are defined. These are finally applied to other alloy systems and hardening mechanisms such as magnetic hardening. *precipitation hardening in aluminum was discovered about 100 years ago by Dr. Alfred Wilm

2:30 PM Invited

On the Origin of the High Resistance to Coarsening of Ω Plates in Al-Cu-Mg-Ag Alloys: *Christopher R. Hutchinson*[1]; X. Fan[2]; S. J. Pennycook[3]; G. J. Shiflet[1]; [1]University of Virginia, Dept. of Mats. Sci. and Eng., Charlottesville, VA 22903 USA; [2]University of Kentucky, Dept. of Chem. and Mats. Eng., Lexington, KY 40506 USA; [3]Oak Ridge National Laboratory, Solid State Div., Oak Ridge, TN 37831 USA

Alloys based on the Al-Cu-Mg-Ag system have been reported to show excellent creep properties at temperatures up to 200°C. This creep resistance has been attributed to the high resistance to coarsening of the dominant strengthening precipitate, Ω, which forms as platelets on the $\{111\}_\alpha$ planes of the matrix. In this investigation, atomic resolution Z-contrast microscopy has been used to examine the association of Ag and Mg with Ω plates for temperatures between 200°C and 300°C for times up to 1000h. Two atomic layers of Ag and Mg were found to be associated with the coherent broad. faces of the plates at all times and temperatures observed and no segregation was found at the risers of thickening ledges or at the less coherent ends of the plates. Furthermore, within experimental error, no Ag or Mg was found within the Ω plates. Analysis of the thick-

ening kinetics as a function of temperature suggests that the necessary Ag and Mg redistribution around a migrating thickening ledge is accomplished readily and it is concluded that the Ag and Mg segregation is not directly responsible for the high coarsening resistance of these plates. The high coarsening resistance is due to the increasing difficulty of ledge nucleation in what becomes an accumulating vacancy strain field normal to a thickening plate. Furthermore, it is concluded that the segregation of Ag and Mg to the coherent face of the Ω is not to help accommodate the large misfit (~ -9%) between Ω and the matrix as has been speculated in the literature. These experimental observations and conclusions will be discussed in this talk. The authors greatly acknowledge the support of the Southeastern Universities Research Association (SURA) 1999 Summer Cooperative Research Program and the NSF under grant No. DMR9904034. The work at Oak Ridge National Laboratory was supported by the US Dept. of Energy under contract No. DE-AC05-960R22464 with Lockheed Martin Energy Research Corporation.

3:00 PM

The Influence of Local Composition on the Precipitation of $Al_{20}Cu_2Mn_3$ Dispersoids in an Al-4Cu-0. 4Mn-0. 2Si-Fe Alloy: William M. Nemeth[1]; *Thomas H. Sanders*[1]; [1]Georgia Tech, School of Dept. of Mats. Sci. and Eng., Atlanta, GA 30332 USA

The ‘θ (Al_2Cu) phase nucleates, grows, and dissolves as the temperatures increase to the preheat temperature in an Al-4Cu-0. 4Mn-0. 2Si alloy with 0.36 and 0.06 Fe variants. It was found that T ($Al_{20}Cu_2Mn_3$) preferentially nucleates on the plate surfaces of the ‘θ phase, so the as-cast Cu distribution affects the eventual dispersoid distribution after preheating. Higher solidification rates produced a more homogeneous Cu distribution after preheating than did a slower solidification rate. A ramp heating rate of 50°C/hr to the preheat temperature resulted in a more homogeneous dispersoid distribution than did an instantaneous heat-up to the preheat temperature. The microstructural observations will be discussed in light of the solute distribution in the as-cast microstructure and the competitive processes of homogenization and precipitation. The results of this investigation will be compared to similar observation in other aluminum alloy systems.

3:30 PM

Coarsening of Al_3Sc & $Al_3(Sc,Zr)$ Dispersoids in Wrought Aluminum Alloys: Yancy W. Riddle[1]; Monique S. McIntosh[1]; Janet M. Hampikian[1]; *Thomas Sanders*[1]; [1]Georgia Institute of Technology, Schl. of Mats. Sci. and Eng., Atlanta, GA 30332 USA

The addition of scandium to aluminum alloys has the potential of providing an additional disperoid system to help control recovery and recrystallization in wrought aluminum alloys. Like zirconium, scandium forms a coherent particle having the $L1_2$ structure (Al_3Sc) and renders the microstructure resistant to recrystallization. However, from limited coarsening data in the literature on Al-Sc and Al-Zr systems, it appears that Al_3Sc particles coarsen faster than the Al_3Zr particles. Therefore, before scandium can be used in commercial aluminum alloys to control recrystallization it is necessary to relate the coarsening kinetics to recrystallization kinetics. This paper will review the results of a coarsening and recrystallization investigation to determine particle size distributions and average particle size as a function of temperature, time, volume fraction of Al_3Sc, and the influence of other elements such as Zr and Mg. The results of this study will shed light on the application of scandium to certain 5XXX and 7XXX alloys to control recrystallization.

3:55 PM

On the Effect of Stress on Nucleation, Growth, and Coarsening of Precipitates in Age-Hardenable Aluminum Alloys: *Birgit Skrotzki*[1]; J. Murken[1]; [1]Ruhr-University Bochum, Dept. of Mech. Eng., Inst. for Mats., Bochum 44780 Germany

Light metals are the materials of choice for technical applications where low specific weight combined with high specific stiffness is demanded. Their mechanical properties at room temperature are usually well characterized. However, their high temperature behavior has not been studied in great detail. Generally, two approaches can be applied to improve high temperature strength: (i) nanodispersion of precipitates, and (ii) use of intermetallic compounds. In the present study, the high temperature behavior and the accompanying microstructural changes during aging with and without external tensile stress were studied for different age-hardenable Al-alloys. Aging with stress represents creep loading, which is expected to occur in technical aircraft and space application. In the nucleation stage it was found that precipitates are preferentially oriented parallel to an external tensile stress in the solution heat-treated condition of a binary Al-Cu and a quaternary Al-Cu-Mg-Ag alloy. The nucleation of precipitates is strongly affected by an external applied stress and there is a critical value of stress above which preferential nucleation on habit plane variants occurs. The effect of an external stress on growth and coarsening of precipitates depends on the specific alloys and precipitates, respectively. Precipitates present in an Al-Cu-Mg-Ag and an Al-Si-Ge alloy were not affected by the creep parameters used in this study. However, δ’ precipitates present in an Al-Mg-Li alloy grew faster with an external stress applied than under stress free conditions. The coarsening behavior is discussed for pure isothermal aging with respect to the parameters given in the Lifshitz-Slyozov-Wagner equation. The effect of an external tensile stress applied during aging is interpreted using thermodynamic calculations performed by Johnson.

4:15 PM Invited

The Use of Phase Diagrams in the Development of Highly Coarsening Resistant Alloys based on the Al-Cu-Mg-Ag System: *Christopher R. Hutchinson*[1]; B. M. Gable[1]; E. A. Starke[1]; G. J. Shiflet[1]; [1]University of Virginia, Dept. of Mats. Sci. and Eng., Charlottesville, VA 22900 USA

Recent work on the thermal stability of the Ω phase has indicated that the presence of Ag and Mg at the coherent $(001)_\Omega$ faces of the plate is necessary for the stability of a fine and uniform dispersion of this phase. In this respect, phases that introduce chemical potential gradients in Ag and/or Mg must be viewed as capable of jeopardizing the stability of the Ω dispersion. Of particular interest here is the equilibrium S (Al2CuMg) phase which is present in many of the alloy compositions used to study the Ω phase. In addition to introducing potential gradients in Mg, Ag has also been reported to segregate to the S phase at long times. The development of a high coarsening resistant alloy based on the Al-Cu-Mg-Ag system hardened by the Ω phase therefore requires precise knowledge of the Al rich corner of the quaternary phase diagram so that alloy compositions can be chosen which avoid precipitation of the S phase. The development of a quaternary phase diagram and some experimental verification will be presented.

4:35 PM

Effect of Aging on the Microstructure and Precipitation Response of Al-Li-Cu AF/C458: *James Fragomeni*[1]; Kumar Jata[2]; Robert Wheeler[2]; Sona Geoffrey[1]; [1]Ohio University, Dept. of Mech. Eng., 251 Stocker Center, Athens, OH USA; [2]Air Force Research Laboratory, Mats. and Manuf. Directorate, AFRL/MLLM, 2230 Tenth St., WPAFB, OH 45433 USA

Microstructures of the Al-Li-Cu alloy AF/C-458 were studied following single and duplex aging treatments for varying aging times. Transmission electron microscopy was used to characterized the changes in the average size, distribution, morphology, volume fraction, number density, and interparticle spacing of various strengthening intermetallic precipitates. The d’ (Al3Li) and T1(Al2LiCu) intermetallic precipitates were analyzed by quantitative microscopy methods for samples that had a six percent deformation preaging stretch and varying heat treating conditions to determine the effect of artificial aging on the precipitation response and microstructure. The artificial aging response was determined based on hardness measurements. Hardness studies were performed to determine the precipitation hardening response of samples that had similar hardness but different aging conditions. Strength and fatigue crack growth properties for select heat treatments will be related to the TEM and hardness results. The interrelationships between the precipitate characteristics and mechanical properties will be discussed in this presentation.

4:55 PM

In-Situ Formation of AlN Reinforced Al Alloy Composites from Ammonia: *Qingjun Zheng*[1]; Banqiu Wu[1]; Ramana G. Reddy[1]; [1]The

University of Alabama, Dept. of Metallur. and Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

In-situ formation of aluminum alloy matrix composites was investigated. The synthesis of aluminum alloy matrix composites reinforced with in-situ formed AlN particles was achieved in the temperature range of 1273K-1473K by directly bubbling ammonia gas into aluminum alloy melts. Products were characterized using X-ray diffraction, optical microscope, SEM, and EDXA. The thermodynamic analysis of Al-Si-N system was made using Gibbs energy minimization method and the ternary phase diagram at 1473K was calculated. The results showed that the AlN content in the composite is up to 27.4 wt. % and AlN particles are uniformly distributed in the Al-alloy matrix. Formed AlN particles are of non-spherical shape and very small size (<5 μm). The results also indicated that AlN reinforcement is thermodynamically stable in Al and Al-Si alloy matrix in a wide temperature range.

Magnesium Technology 2001: Refining and Recycling

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

Monday PM — Room: 203-205
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Nigel Jeffrie Ricketts, CSIRO, Manuf. Sci. and Techn., Technology Court, Pullenvale 4069 Australia

2:00 PM

Hydrofluorocarbons as Cover Gases for Magnesium Melt Protection: Nigel Jeffrie Rickets[1]; [1]CAST, University of Queensland, Dept. of Min., Mine. and Mats. Eng., St. Lucia 4072 Queenland, Australia

Melt protection is one of the major issues for users and potential users of magnesium. This is particularly so in recent times when the use of sulphur hexafluoride has come under increased scrutiny as this gas is now recognised as the worst greenhouse gas known. It has been demonstrated that sulphur hexafluoride prevents molten magnesium from oxidation by adding magnesium fluoride to the unprotective magnesium oxide surface film. Research with hydrofluorocarbon gases as alternative sources of fluorine is discussed. These gases are much more environmentally friendly than sulphur hexafluoride and are readily available. The use of HFC gases for magnesium melt protection has been patented by CAST. CAST researchers have demonstrated that these gases are effective in preventing magnesium oxidation. Operational aspects of their use are discussed, with particular emphasis on thermal decomposition of the gases.

2:25 PM

Interfacial Reactions Between SF6 and Molten Magnesium: Mary Jane Walzak[2]; Ross Davidson[2]; Stewart McIntyre[2]; *Boyd R. Davis*[1]; Donald Argo[3]; [1]Queen's University, Dept. of Mats. and Metall. Eng., Kingston, Ontario K7P 3N6 Canada; [2]University of Western Ontario, Surface Science Western, London, Ontario N6A 5B7 Canada; [3]Noranda, Inc., Techn. Cen., Pointe Claire, Quebec H9R 1G5 Canada

Sulfur hexafluoride (SF6) gas has long been used as a cover gas for magnesium to suppress oxidation and volatilization of the metal. However, SF6 is a greenhouse gas, and its use is being significantly curtailed under the Kyoto protocol. It is therefore imperative that the protective mechanisms associated with SF6/Mg reaction products be identified, and that alternate cover gases be tested to determine if similar products result from their interaction with molten magnesium. A novel technique has been employed to examine the initial interactions of SF6 and molten magnesium. A stream of cover gas (1% SF6 in dry air) is bubbled through a small charge of molten magnesium contained in a steel crucible. After an induction period, the crucible is rapidly quenched to freeze the bubbles into the solidified magnesium. Under such conditions, the gas bubble/solid interface composition is a realistic reflection of the initial reaction products between SF6 and molten magnesium. These interfaces are revealed by metallographic polishing or fracture, and they are analysed by scanning electron microscopy combined with energy dispersive X-ray spectroscopy (SEM/EDX) and laser Raman spectroscopy. Initial results show magnesium oxide and magnesium fluoride phases are formed with differing stoichiometries.

2:50 PM

SF6 Emission Reduction Partnership for the Magnesium Industry: An Update on Early Success: *Scott C. Bartos*[1]; [1]U.S. Environmental Protection Agency, Climate Protection Div., 1200 Pennsylvania Ave. (6202J), Washington, DC 20460 USA

The SF6 Emission Reduction Partnership for the Magnesium Industry brings together the U.S. Environmental Protection Agency (EPA) and environmentally consious magnesium producers and casting companies in a collaborative effort to protect the climate. Launched in September 1999, EPA and the partner companies are seeking to identify and implement cost-effective technologies that will reduce emissions of sulfur hexafluoride (SF6), a potent greenhouse gas and expensive resource. The voluntary partnership is already yielding successes and accelerating the transfer of information. This presentation/paper will provide an update on the partnership's activities, share the results of the first annual emissions reporting period, and discuss successful emission reduction strategies developed by EPA's partners.

3:15 PM

A New Conti-Process for the Fluxless Recycling of High Purity Magnesium: *Ulrike Galovsky*[1]; [1]Austrian Research Centers Seibersdorf (ARCS), Leichtmetall-Kompetenzzentrum Ranshofen (LKR), Postfach 26, 5282-Ranshofen, Austria

As the production of magnesium die-castings for automotive applications increases, the recycling of inhouse scrap and second generation components plays more and more an important role in the supply of magnesium in the long term. An innovative recycling concept for low-cost recycling of magnesium scrap is presented. The operation of this new fluxfree conti-process for the recycling of return material class 1 is introduced. The components of the remelting unit are explained. The recycled material from this fluxless process is compared with recycled material from flux-based processes in terms of mechanical properties and microstructural analysis. It is shown that the used gas atmosphere in the furnace during melting process has an influence on the inclusion content. The mechanical properties differ markedly according to the atmosphere and the ratio of surface to volume of the used scrap.

3:40 PM Break

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Distillation for Magnesium Recycling: *Tianbai Zhu*[1]; *Naiyi Li*[2]; Xiaoming Mei[1]; *Alfred Yi*[3]; [1]Nanjing Welbow Metals Company, Ltd., Quality & Techn., Jingqiao, Lishui, Nanjing, Jiangsu 211224 China; [2]Ford Motor Company, Manuf. Sys. Dept., 2101 Village Rd., P.O. Box 2053, MD 3135, Rm 3011, SRL, Dearborn, MI 48121-2053 USA; [3]Nanjing Welbow North America Office, 32 Craigton Dr., Apt. 102, Scarborough, Ontario M1L 2N7 Canada

As magnesium consumption grows fast around the world, magnesium recycle becomes a very important project. A new magnesium recycle way, distillation, is developed and tested for recycling magnesium scraps especially for machining chips, oily magnesium, smelting dreg, dross or the mixture. Under a special condition of temperature and environment variable, magnesium in scraps will be gasified, magnesium vapor meets cooler and becomes crystal magnesium crown. This magnesium crown is taken out and used as alloys raw material. Experiments are conducted in different conditions for different scraps. The results show that recycling magnesium by using distillation is a feasible method, the cost of this method is reasonable, and the composition of scraps recycled has a direct relationship with its recovery ratio.

4:15 PM

Mathematical Modeling of the Magnesium Refining Furnace: *Rung T. Bui*[1]*;* Rémy Hachette[1]; Guy Simard[1]; Randy Sheng[2]; Don Argo[2]; Christine Brochu[2]; Michael Smith[3]; [1]Université du Québec à Chicoutimi, Dept. of Appl. Sci., 555 boul de l'Université, Chicoutimi, Quebec G7H 2B1 Canada; [2]Noranda Technology Center, 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada; [3]Magnola Metallurgy Inc., 240 Hymus Blvd., Pionte-Claire, Quebec H9R 1G5 Canada

At Magnola, Noranda's magnesium plant, magnesium is refined using salt-heated, electric furnaces in which the liquid metal comes in contact with the salt and deposits the unwanted inclusions into it by gravity.The metal is poured into the furnace, flows through chambers where it is cooled by the salt and submitted to the refining process before being pumped out to the casting station. The salt is heated by AC electrodes. The process requires proper heating of the salt, good temperature distribution within the salt and the metal, and an appropriate metal flow pattern. As a joint project between the University and industry, a mathematical model has been built, calibrated and validated using plant data.It is three-dimensional, dynamic, and accounts for all the relevant mechanisms including electric heating,heat transfer in the salt,metal and refractories,and fluid flow in the metal.The paper describes the model and shows how it can be used as tool for the analysis and design of the process.

4:40 PM

A New Technique for Rapid Assessment of the Cleanliness of Liquid Magnesium: *Yu Fang*[1]; Shang Shixian[1]; [1]Nanjing Welbow Metals Co., Ltd., Div. of Res. and Dev., Jingqiao, Lishui, Nanjing, Jiangsu 211224 China

A new technique has been developed, which is capable of quick, easy, and inexpensive assessment of the cleanliness of liquid magnesium. The technique is based upon filtering the same quantity of liquid magnesium through two steel tubes, one tube is covered with a filter, another is not, which are placed in the liquid magnesium at site, and measuring the different times the liquid magnesium need to pass, and analyzing the ratio of the different times which are taken during the filtration. Then the rapid assessment of the cleanliness of liquid magnesium can be obtained. The whole evaluation process takes just several minutes. The technique is particularly suited for quality control during magnesium melting and refining. Also the inclusions in liquid magnesium can be collected on the filter. The following metallographic analysis can provide the numbers and identification of inclusions.

Materials Processing Fundamentals II

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

Monday PM — Room: 218
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: David H. DeYoung, Alcoa, Ingot & Solidification Platform, Alcoa Center, PA 15069-0001 USA

2:00 PM

Quantitative Analysis of the Effect Some Fluxes in Several Pyrometallurgical Processes: *Florian Kongoli*[1]; Ian McBow[1]; [1]FLOGEN Technologies, Metall., P.O. Box 49529, CP du Musee, Montreal, Quebec H3T 2A5 Canada

Fluxes are an important regulatory tool in several pyrometallurgical processes. They are used among others to modify the chemical composition of the slags in order to decrease the liquidus temperature, improve viscosity etc. Fluxing strategies have become in fact an indispensable step in several industrial processes since they help to increase the efficiency of smelting and converting processes and to improve the quality of the products. However, the effects of several fluxes used in practice today are known only empirically and sometimes they have been globally asserted without taking into account the characteristics of individual processes, the positioning of the initial slag composition or the particularities of certain laboratory procedures used to assert these effects. In the today's reality of frequent changes in the composition of the raw materials and that of the fluxes themselves, in existing or new developing technologies, the quantification of the effect of several fluxes becomes indispensable. In this work, the quantification of the effect of several fluxes in certain smelting processes has been carried out in close relation to individual characteristics of these processes. Several examples have also been given in order to demonstrate the fact that when taken outside the context some fluxes can become in fact anti-fluxes.

2:25 PM

Fundamental Studies on the Removal of Tramp Elements from Steel: *Luben Petrov Savov*[1]; Shiwei Tu[1]; Dieter Janke[1]; [1]Institute of Iron and Steel Technology, Leipzigerstr. 34, Freiberg, Sacshen D-09596 Germany

This paper presents recent research works on the removal of the tramp elements Cu, Sn, Zn and Pb from steel. The evaporation of Cu and Sn in iron-based melts treated at reduced pressure of the gas phase was studied in a vacuum induction melting furnace. The effect of chamber pressure, temperature and melt composition was investigated. Since the main source of contamination of steel with Sn and Zn is the recycling of tinplate and zinc coated steel, respectively, possibilities for the removal of metallic coatings in a scrap pretreatment stage were studied, too. Tin was removed in the temperature range 400-550°C by treatment with reactive gases featuring high sulphur potential. Zinc can be removed by evaporation or by a combination of thermal and mechanical treatment. The paper discusses the thermodynamics of Pb in carbon-saturated iron melts, too.

2:50 PM

Physical Properties of Selected Brazing Filler Alloys: *Mario F. Arenas*[1]; Viola L. Acoff[1]; Ramana G. Reddy[1]; [1]University of Alabama, Box 870202, Tuscaloosa, AL 35487 USA

A suitable selection of the filler alloys is vital for producing satisfactory brazed joints. The wettability of filler alloys with basemetals depends on physical properties such as surface tension, specific gravity, melting point, and viscosity. Other properties including thermal conductivity and electrical resistivity are also important since they are frequently required to have similar values to

that of the base metal. In this paper, the physical properties of liquid alloys relevant to brazing have been evaluated. Six different filler alloys were analyzed: Ag-, Al-, Au-, Cu-, Ni-, and Ti-based alloys. Results showed that the viscosity values of most brazing filler alloys are in the order of 2 to 8 MPas with Cu and Al alloys exhibiting the lowest viscosities. The surface tension of brazing alloys are in the range of 800 to 1800 mNm^{-1}. The lowest surface tension values corresponded to Ag and Al alloys which consequently have increased wettability. Thermal conductivity and electrical resistivity ranged between 30 to 200 $Wm^{-1}K^{-1}$ and 17-300 $\mu\Omega cm$, respectively. Implications of the results on industrial applications are also discussed.

3:15 PM

Surface Modification of Aluminides Processed by a Plasma Arc Lamp: *Sherman A. McElroy*[1]; Ramana G. Reddy[1]; Vinod K. Sikka[2]; Craig A. Blue[2]; [1]The University of Alabama, Dept. of Metall. and Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 25487 USA; [2]Oak Ridge National Laboratory, Met. and Ceram. Div., P.O. Box 2008, Oak Ridge, TN 37831 USA

A series of experiments have been conducted on FeAl, Fe3Al, and TiAl, to explore the possibility of applying high-power arc lamp processing on aluminides and to help in the understanding of infrared processing of aluminides. SEM, XPS, and optical microscopy were used to determine the surface morphological, chemical, and compositional characteristics of the Vortek processed samples.

3:40 PM Break

4:00 PM

Electrochemical and Shape-Characteristic Aspects of Electrolytically Precipitated Copper Powders: Anita R. Kang[1]; *Gerard P. Martins*[1]; [1]Colorado School of Mines, Metallur. and Mats. Eng., Golden, CO 80401 USA

The production of copper powders by electrolysis of a sulfate electrolyte is considered "a well-developed (mature) field". Paradoxically, although production facilities in North America have been placed out of service during the latter two decades of the last century, the demand for this type of powder now represents a growing market. The principal reasons have been the cleanliness and consistent physical-characteristics from lot to lot, the wide range of bulk (apparent) densities and the high green-strength of powder-metallurgy parts manufactured with these powders. The recent emergence of technology by Electrolytic Copper Products Limited (AZ, USA), both in continuous electrowinning of powders and their subsequent use for high-rate powder extrusion of wires, tubes and other shapes has added a new dimension to this field. The paper to be presented reports on a laboratory study conducted with a sulfate electrolyte (and copper anode), in which the influence of particle size and (dendrite) shape were investigated. In addition, the theoretical aspects of the galvanostatic cell operation, typical of commercial systems, has been reexamined in deference to the earlier definitive contributions by Popov and co-workers.

4:25 PM

Dissolution of Scrap into a Liquid Bath Stirred with Mitsubishi Top Injection: *Fumito Tanaka*[1]; Nozomu Hasegawa[1]; [1]Mitsubishi Materials Corporation, Central Rsch. Instit., 1-297 Kitabukurocho, Omiya, Saitama 330-8508 Japan

In the Mitsubishi Continuous Copper Smelting & Converting Process, the heat for the dissolution of scraps is effectively supplied from the melts strongly stirred by top injection. Various kinds of shape of scraps, not only powder but also lump, sheet, cube and so on, can be treated, without any emission of dust and fugitive gas, and no additional furnaces for scrap processing is necessary. Therefore, this process is much suitable for recycling. The authors have investigated the melting rate of ice sphere into a water bath equipped with top blowing lances. Hydrodynamic experiments were carried out, and mathematical models were applied for the results. The results show that the melting rate of ice sphere strongly depends on the standing waves on a bath surface.

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Overview of NSR Intermediate Strategic Expansion Plan: *Masayuki Kawasaki*[1]; Osamu Iida[1]; [1]Mitsubishi Materials Corporation, Naoshima Smelter & Refinery, 4049-1, Naoshima-cho, Kagawa-gun, Kagawa 761-3110 Japan

Since start-up the larger Mitsubishi Continuous Copper Smelting Process in 1991, furnace relining campaign was gradually prolonged to once every 2 years according to deregulation of governmental boiler inspection. On the other hand, in 1998 NSR embarked on an intermediate strategic expansion plan to strengthen its international competitiveness. As part of this plan, modifications were implemented in the copper smelter designed to increase output of anodes from new charge source to 270,000 tpa. In May 1998, treatment of dried neutralized residue was started in C furnace, and during the shutdown for brick relining in April 1999, intensive modifications to the boilers as well as the installation of new spray coolers for furnace offgas treatment were implemented. Finally, the new larger oxygen plant was commenced its operation in May 2000 and the expansion was achieved around 30% increasing of anode production from original design with no significant troubles.

Materials & Processes for Submicron Technology: Materials and CMP Related Issues

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, Department of Mechanical Engineering, Tampa, FL 33620 USA; Sailesh Merchant, Lucent Technologies, Orlando, FL 32819 USA; Mahesh Sanganeria, Novellus Systems, Inc., San Jose, CA 95134 USA

Monday PM — Room: 226
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Steve Lassig, Lam Research Corporation, 4650 Cushing Pkway, Fremont, CA 94588 USA; Shlomo Berger, Technion, Mats. Eng., Haifa 32000 Israel

2:00 PM Invited

Sputter Deposition of Ta/TaN Diffusion Barriers for Cu Interconnects: *Hao Zhang*[1]; [1]Tosoh SMD, Inc., R&D, 3600 Gantz Rd., Grove City, OH 43123 USA

Copper has been used as an interconnect material in sub-0. 18 micron IC devices because of its low resistivity, excellent electromigration and stress migration resistance. However, Cu can readily diffuse into Si and SiO2-based dielectrics, causing degradation and failure in IC devices. Therefore, the application of Cu interconnect requires an effective diffusion barrier which can prevent Cu from diffusing into Si and SiO2. Among many barrier materials have been studied, Ta and TaN are reported to have excellent diffusion barrier properties between Cu and Si. Sputtering is an effective method to deposit Ta and TaN in the Cu/barrier/Si or Cu/barrier/SiO2 structures, and has attracted considerable attention. In this study, the effects of process parameters such as N2/Ar flow ratio on film properties such as electrical resistivity, mechanical stresses and film uniformity were studied. In addition, the Ta film thickness distribution across 200 mm Si wafer was simulated by using SIMBAD, and compared to the experimental results. The phases and the crystallographic texture of the Ta and TaN films were studied by using X-ray diffraction and pole figure analysis.

2:30 PM Invited

Amorphous Structures of Buried Oxide Layer in SiC-On-Insulator Wafer: *Manabu Ishimaru*[1]; [1]Osaka University, The Insti. of Sci. and Indust. Res., 8-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan

Microstructures of oxygen ion implanted SiC have been examined using transmission electron microscopy (TEM) and scanning transmission electron microscopy equipped with an energy-dispersive X-ray spectrometer. 6H-SiC (0001) substrates were implanted with

180keV oxygen ions at 650°C to fluences of 0. 7x10^{18} and 1. 4x10^{18} /cm^2. A continuous buried oxide layer was formed in both samples, while the surrounding 6H-SiC contained minimal damage. These results suggest that oxygen implantation into SiC is a useful technique to establish SiC-on-insulator structures. In bright-field TEM images, the amorphous layer possessed uniform contrast in the low-dose sample, while it consisted of three distinct layers in the high-dose sample: (1) a bubbled or mottled layer; (2) a dark contrast layer; and (3) a light contrast layer. Chemical measurements revealed that the bubbled and light contrast regions have low silicon and oxygen contents, while carbon enrichment was found in these layers.

2:50 PM Invited

Processing of Ta205 Powders for Electronic Applications: *Raj Singh*[1]; [1]OSRAM Sylvania, Chems. Res. and Dev., Hawes St., Towanda, PA 14848 USA

Tantalum pentoxide (Ta2O5) has been increasingly used in electronics applications such as high K materials for gate dielectrics, in the preparation of surface acoustic wave filters, pyroelectric infrared sensors and optoelectronic devices. High purity tantalum pentoxide is also required for the preparation of tantalate X-ray phosphors for X-ray intensifier screens. The particle size, surface area, morphology and purity of tantalum pentoxide are critical for its application in the above-mentioned products. This paper would discuss various methods of the processing of tantalum pentoxide powders suitable for electronic applications. Specifically, purity, typical morphology and particle size of Ta2O5 powders prepared from various methods will be presented.

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Oxide Pattern Density and Deposition Profile Effects on Shallow Trench Isolation Chemical-Mechanical Polishing: *Young-Bae Park*[1]; J. Y. Kim[1]; H. H. Ryu[1]; W. G. Lee[1]; [1]Hyundai Electronics Industries Co., Ltd., L15 Proc. Dev. Team, System IC R&D Cen., 1, Hyangjeong-dong, Hungduk-gu, Cheongju-si 361-725 Korea

Based on experimentally obtained interaction distance, new test masks for characterizing and modeling pattern dependent variation of the remained thickness after chemical-mechanical polishing(CMP) are designed. Using these masks, we characterize polishing behavior with layout pattern density and pitch variations. Also deposition profile effects are compared between PETEOS(Plasma Enhanced Tetra Ethyl Ortho Silicate) and HDP(High Density Plasma) oxide in STI(Shallow Trench Isolation) CMP. Both remained silicon nitride thickness and expected oxide pattern density considering deposition profile effects show a good correlation with respect to pitch variation for a constant layout pattern density. And the relation between the remained silicon nitride thickness and the true layout pattern density are deduced. Also, the remained thickness increases nearly linearly with the layout pattern density for a constant layout pitch, which can be explained from the simple pattern density model.

4:10 PM Invited

Dishing and Nitride Erosion of STI-CMP for Different Integration Schemes: *Daniel Lim Lim Hwee*[1]; [1]Chartered Semiconductors Manufacturing, Ltd., Special Project, 60 Woodlands Industrial Park D, St. 2, Singapore S738406 Singapore

Shallow Trench Isolation (STI) is an enabling technology for the isolation technique of choice in the fabrication of advanced integrated devices. In STI scheme, STI-CMP is one of the critical processes since good CMP polish uniformity is a key to a tight Vt - distribution in the high-density array. Greatest challenge in STI-CMP is to provide global planarization with minimum dishing and nil nitride erosion. In addition, STI integration scheme is also one of the key factors in improving dishing performance. In this paper we report on the dishing and nitride erosion studies after STI-CMP for three different schemes: (1) direct polishing (2) with reverse etch and (3) oxide dip and reverse mask on 0.18 um test chip. CMP was carried out on two polishers with different consumable sets. The amount of dishing and nitride erosion also depends on width of the active lines and space in between them. In the present investigation, we have studied the dishing and nitride erosion using Tencor Profiler, AFM and X-SEM.

4:40 PM Invited

Scanning Ultrasonic Study of CMP Pads: *A. Belyaev*[1]; .I Tarasov[1]; F. Diaz[1]; W. Moreno[1]; S. Ostapenko[1]; [1]University of South Florida, 4202 E. Fowler Ave., Tampa, FL 33620 USA

We have developed an automatic scanning ultrasonic transmission (UST) system, which allows nondestructive metrology and analysis of visco-elastic properties in full-size CMP pads prior to their use in CMP processing. The system consists of a specially designed ultrasonic transducer as an emitter of acoustic vibrations and an ultrasonic probe as a receiver. The probe is aligned with the center of the transducer and measures, with high accuracy and repeatability, the amplitude if transmitted ultrasonic vibrations through the pad either in a contact or non-contact mode. The UST system is completely computer controlled and fully automated. The scanning UST measurements were performed on commercial donut-shaped CMP pads with an external diameter of up to 32". A noticeable inhomogeneity of the pads was revealed and quantitatively analyzed. The UST metrology is applicable as a quality assurance means in CMP for microelectronics.

Second Global Symposium on Innovations in Materials Process & Manufacturing: Sheet Materials: Innovative Tooling and Forming Methods

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

Program Organizers: Mahmoud Y. Demeri, Ford Motor Company, Manuf. Sys. Dept., Northville, MI 48167 USA; Iver Anderson, Iowa State University, Ames Lab., Ames, IA 50011-3020 USA; David L. Bourell, University of Texas, Mech. Eng. Dept., 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

Monday PM — Room: 228
February 12, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Klaus Siegert, University of Stuttgart, Stuttgart D-70174 Germany; John Papazian, Northrup Grumman Corporation, Bethpage, NY 11714 USA

2:00 PM Invited

Current Status and Future Directions in Stamping Sheet Metal: *Klaus Siegert*[1]; [1]University of Stuttgart, Inst. for Met. Form. Techn., Holzgartenstrasse 17, Stuttgart 70174 Germany

Single action presses with CNC-controlled hydraulic cushion systems in the press table and new segment elastic draw dies provide a robust press system which can be easily adjusted. Such a system can accomodate integration of closed loop circuits to produce good parts even when tribological conditions (lubrication, lubricant, sheet surface) change. Pulsating blankholder forces and height adjusted draw beads can help to stamp difficult-to-form sheet metal such as HSLA-steels and aluminium alloys.

2:30 PM

Segmented Die with Local Adaptive Controllers in Sheet Metal Forming: *Jian Cao*[1]; Brad L. Kinsey[1]; [1]Northwestern University, Dept. of Mech. Eng., 2145 Sheridan Rd., Evanston, IL 60208 USA

The proposed forming technology using segmented die with local adaptive controllers offers the capability of imposing the desired deformation paths of material points inside the forming zone. Compared with the traditional deep-drawing process, the external energy is now applied locally at discrete locations in the punch area in a controllable manner. Consequently, a more optimized strain-path trajectory can be imposed in sheet metal forming leading to a higher

forming depth without failures. The advantage of this system will be demonstrated through forming of an aluminum Tailor-welded blank where the formability of the blank is reduced significantly due to the welding process prior to the forming. The method to determine the location of the controllers, the control strategy, the selection of the tooling tips and the experimental implementation will be presented.

2:50 PM

Innovative Tooling for Sheet Metal Forming: *John M. Papazian*[1]; Elias Anagnostou[1]; Robert Christ[1]; David Hoitsma[1]; John Melnichuk[1]; Patricia Ogilvie[1]; Allan Pifko[1]; Robert C. Schwarz[1]; [1]Northrop Grumman, Tech. Dev. A01-26, S. Oyster Bay Rd., Bethpage, NY 11714 USA

The "discrete-die" tooling concept has been scaled-up and adapted to production-scale forming of sheet metal parts for aircraft. In this concept, the tooling surface is made up of the hemispherical ends of individual pins, where each pin has a square cross section and can be independently moved up and down. The tool is capable of assuming an arbitrary three-dimensional shape within its working volume of 4 ft. by 6 ft. by 1 ft. The availability of an easily reconfigurable tool facilitates minor shape adjustments when required, and associated software permits calculation of springback compensated tool shapes. The reconfigurable tool is a critical element of a "one-of-a-kind" parts factory where fixed tooling is eliminated and parts are produced directly form CAD files. The technical challenges of producing a smooth part surface and controlling the final shape of the part have been addressed in this program. Recent data from shake-down trials in a production facility will be presented. Partially supported by the DARPA Flexible Fabrication Program through ONR Agreement N00014-95-2-0003. DARPA Program Manager-Dr. W. Coblenz, ONR Program Manager-Dr. George Yoder.

3:10 PM

Sheet Hydroforming: State of the Art: *Ralf Kolleck*[1]; [1]Schuler SMG GmbH & Company KG, New Form. Tech., Louis-Schuler-StraBe 1, Waghausel 68753 Germany

In this paper the procedure of Active Hydromechanical Deep Drawing and its advantages are described. Large extensive deep drawn parts (e.g. roof or door panels in the automotive industry) often show minor denting resistance in their middle part. This results from the lower stretching of these regions in the conventional deep drawing process. Active Hydromechanical Deep Drawing based on a working media, offers the possibility to compensate this disadvantage by pre-stressing the sheet. The process also includes a simplification of the tool and thereby a reduction of investment and manufacturing costs. Another advantage is the flexibility of the system. With only one tool system, various sheet materials (steel, aluminium alloys, high-strength steel) and different sheet thicknesses can be processed. Numerical investigations give the opportunity to optimize the tool design. Active Hydromechanical Deep Drawing produce good parts with optimum properties.

3:30 PM Break

3:50 PM Invited

Electromagnetic Impulse Assisted Stamping: Applications and Analysis: *Glenn S. Daehn*[1]; Peihui Zhang[1]; Vincent J. Vohnout[1]; [1]Ohio State University, Dept. of Mats. Sci. and Eng., OH USA

This presentation describes a fundamentally new and different way of forming sheet metal that involves the use of traditional stamping on regions of a component where stamping is effective and using an elecromagnetically induced mechanical impulse where it is needed. The electromagnetic part of the forming is based on the Loretz repulsion that is always present when a current pulses through a conductor placed in a component with high electrical conductivity. The electromagnetic pulse can induce very high velocity deformation. This in turn gives rise to inertial effects that can improve formability and inhibit wrinkling. There are many ways stamping and electromagnetic forming can be integrated as will be demonstrated with examples. Also, this presentation will show how simulations can aid in the design of integrated processes for the production of class-A surface panels. Finally a road map to the widespread use of this technology will be broadly discussed.

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Superplastic Forming of Aluminium Sheets: *Mihai Vulcan*[1]; [1]University of Stuttgart, Inst. for Met. Form. Techn., Holzgartenstrasse 17, Stuttgart 70174 Germany

The purpose of this investigation is to study the superplastic forming of AA5xxxx aluminum alloys. The number and size of cavities that develop during superplastic forming can be reduced by choosing the proper forming parameters. Cavity reduction is important in increasing the forming potential of superplastically formed parts for subsequent cold forming operations which are necessary for part dimensional control and precision. Hydraulic bulge tests, using a special synthetic fluid, were conducted. The advantages of using liquids versus gases include even temperature and pressure distribution as well as a more acurate strain rate control.

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Tool Heating Concepts for Deep Drawing of Magnesium Sheets: *Eckart Doege*[1]; Wolfgang Sebastian[2]; Klaus Droeder[2]; Gerrit Kurz[1]; [1]University of Hanover, Inst. for Met. Form. and Met. Form. Mach.Tools; Welfengarten 1A, 30167 Hannover, Germany; [2]Volkswagen AG, Grp. Res., Environ. and Transp. Mats., Letter box 1777, 38436 Wolfsburg, Germany

According to the demand of reducing the fuel consumption, it is necessary to decrease the weight of automobile constructions by use of lightweight materials. Especially magnesium alloys become more and more important because of their low specific weight. A promising alternative to large surfaced and thin die casting parts has been observed in components manufactured by sheet metal forming. Magnesium alloys show a limited formability at room temperature. A considerable improvement of the forming qualities can be reached by heating the material. This paper describes, how certain process parameters, like temperature, influence the deep drawing process. In this context, a new heated tool concept that improves the deep drawing quality will be introduced. Also, an overview of the part quality of magnesium alloys formed at elevated temperatures will be given. The results of these deep drawing tests led to the conclusion that it is possible to replace conventional aluminum sheet metal parts by magnesium sheet metal parts.

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Ceramic Tools for Sheet Metal Forming: *Jens Stefan Mueller*[1]; [1]Dipl.-Ing., DaimlerChrysler AG, Van Dev.-Adv. Eng. (ET/KG), Mercedesstraβe 137, 70546 Stuttgart, Germany

Ceramic materials are characterized by high hardness, high wear strength and a low affinity to metallic materials. Therefore ceramic surfaces are suitable for application in tools for sheet metal forming. They show, particulary by using steel and aluminum sheet metal, a very low friction and excellent wear behavior. The use of ceramic tools leads to considerable cost savings which have to be put down to a reduction of the amount of lubrication, a lower maintenance effort and a higher tool lifetime in the volume production. The downtimes of the presses can be reduced by the low maintenance effort, which leads to a more effective use of presses in connection with a higher production speed.

Solution Concentration and Purification in Aqueous Processing

Sponsored by: Extraction & Processing Division, Aqueous Processing Committee
Program Organizer: Akram Alfantazi, Laurentian University, School of Engineering, Ontario P3E 2C6 Canada

Monday PM Room: 221
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Norbert L. Piret, Piret & Stolberg Partners Consulting Engineers, Duisburg D-47279 Germany

2:00 PM
Reactions of Arsenic with Oxygen in Abiotic and Biotic Systems: *Batric Pesic*[1]; Victor C. Storhok[1]; [1]University of Idaho, Coll. of Mines, McClure Hall, Moscow, ID 83844-3024 USA

Reactions of arsenic with oxygen are of importance from biohydrometallurgical processing and environmental point of views. It is well known when As(III) is oxidized to As(V) that As(V) can readily be removed by precipitation reaction with ferric iron as ferric arsenate. The literature information is polarized (to be reviewed) with respect to possibility of As(V) formation from As(III) by oxidation with oxygen. This paper will examine the reactions of As(III) with oxygen under various conditions such as: effect of partial pressure of oxygen, temperature, purity of water, the presence of solid surface such as pyrite, etc. Finally, reactions of As(III) with oxygen will be examined in biotic systems in the presence of Thiobacillus ferrooxidans.

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Use of Calcium Silicate and Magnesium Oxide for Precipitation of Heavy Metals: *Batric Pesic*[1]; [1]University of Idaho, Coll. of Mines, McClure Hall, Moscow, ID 83844-3024 USA

Calcium silicate and magnesium oxide were used for precipitation of heavy metals from aqueous solutions. The sources of calcium silicate and magnesium oxide were slag and condensate, respectively, produced by Northwest Alloys (Subsidiary of Alcoa). Solutions containing heavy metals were either synthetic solutions or real mine waste waters. The major parameter explored was the stability of heavy metal precipitates prepared by treatment with calcium silicate and magnesium oxide in comparison to the precipitates prepared by lime addition. Stability tests of metal precipitates were very long, more than 1000 days. It was found that calcium silicate produced more stable precipitates than lime. MgO precipitates were least stable. The chemistry of calcium silicate with water and some of the heavy metals will be reviewed. It should be noted that calcium silicate is a major component of Portland cement whose reactions with water surprisingly are still not well understood. One should appreciate therefore the complexity of systems containing heavy metals. Therefore the chemical reactions of calcium silicate with heavy metals represents a new and very interesting area to study.

2:40 PM
Chalcopyrite Leaching with Silver Salts; Properties of Ag2S Film: *Charles Irwin Richman*[1]; [1]University of Nevada Reno, Makay Sch. of Mines, Dept. Metall. Eng, Mail Stop 388, Reno, NV 89557-0136 USA

The present work is a study on the effect of silver sulfide on chalcopyrite leaching. Cyclic voltammography was used to gage the presence and extent of the reaction species at different poentials with the aid of the Pourbaix diagram. EIS was employed to model the surface impeadence properties that control the current and ion kinetics. SEM and EDXA were used to show the elemental surface, and presence of diffusion pores. The AFM showed that surface roughness occures without the addition of AgSO4, while treated samples remained smooth. X-Ray diffraction identified AG2S as a film component. Optical adsorption and scanning tunneling microscopy measured the Eg of the Ag2S film, and showed that it is a semiconductor. Ion selective electrodes and ICP were used to measure the concentrations with time of Cu (I,II) Fe(II,III) and Ag(I), and to measure the silver adsorption isotherm on the mineral surface, and to qualify how the leaching rate is improved. Other impure chalcopyrite ores with quartz and clay veins were studied.

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Study of EDTA Complexed Electroless Copper Plating for ULSI and Electronics Application: *Inho Chi*[1]; [1]Hong Ik University, Dept. of Metall. Eng. and Matls. Sci., Seoul 121-791 Korea

In copper metallization resistivity of copper seed layer is very important since the thickness of seed layer is less than 200nm. Conventionally MOCVD has been used for this purpose however electroless plated copper is simple process and the resistivity of copper deposit is less than that of copper prepared by MOCVD. In this study electroless depositions of copper were conducted on different substrate to find optimum conditions of electroless copper plating for electronic applications. To find optimum conditions, the effects and selectivity of activation method on several substrates were investigated. The effects of copper salt concentration, reducing agent, complexing agent and inhibitor on deposition rate was investigated. The resistivity of copper with thickness was also measured.

3:40 PM
Utilization of Copper from the Process of Circuit Boards Etching by Copper-Ammonia Solution: *Olga Yu Goriaeva*[1]; Yuri V. Anikin[1]; Vladimir I. Skorohodov[1]; Natalia I. Putina[1]; [1]Ural State Technical University, Metall. Dept., Mira St. 19, Ekaterinburg 620002 Russia

A technology for copper utilization from circuit boards etching is proposed. The technology is based on combinations of electrochemical and sorption methods. The conditions of copper electroextraction from ammonia-chloride solutions to obtain a compact cathode copper are defined. The stage of electrolysis is designed for copper recuperation from etching process. Because the electrochemical processes are not acceptable for the cathode precipitation of the copper from diluted solutions such as rinse waters, the study focused on on the strong-based cationites. After filtration, the sorption columns solution is directed to the stage of circuit boards washing. A close loop for water is possible. The resin, after saturation, is regenerated by chloride ammonium solutions. The resultant rafinate is then directed to the etching stage. Generally, the technology provides copper recuperation and minimizes the amount of waste.

Structural Biomaterials for the 21st Century: Properties of Biocompatible Metallic Materials

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Corrosion and Environmental Effects Committee, Structural Materials Committee, Titanium Committee
Program Organizers: Mitsuo Niinomi, Toyohashi University of Technology, Department of Production System Engineering, Toyohashi 441-8580 Japan; Donald R. Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA; Henry E. Lippard, Allvac R&D, Monroe, NC 28110 USA; Toru Okabe, Baylor College of Dentistry, Texas A&M Health Science Center, Department of Biomaterials Science, Dallas, TX 75246 USA; Eric M. Taleff, University of Texas, Mechanical Enginering Department, Austin, TX 78712-1063 USA

Monday PM Room: 229
February 12, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Mitsuo Ninomi, Toyohashi University of Technology, Dept. of Prod. Sys. Eng., Toyohashi 441-8580 Japan

2:00 PM Keynote
A Study of Dental Casting at Baylor College of Dentistry: *Toru Okabe*[1]; [1]Baylor College of Dentistry, Texas A&M Health

Sci. Cen., Dept. of Biomatls., 3302 Gaston Ave., Dallas, TX 75246 USA

Casting of dental prostheses such as crowns and bridges has traditionally been carried out using gold alloys or base metals such as Ni-Cr and Co-Cr alloys. Due to the favorable properties of titanium, such as its light weight and biocompatibility, this metal has gradually been adapted for use in dentistry. However, more study is needed for titanium to be fully accepted as a viable option to the conventional casting metals. During our five-year study, various experimental binary and ternary titanium alloys have been prepared with elements such as Ag, Co, Cr, Cu, Fe, and Mn and have been cast in magnesia-based investment molds using a high-speed (3000 rpm) centrifugal casting machine. This presentation will give an overview of test results of mechanical properties, castability/mold filling ability, electrochemical behavior, and wear resistance.

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Evaluation of Mechanical Properties and Biocompatibility of Cast Titanium with Silicon Addition: *Jun Zhu*[1]; Akira Kamiya[1]; Takahiko Yamada[1]; Wen Shi[1]; Akira Watazu[1]; Toru Nonami[1]; Katsuyoshi Naganuma[2]; [1]National Industrial Research Institute of Nagoya, Matl. Process. Dept., Kita ku Hirate-cho 1-1, Nagoya, Aichi 462-8510 Japan; [2]National Industrial Research Institute of Nagoya, Matl. Struct. Form. Process. Dept., Kita ku Hirate-cho 1-1, Nagoya, Aichi 462-8510 Japan

The mechanical properties and biocompatibility of Ti-Si and Ti-Si-Ca cast alloys prepared by dental casting machine were investigated. The results show that silicon significantly modified the microstructure of titanium alloys. In addition, Ti-Si and Ti-Si-Ca alloys show good combination of strength and ductility in a wide range of silicon contents in contrast to the pure titanium and Ti-6Al-4V alloy samples which were obtained with the same casting method. A dense and uniform bonelike apatite layer was formed on the surface of substrate made by Ti-Si-Ca and Ti-Si alloys in simulated body fluid. The apatite-forming ability was higher than that of pure titanium and similar to the pure titanium treated with NaOH aqueous solution. It is revealed that Ti-Si and Ti-Si-Ca cast alloys can be expected as promising candidates for dental application because of a good balance between mechanical properties and bioactivity.

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Effect of Microstructure on Fatigue Strength of Dental Ag-Pd-Cu-Au-Zn Alloy in Artificial Saliva: *Toshio Mizumoto*[1]; Mitsuo Niinomi[1]; Hisao Fukui[2]; Jiro Hasegawa[2]; [1]Toyohashi University of Technology, Product. Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580 Japan; [2]Aichi Gakuin University, Dept. of Dental Matls. Sci., 1-100 Kusumoto-cho, Chikusa-ku, Nagoya, Aichi 464-8650 Japan

Ag-Pd-Cu-Au-Zn alloys have been widely used as dental alloys for inlays, clasps, crowns, bar and bridges in Japan. Since restorations made by these type alloys often fractured by mastication for cyclic stress, a lack of reliability for fatigue characteristics of the alloys are pointed out. The heat-treatment conditions showing excellent tensile properties and fracture toughness have been reported recently. Fatigue tests were carried out on an Ag-Pd-Cu-Au-Zn alloy conducted these heat-treatments in artificial saliva. The effect of microstructure and artificial saliva on fatigue characteristics were then discussed. Fatigue strength of aged alloy in artificial saliva is nearly equal to that in air in the low cycle fatigue (LCF) region, while that in the high cycle fatigue (HCF) region tends to the lower than that in air. This decreasing fatigue strength in HCF region may be caused by corrosion of the specimen surface due to the long time exposure to artificial saliva.

3:10 PM

Improvement of Mechanical Performance of Cast Titanium Alloys for Dental Applications by Thermomechanical Processing: *Toshikazu Akahori*[1]; Mitsuo Niinomi[1]; Ryosuke Isohama[2]; Akihiro Suzuki[3]; [1]Toyohashi University of Technology, Dept. of Product. Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580 Japan; [2]Nikken Kosakusyo Works, Ltd., 6-53 1-Chome, Motomati-cho, Higashiosaka, Osaka 579-8005 Japan; [3]Daido Steel Company Ltd., R and D Lab., 2-30 Daido-cho, Minami-ku, Nagoya, Aichi 457-8545 Japan

Thermochemical processing, that is, hydrogenation and dehydrogenation followed by various heat treatments were investigated in order to improve the balance of strength and ductility of castings of Ti-6Al-7Nb and Ti-6Al-4V for dental applications. Microstructures of both cast alloys change from coarse Widmanstätten α structure to super fine α structure with an average α diameter of 3 μm. Mechanical performances of both cast alloys tend to rise remarkably by heat treatments after hydrogenation and dehydrogenation process. The development in mechanical performance in this case is due to increasing plastic deformability in unstable β phases because the lattice constants of both cast alloys conducted with post hydrogenation and dehydrogenation heat treatments are much greater than those of both as-cast alloys.

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Mechanical Performance of Beta-Type Titanium Alloy, Ti-29Nb-13Ta-4. 6Zr, For Biomedical Applications: *Daisuke Kuroda*[1]; Mitsuo Niinomi[1]; Toshikazu Akahori[1]; Hisao Fukui[2]; Akihiro Suzuki[3]; *Jiro Hasegawa*[2]; [1]Toyohashi University of Technology, Product. Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580 Japan; [2]Aichi Gakuin University, Dent. Matls. Sci., 1-100 Kusumoto-cho, Chikusa-ku, Nagoya, Aichi 464-8650 Japan; [3]Daido Steel Co., Ltd., R&D Lab., Minami-ku, Daido-cho, Nagoya, Aichi 457-8545 Japan

Beta-type titanium alloy, Ti-29Nb-13Ta-4. 6Zr, which is composed of non-toxic elements with low modulus of elasticity is newly developed for biomedical applications. The practical size ingot of Ti-29Nb-13Ta-4.6Zr with a weight of about 17kg is fabricated. Forging, cold rolling and heat treatments are conducted with Ti-29Nb-13Ta-4.6Zr in order to achieve greater mechanical performance. Tensile tests and measurements of modulus of elasticity of rolled Ti-29Nb-13Ta-4.6Zr were carried out in order to investigate the basic mechanical properties for biomedical use. Solution treatment after cold rolling and aging gives around 1000 MPa tensile strength and over 15% elongation. The direct aging followed by cold rolling gives greater strength compared with the strength given by aging followed by solutionizing. Additionally, Ti-29Nb-13Ta-4.6Zr has lower modulus of elasticity compared to that of conventional biomedical titanium alloy like Ti-6Al-4V ELI. Ti-29Nb-13Ta-4.6Zr is highly expected to be used for biomedical applications like artificial hip joint and dental implants etc.

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Fretting Fatigue Behaviors of Ti-6Al-4V Eli and Ti-6Al-7Nb for Biomedical Applications in Air and Ringers' Solution: Hisao Fukui[1]; *Wei Yang*[1]; Mitsuo Niinomi[2]; Kei-ichi Fukunaga[2]; Akahori Toshikazu[2]; Jiro Hasegawa[1]; Ikuhiro Inagaki[3]; [1]Aichi Gakuin University, Dental Matls. Sci., 1-100 Kusumoto-cho, Chikusa-ku, Nagoya, Aichi 464-8650 Japan; [2]Toyohashi University of Technology, Dept. of Product. Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580 Japan; [3]Sumitomo Metal Industries, Ltd., Rail. Parts and Forg. Manufact. Dept., 1-109 5-chome Shimaya, Konohana-ku, Osaka, 554-0024 Japan

Present work focuses on fretting fatigue behaviors of two titanium alloys for biomedical applications, Ti-6Al-4V ELI and Ti-6Al-7Nb. Fretting fatigue and plain fatigue of the both alloys were conducted in air at room temperature and in Ringers' solution at 36°C simulating human-body environment, respectively, under conditions of frequency, f,=10, stress rate, R,=0. 1, and a constant normal pressure of 30MPa maintained through each foot of fretting pads. The fretting-fatigue limits of the alloys are dramatically lower than their plain-fatigue limits. A special fracture zone, SFZ, in the edge of fracture surfaces of the both alloys is found. SFZ initiates underneath fretted area and its feature is different from other parts of the fracture surface. The distinction of the shape and size of SFZ is surveyed between Ti-6Al-4V ELI and Ti-6Al-7Nb. SFZ is suggested to originate from the divergences between mechanical properties and microstructure of the both alloys.

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Mechanical Properties of Ti-6Al-7Nb and Ti-5Al-13Ta Alloys under Combined (Tensile/Torsional) Stress: *Equo Kobayashi*[1]; Hiroto Mochizuki[2]; Hisashi Doi[1]; Takayuki Yoneyama[1]; Masahisa Otsuka[2]; Hitoshi Hamanaka[1]; [1]Tokyo Medical and Dental University, Instit. of Biomatls. and Bioeng., 2-3-10, Kanda-surugadai, Chiyoda-ku, Tokyo 101-0062 Japan; [2]Shibaura Institute of Technology, Dept. of Matls. Sci. and Eng., 3-9-14, Shibaura, Minato-ku, Tokyo 108-8548 Japan

Mechanical properties of novel biomedical titanium alloys, Ti-6Al-7Nb and Ti-5Al-13Ta alloys, under tensile/torsional combined stress were evaluated. The test was carried out using a combined stress testing machine. The strength of the alloys under combined stress was estimated by applying of von Mises-type yield condition and effective stress. And it is concluded that the data obtained might contribute greatly to biomedical devices design used in the living body, which is considered as a complex stress field.

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Tensile/Torsional Fatigue of Biomedical Titanium Alloys: *Equo Kobayashi*[1]; Hiroto Mochizuki[2]; Hisashi Doi[1]; Takayuki Yoneyama[1]; Masahisa Otsuka[2]; Hitoshi Hamanaka[1]; [1]Tokyo Medical and Dental University, Instit. of Biomatls. and Bioeng., 2-3-10, Kanda-surugadai, Chiyoda-ku, Tokyo 101-0062 Japan; [2]Shibaura Institute of Technology, Dept. of Matls. Sci. and Eng., 3-9-14, Shibaura, Minato-ku, Tokyo 108-8548 Japan

Fatigue tests under tensile/torsional combined stress for biomedical titanium alloys were carried out using a combined stress testing machine. The relation of effective stress, which is introduced by von Mises-type yield condition, and fatigue life was evaluated same method to conventional fatigue test. Fracture surface observation by SEM was conducted to judge the fracture mode.

NOTES