TMS

Become A TMS Member

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

FACILITATING NETWORKING:

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

PRODUCING JOM AND OTHER PUBLICATIONS:

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

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

PROMOTING LIFE-LONG LEARNING:

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

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

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

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

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

TMS THE MINERALS, METALS & MATERIALS SOCIETY

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

184 THORN HILL ROAD
WARRENDALE, PA 15086-7514
USA

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

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

GREAT MEMBER BENEFITS

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

ADMISSION REQUIREMENTS

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

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

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

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

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

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

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

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

MEMBERSHIP APPLICATION

PLEASE TYPE OR PRINT

❒ Mr. ❒ Mrs. ❒ Ms.
❒ Dr. ❒ Professor

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

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

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

BIRTHDATE:

_____/_____/_____

Members automatically receive a monthly print subscription to JOM.

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

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

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

FOR OFFICE USE ONLY
ID
BIRTH
ELECTED
TYPE
CATEGORY
SECTION
APPROVED

NAME: _____ (LAST / FIRST / MIDDLE INITIAL)

TITLE: _____

COMPANY OR ORGANIZATION: _____

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

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

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

_____ (PHONE / FAX / E-MAIL)

_____ (MONTH / DAY / YEAR)

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

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

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

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

OTHER SOCIETY AFFILIATIONS: _____

EDUCATION TO DATE:

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

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

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

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

TO APPLICANT

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

Member's Name _____ Member # _____

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

Signature _____ Date _____

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

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

Credit Card # _____ Expiration Date _____

Cardholders Name _____

Signature _____

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

Advance Registration Form

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

PLEASE CHOOSE ONLY ONE OPTION FOR SENDING FORM

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

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

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

TMS2001 130TH ANNUAL INTERNATIONAL MEETING & EXHIBITION

Advance Registration Deadline: January 22, 2001

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

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

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

☐ Dr. ☐ Prof. ☐ Mr.

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

Address: _____

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

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

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

REGISTRATION FEES:

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

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

PUBLICATION ORDERS:

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

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

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

TUTORIAL LUNCHEON LECTURE TICKETS:

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

SOCIAL FUNCTION TICKETS:

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

PLANT TOUR:

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

2001 MEMBERSHIP DUES—FOR CURRENT TMS MEMBERS ONLY:

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

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

TOTAL FEES PAID: $ _____

PAYMENT ENCLOSED:

☐ Check, Bank Draft, Money Order
Make checks payable to TMS. Payment shall be made in US dollars drawn on a US bank.

☐ Credit Card Expiration Date: _____

Card No.: _____

☐ Visa ☐ MasterCard ☐ Diners Club ☐ American Express

Cardholder Name: _____

Signature: _____

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

Housing Registration Form

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

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

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

TMS 2001 130TH ANNUAL INTERNATIONAL MEETING & EXHIBITION

Arrival Date _____ Departure Date _____

Last Name _____ First Name _____ MI _____

Company _____

Street Address _____

City _____ State/Country _____ Zip/Postal Code _____

Daytime Phone _____ Fax _____

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

Accompanying Person _____

☐ Non-Smoking Room Requested ☐ Special Needs

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

1. _____
2. _____
3. _____

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

Accommodations (check one)

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

Hotels

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

Doubletree Hotel
$169/single
$189/double

Embassy Suites
$179/single
$199/double

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

Hilton Garden Inn
$182/single
$202/double

Holiday Inn Select
$165/single
$165/double

Marriott Hotel
$199.00/single
$199.00/double

Wyndham Canal Place
$195/single
$195/double

Wyndham Riverfront Hotel
$179/single
$199/double

CONFIRMATIONS
Confirmation will be mailed, faxed or e-mailed to you from the TMS Housing Bureau once your reservation has been secured with a deposit. You will not receive a confirmation from your hotel. If you do not receive a confirmation within 2 weeks, please call the Housing Bureau.

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

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

CREDIT CARD:

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

Expiration Date: _____

Card No.: _____

Cardholder Name: _____

Authorized Signature: _____

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

Continuing Education Registration Form

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

PLEASE CHOOSE ONLY ONE OPTION FOR SENDING FORM.

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

TMS2001 130TH ANNUAL INTERNATIONAL MEETING & EXHIBITION

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

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

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

Employer/Affiliation: _____

Address: _____

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

Telephone: _____ Fax: _____

E-Mail Address: _____

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

PAYMENT ENCLOSED:

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

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

☐ Visa ☐ MasterCard ☐ Diners Club ☐ American Express

Cardholder Name: _____

Signature: _____

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

Accompanying Tour Registration Form

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

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

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

TMS2001
130TH ANNUAL INTERNATIONAL MEETING & EXHIBITION

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

Name: _____

Address: _____

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

Phone: _____ Fax: _____

PAYMENT OPTIONS

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

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

Card No.: _____ Expiration Date: _____

Cardholder's Name: (please print) _____

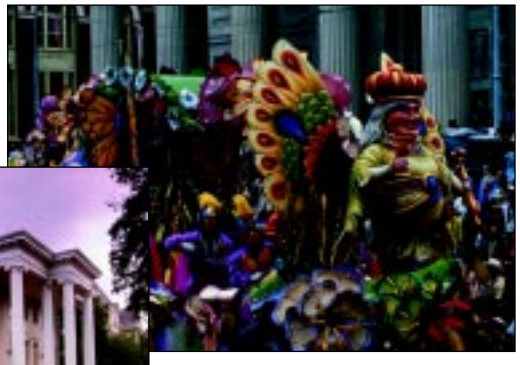
Signature: _____

WE CANNOT ACCEPT PHONE ORDERS

Please make checks payable to and mail to:

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

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



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

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

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

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

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

VISIT THE PUBLICATIONS SALES AREA TO PURCHASE ANNUAL MEETING PROCEEDINGS VOLUMES

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

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

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

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

2001 TMS Annual Meeting & Exhibition

	Monday-February 12		Tuesday-February 13		Wednesday-February 14		Thursday-Feb. 15
	AM	PM	AM	PM	AM	PM	AM
201	Computational Thermodynamics and Materials Design I	Computational Thermodynamics and Materials Design II	Computational Thermodynamics and Materials Design III	Computational Thermodynamics and Materials Design IV	Computational Thermodynamics and Materials Design V	Computational Thermodynamics and Materials Design VI	Computational Thermodynamics and Materials Design VII
202	GA: Mechanical Properties A	GA: Mechanical Properties B	Hume Rothery Award Symposium- Electronic Structure and Alloy Properties I	Hume Rothery Award Symposium- Electronic Structure and Alloy Properties II	Teaching & Learning Solid State Diffusion I	Teaching & Learning Solid State Diffusion II	
203-205	Magnesium Technology 2001 I	Magnesium Technology 2001 II	Magnesium Technology 2001 III	Magnesium Technology 2001 IV	Magnesium Technology 2001 V	Magnesium Technology 2001 VI	Magnesium Technology 2001 VII
206-207	Aluminum Reduction Technology I	Aluminum Reduction Technology II	Aluminum Reduction Technology III	Aluminum Reduction Technology IV	Aluminum Reduction Technology V	Aluminum Reduction Technology VI	Aluminum Reduction Technology VII
208-210	Cast Shop Technology I	Cast Shop Technology II	Cast Shop Technology III	Cast Shop Technology IV	Cast Shop Technology V	Cast Shop Technology VI	Cast Shop Technology VIII
211	Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys I	Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys II	Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys III	Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys IV	Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys VI	Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys VII	Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys VIII
212	GA: Mechanical Properties C	GA: Adhesion	Reactive Metals - General Session I	Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys V	GA: Solidification Processing	GA: Extraction & Processing	
213	GA: Thin Films, Granulation, Aluminum	Lightweight Alloys for Aerospace Applications I	Lightweight Alloys for Aerospace Applications II	Lightweight Alloys for Aerospace Applications III	Lightweight Alloys for Aerospace Applications IV	Lightweight Alloys for Aerospace Applications V	Lightweight Alloys for Aerospace Applications VI
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2001 TMS Annual Meeting & Exhibition

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

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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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: 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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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

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 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

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

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.

4:50 PM

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.

3:10 PM

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.

3:40 PM Break

4:00 PM

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.

4:30 PM

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.

2:55 PM

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.

3:20 PM

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.

3:45 PM Break

3:55 PM

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.

4:20 PM

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.

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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.

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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.

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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.

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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.

5:05 PM

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

2:00 PM

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.

3:15 PM

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

2:00 PM

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

2:00 PM

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.

3:00 PM

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.

3:30 PM

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.

3:00 PM

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.

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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

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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.

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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.

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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.

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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.

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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.

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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.

4:15 PM

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.

4:40 PM

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.

2:25 PM

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.

3:15 PM

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.

3:40 PM Break

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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.

4:40 PM

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.

2:25 PM

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.

2:50 PM

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.

3:40 PM Break

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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.

4:15 PM
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.

3:30 PM Break

3:50 PM Invited

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.

4:20 PM

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.

4:40 PM

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.

5:30 PM

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.

3:20 PM Break

3:40 PM Invited

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.

4:20 PM

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.

4:40 PM

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.

5:00 PM

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.

2:20 PM
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.

3:00 PM Break

3:20 PM
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.

2:30 PM

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.

2:50 PM

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.

3:30 PM Break

3:40 PM

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.

4:00 PM

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.

4:20 PM

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.

4:40 PM

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

2001 HUME-ROTHERY AWARD SYMPOSIUM
"On the Quasi-Particle Spectra of
Superconducting Random Alloys"
8:30 AM - 9:10 AM
Ernest N. Morial Convention Center - Room 202
★★★
2001 EXHIBITION
9:30 AM - 5:30 PM
Ernest N. Morial Convention Center - Hall A
★★★
Complimentary Lunch
12:00 PM - 1:30 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
★★★
TECHNICAL DIVISION LUNCHEON & LECTURE
Extraction & Processing Division Luncheon
12:00 PM - 2:00 PM
Hilton Riverside Hotel - Grand Ballroom A
★★★
Extraction & Processing Division Distinguished Lecture
"Recycling at U.S. Plants Operated
Solely to Recycle Metal-Rich Wastes"
1:30 PM - 3:00 PM
Hilton Riverside Hotel - Grand Ballroom B
★★★
TMS ANNUAL AWARDS & DINNER
Reception and Dinner
6:00 PM - 9:30 PM
Hilton Riverside Hotel - Grand Ballroom B

Alumina & Bauxite: Developments in Handling of Bayer Residue

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

Tuesday AM — Room: 217
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Fred S. Williams, Alcoa World Alumina-Atlantic, Point Comfort Operations, TX USA

8:30 AM
Effect of Particle Characterisitics on the Solids Density of Bayer Muds: *Gerald I.D. Roach*[1]; *Evan Jamieson*[1]; N. Pearson[1]; A. B. Yu[2]; [1]Alcoa World Alumina, Techn. Del. Grp., Cockburn Rd., Kwinana, Western Australia 6167 Australia; [2]University of New South Wales, Sch. of Matls. Sci. and Eng., Sydney, NSW 2052, Australia

The solids density that is achieved in residue areas, washer undeflows or on filters varies considerably with bauxite type. In particular Jamaican mud settles to a much lower g/l solids than those derived from most other bauxites. A fundamental study of the physical properties that affect solids density was undertaken to determine if the differences could be explained. This included particulate modelling based on particle interactions. Various characteristics of muds were measured including particle size, porosity, surface area, mineralogy and absorbtivity. Sizing of muds was complicated by the effect of pH on sizing. Those data, together with the particulate modelling, enabled predictions of solids densities that compared well with those obtained in practice. Such comparisons must be on a volume/volume basis.

9:00 AM
Shear-Induced Flocculation and Break Up of Red Mud Aggregates: *Michel J. Gagnon*[1]; Guy Simard[1]; André Charette[1]; Martin Brassard[1]; Robin Veillette[1]; Guy Péloquin[2]; [1]Université du Québec à Chicoutimi, 555 Blvd. Universite, Chicoutimi, Quebec G7H 2B1 Canada; [2]Alcan International Limitée, Jonquière, Quebec G7S 4K8 Canada

In the Bayer process, flocculants are introduced in the feed well to enhance the aggregation of red mud particles. The design of the feed well and the agitation conditions found in it are important for the formation of the aggregates, and certainly contribute to the efficiency of the gravity settlers. To gain a better understanding of the flocculation process taking place in the feed well, shear-induced flocculation was achieved using concentric rotating cylinders (Couette system). In this article, the effects of agitation and residence time on aggregate settling rate and residual turbidity of a diluted Bayer liquor are presented. Both the flocculation and the break up of aggregates were, to some extent, present during the testing. However, their relative importance depends on the manner the aggregates are produced and introduced into the system.

9:30 AM
A Model for Solids Settling: *Walter M. Bounds*[1]; [1]Kaiser Aluminum/Gramercy Business Unit, P.O. Box 3370, Gramercy, LA USA

A mathematical model has been prepared which includes the concept of hindered settling, in order to describe behavior of discreet solid particles in liquid. The basis for calculation is a column of slurry with given dimensions, initial uniform solids concentration, particle size distribution, and liquid/solid properties. The column is divided into finite elements, with mass flow calculation between elements based on Stokes' law, as well as a provision for determining slurry viscosity as a function of solids concentration. Calculated results include solids concentration and particle size distribution, versus time and versus vertical distance in the column. The boundary between "free-fall" and "compression" zones is computed versus time. In addition, model calibration is discussed, along with methods for extracting settling rate and particle size data for use in designing or rating separation equipment.

10:00 AM Break

10:30 AM
Red Mud Stacking: *Marie J. Bélanger*[1]; [1]Alcan International, Ltd., Raw Mats. Grp., 1955 Mellon Blvd., P.O. Box 1250, Jonquiere, Quebec G7S 4K8 Canada

The red mud slurry "stacking" method used in many Alcan Plants has been developed in the 1980's. The aim of this technique is to use minimum space for the disposal of the residue and to rapidly obtain consolidated material. The consistency of the mud slurry plays a key role in the steepness (angle) of the stacking slope. A small pilot stacking unit was built in order to determine the parameters influencing red mud consistency (expressed as the yield stress). A relationship was established between the mud rheology and the stacking slope observed.

11:00 AM
The Effect of Organics on the Segregation of Red Mud: *Xie Yanli*[1]; [1]Northeastern University, Mat. and Metallu. Div., Shenyang, Liaoning 110006 China

The segregation of red mud is one of the important procedure in alumina production, and organics have great influences on the sediment properties of red mud, but the researches on it is limited. In order to make up it, the effect of some organics, such as yellow humic acid, black humic acid, benanedicarboxylic sodium, phenol, oxalic sodium, formic sodium and acetic sodium on the segregation of red mud were studied in this paper. As a result, we found that organics with high molecular weight made the sediment property and pressure property of red mud become worse, and it's difficult to

be sedimentated. But the effect of organics with low molecular weight on the sediment is insignificant. The method to eliminate the disadvantages of organics is also studied in this paper.

Aluminum Reduction Technology: Perfluorocarbon Gas Emissions

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

Tuesday AM Room: 206-207
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Eric Jay Dolin, USEPA, MC 6202J, Washington, DC 20460 USA

TUESDAY AM

8:30 AM Introductory Remarks

8:35 AM Invited
Development of a TDLAS Based Methodology for Monitoring Perfluorocarbon Production During the Aluminium Smelting Process: *Heather A. Gamble*[1]; Gervase I. Mackay[1]; David R. Karecki[1]; John T. Pisano[1]; Harold I. Schiff[1]; [1]Unisearch Associates, Inc., 96 Bradwick Dr., Concord, Ontario L4K 1K8 Canada

Obtaining a realistic global inventory of greenhouse gas emissions is a desired goal of environmental agencies in Canada, the United States, and worldwide. Unisearch Associates, under contract to the Aluminium Association of Canada, has participated in the development of a methodology designed to monitor total perfluorocarbon emissions from primary aluminium production plants. This methodology involves the use of tunable diode laser absorption spectroscopy (TDLAS) to monitor CF4 and C2F6 emission levels in the ducts leading from the potrooms. Complementary measurements of fugitive emissions from the rooftop vents in the potrooms were made using open path Fourier Transform Infrared Spectroscopy. The measured total emissions can be correlated with routinely monitored plant parameters to facilitate the calculation of annual emission totals for each plant. Technology dependent parameters derived from these measurements can also can be used to estimate annual emission levels from plants which do not routinely measure perfluorocarbon emissions.

8:55 AM Invited
Monitoring of Perfluorocarbon Emission During the Primary Smelting Process by Canadian Producers of Aluminium: Heather A. Gamble[1]; Gervase I. Mackay[1]; David R. Karecki[1]; John T. Pisano[1]; Harold I. Schiff[1]; *Guy G. Bouchard*[2]; Celine Lavallee[3]; Nancy Ouellet[3]; Alton Tabereaux[3]; Alain Moras[3]; Michel Lalonde[3]; Christian Van Houtte[3]; Lucien Laroche[3]; Jerry Marks[3]; [1]Unisearch Associates, Inc., 96 Bradwick Dr., Concord, Ontario L4K 1K8 Canada; [2]Alcan International, Arvida Rsch. and Dev. Ctr., 1955 Blvd. Mellon, C.P. 1250, Jonquiere, Quebec G7S 4K8 Canada; [3]Aluminium Association of Canada, PFC Steering Committee, Canada

Long term control of perfluorocarbon byproducts (CF4 and C2F6), which are potent greenhouse gases, is of great interest to producers of aluminium and to environmental agencies. Unisearch Associates, under contract to the Aluminium Association of Canada, has used their mobile tunable diode laser spectroscopy (TDLAS) laboratory to measure real time perfluorocarbon emissions on site at numerous aluminium plants in Quebec and in British Columbia. The emission factors from plants using the centre work prebake (CWPB) technology were consistently the lowest. This paper will detail results obtained for each type of technology during a 1999-2000 measurement campaign and will discuss the correlation between PFC emissions and relevant process parameters. Environmentally, a significant trend was observed when comparing emission levels measured in 1999 with measurements made previously. All the plants for which data were available either reduced or maintained their perfluorocarbon emissions, with levels up to an order of magnitude lower at some sites.

9:15 AM Invited
Measurements of Perfluorocarbon Emissions from Norwegian Aluminum Smelters: *Halvor Kvande*[1]; Helge Nes[2]; Lars Vik[2]; [1]Hydro Aluminium Metal Products Division, N-0240 Oslo, Norway; [2]Elkem Aluminium Mosjøen, N-8655 Mosjøen, Norway

CF4 gas emissions from all potlines at the seven Norwegian aluminum smelters were analyzed by use of a portable photo-acoustic gas monitor. A total of 8 different types of point fed prebake (PFPB) cells and 6 types of vertical stud Söderberg (VSS) cells were studied. Measurements were made in the gas exhaust duct from the potlines, and for the Söderberg cells also the fugitive gas emissions from the rooftop were measured. The results were found to be in good agreement with the corresponding series of measurements done in Norway in 1992/1993.

9:35 AM Invited
Factors Affecting PFC Emissions from Commercial Aluminum Reduction Cells: *Jerry Y. Marks*[1]; Vikram Bakshi[2]; Alton Tabereaux[3]; Eric J. Dolin[4]; [1]IAI Environmental Consultant, 312 Brockton Dr., Kansas City, MO 64064 USA; [2]ICF Consulting, Washington, DC USA; [3]Alcoa, Inc., Muscle Shoals, AL USA; [4]USEPA, Washington, DC USA

Measurements sponsored by USEPA and the Aluminum Association of the PFC gases tetraflurometh ane and hexafluroethane made at six primary aluminum production facilities in the USA provided data on emissions of these compounds during normal aluminum smelting operations. Also, because the measurements were made using process mass spectrometry, a technique capable of monitoring the rate of emissions with seconds time resolution, increased understanding was gained on how the emissions were occurring. The PFC concentration measurements were combined with smelting process data collected during the measurements to provide new insights into the relationship of the process variables and PFC emissions. Detailed data was obtained at several locations relating overall cell voltage and PFC emission rates. The data allow comparison of emission rates from commercial cells as a function of cell voltage with similar data developed on bench scale experiments. Other process variables studied included an examination of the emission rate as a function of anode effect (AE) duration, a comparison of emission rate differences among different cell technologies and consideration of the effect of differences in AE kill strategy. Variability in emissions among anode effects of similar duration was examined along with the implications of these outlier points.

9:55 AM Invited
Towards Elimination of Anode Effect and PFC Emissions via Current Shunting: Hongmin Zhu[1]; *Donald R. Sadoway*[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. and Eng., 77 Massachusetts Ave., Rm. 8-109, Cambridge, MA 02139-4307 USA

Cyclic voltammetry, stepped-current chronopotentiometry, and stepped-potential chronoamperometry have shown that at potentials exceeding ~3.5 V vs Al/Al3+current drops off precipitously. Furthermore, if the potential on the anode falls below ~3.0 V vs Al/Al3+current is restored. The mechanism at work is speculated to be a highly resistive surface film whose formation is strictly potential dependent. Controlled-potential electrolysis in a laboratory-scale cell shows that at potentials exceeding ~3.5 V vs. Al/Al3+the cell goes on anode effect and PFCs are generated. If cell voltage is stepped down to values below ~3.0 V vs. Al/Al3+the cell immediately returns to normal operation. Electrolysis testing has demonstrated that anode effect and PFC generation can be completely avoided by stepping down cell current in small increments whenever cell voltage reaches a setpoint. The research was sponsored jointly by the Aluminum Association and the U.S. Environmental Protection Agency.

10:15 AM Break

10:25 AM Invited
Anode Effects Survey of the Primary Aluminum Industry: *Bernard P. Leber*[1]; [1]Kaiser Aluminum, 534 E. Trent, Ste. 300, Spokane,

WA 99202 USA

The International Primary Aluminium Institute (IPAI) conducted a survey to collect anode effect related process data and production data. This survey covered the period 1994 to 1997 and supplemented a 1990 to 1993 survey. The survey questionnaire was sent to producers representing about 75% of world primary aluminium production for 1997. Participation in this survey averaged just over 60% of world production. In calculating specific emission rates (kg/te Al) for tetrafluoromethane (CF4) from the process data provided, the Intergovernmental Panel on Climate Change's (IPCC) recommended good practice guideline was followed. Specific emission rates and trends were calculated by production technology type for the period 1990 to 1997. Based only on the participating production volumes, a 47% reduction in the specific emission rate of CF4 occurred between 1990 and 1997. Global emission estimates, taking into account non-participating production have also been made.

10:45 AM Invited

The Aluminum Industry, a Greenhouse Challenge–The Australian Experience: *Lee Eeles*[1]; [1]Australian Greenhouse Office, GP.O. Box 621, Canberra 2601, Australian Capital Territory

As a signatory to the United Nations Framework Convention on Climate Change (FCCC) and the Kyoto Protocol, Australia has accepted a range of obligations in respect of greenhouse gas emissions abatement. 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 over five years to a range of greenhouse gas abatement and energy efficiency programs. Key programs include the development of environmental management strategies for each of the synthetic gases included in the Kyoto Protocol–HFs, FPCs and SF6–and the Greenhouse Challenge. The Greenhouse Challenge is joint initiative between the Australian Government and Australian industry. As participants in Greenhouse Challenge, enterprises are encouraged to take a voluntary and self-regulatory approach to emissions reductions. 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 percent. Future emissions reductions are likely to be in energy supply and use.

11:05 AM Invited

International Efforts to Reduce PFC Emissions from Primary Aluminum Production: *Eric Jay Dolin*[1]; Joe Casola[2]; [1]U.S. Environmental Protection Agency, MC 6202J, 1200 Pennsylvania Ave. NW, Washington, DC 20460 USA; [2]ICF Consulting, Washington, DC USA

Ten countries now have voluntary and/or regulatory programs to reduce PFC emissions from primary aluminum smelters. The nature and success of these programs was detailed in a report issued by the U.S. Environmental Protection Agency in September 1999. This paper will present an updated snapshot of how these country programs are faring and will consider the prospects for additional government action on PFC emissions in the future. The paper will also reflect on the status of international climate change negotiations, specifically how they relate to PFCs and primary aluminum production.

11:25 AM Panel Discussion:
PFC and Aluminum Production: What Now?

Automotive Alloys 2001: Session II

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

Tuesday AM — Room: 214
February 13, 2001 — Location: Ernest N. Morial Convention Center

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

8:30 AM

Quantitative Characterization of Three-Dimensional Damage as a Function of Compressive Strains in an Al Alloy: *Himanshu Agarwal*[1]; Arun Gokhale[1]; Mark F. Horstemeyer[2]; Sam Graham[2]; [1]Georgia Institute of Technology, Sch. of Mats. Sci. and Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA; [2]Sandia National Laboratories, P.O. Box 969, Livermore, CA 94551-0969 USA

Al-Si-Mg base wrought alloys are widely used for automotive and aerospace structural applications, where mechanical properties are of central importance. Aluminum 6061 alloy is one of the alloy of this kind, which is widely used for structural applications. An extensive study on the damage evolution in Al 6061 alloy is performed at various strains under uniaxial compression. Three-dimensional digital image analysis techniques are used to study the damage at different strains. It has been found that the particle fracture is the main mechanism of damage in 6061 Al alloy under compressive loading. It has also been found that the smaller particles with equiaxed and elongated shape and big particles with equiaxed shape are required to avoid the damage of the alloy.

8:55 AM

Mechanical and Physical Property Evaluation of a 359/SiC/20p MMC Prepared by a Novel Rapid Mixing Technique: *Darrell R. Herling*[1]; Mark T. Smith[1]; Warren H. Hunt[2]; David M. Schuster[3]; Mike D. Skibo[3]; [1]Pacific Northwest National Laboratory, Engy. Sci. and Techn. Div., 902 Battelle Blvd., MSIN: P8-35, Richland, WA 99352 USA; [2]Alunimum Consultants Group, Inc., 4530 William Penn Hwy., #3900, Murrysville, PA 15668-2002 USA; [3]Mc-21, Inc., 5100 Convair Dr., Carson City, NV 89706 USA

Aluminum metal matrix composites (MMCs) has found applications in many industries, from aerospace and automotive to sporting goods and electronics packaging. Nevertheless, relatively high materials costs have been the primary limit to widespread use of such a material family. The use of ceramic particulate instead of fibrous reinforcement has help to reduce the over-all material cost, for those applications that do not require the additional strength obtained from a fiber-reinforced composite. However, for many cost sensitive industries, such as the transportation industry, widerspread application of particulate reinforced MMCs is still limited due to materials cost. One source of cost is related to the compositing processes used to make aluminum MMC materials, which stir-casting techniques, such as the one employed by Alcan Aluminum, Ltd. for their Duralcan product, have become the most common. Metal Matrix Composites for the 21st Century (MC-21, Inc.) in Carson City, Nevada has developed a novel rapid mixing process for the preparation of MMC materials. This is a proprietary process, with the focus of rapidly mixing the particulate into the matrix alloy. The process claims to significantly reduce the time required for mixing, and therefore can reduce labor and ultimately material costs. In addition, it is proposed to place such a modular mixing unit at the site of a foundry, producing the composite material as needed and transferring the molten material directly to the casting floor, without the need for remelting of ingot. This would potentially aid in reducing costs. Further cost savings is found in the use of a low cost SiC material for the reinforcement, compared to the standard F-500 used in the industry. This paper presents the results of a study to compare the mechanical and physical properties of this lower cost aluminum MMC, with a standard Duralcan MMC product. Both

populations of materials were a 359 aluminum alloy with 20% by volume SiC particulate reinforcement.

9:20 AM

Precipitation and Aging in Al-Si-Ge-Cu: *David Mitlin*[1]; Velimir Radmilovic[2]; Ulrich Dahmen[2]; J. W. Morris[1]; [1]U.C. Berkeley and LBNL, Matls. Dept., LBNL ms 66-200, 1 Cyclotron Rd., Berkeley, CA 94720 USA; [2]National Center for Electron Microscopy, Lawrence Berkeley Nat. Lab., USA

Al-Cu based alloy 2219 is one of the most popular structural aluminum alloys for use in high temperature environments. For many applications, however, it does not possess sufficient strength (or hardness which is an indicator of strength). Al-xGe-xSi-xCu alloys display a superior peak hardness compared to 2219, while having equal, if not better stability after extended aging at high temperatures. Additionally, Al-xGe-xSi-xCu requires less aging time to achieve maximum hardness than does 2219, making this new class of alloys less expensive to heat treat. The Al-xGe-xSi-xCu system relies on an extremely numerically dense distribution of readily nucleated, ultra-fine Si-Ge precipitates as a template for heterogeneous nucleation of q (metastable Al2Cu) that are also are very fine and densely distributed. Because of their small size the q? are short and thick, making them resistant to shearing during deformation. This is a result of general precipitation theory [Khachaturyan] which states that for a precipitate in a form of a plate, its aspect ratio (length/thickness) varies as the square root of its length. The q? are uniformly distributed and are relatively monodispersed in their size, making them resistant to coarsening at high temperatures. The fast initial hardening response may make this alloy very attractive for applications where the structure will undergo multiple pass welds, since each subsequent weld will harden the heat-affected zone.

9:45 AM

Mechanical Strength anf Thermal Stability in Al-Si-Ge-Cu Alloys: *David Mitlin*[1]; V. Radmilovic[2]; U. Dahmen[2]; John William Morris[1]; [1]Univeristy of California, Mats. Sci. and Eng., Berkeley, CA 94720 USA; [2]Lawrence Berkeley National Laboratory, Nat. Cent. Electron Micros., 1 Cyclotron Rd., Berkeley, CA 94720 USA

This paper concerns the properties of newly developed Al-Si-Ge-X alloys, which have superior peak hardness compared to 2219, while having equal, if not better stability after extended aging at high temperatures. Additionally, Al-xGe-xSi-xCu requires less aging time to achieve maximum hardness than does 2219, making this new class of alloys less expensive to heat treat. The Al-xGe-xSi-xCu system relies on a dense distribution of readily nucleated, ultra-fine Si-Ge precipitates as a template for heterogeneous nucleation of theta' (metastable Al2Cu) that are also fine and densely distributed. The theta' are short and thick, making them resistant to shearing during deformation, and are relatively uniform in size, making them resistant to coarsening at high temperatures. The fast initial hardening response may make this alloy attractive for applications where the structure will undergo multiple pass welds.

10:10 AM Break

10:20 AM

Response of Aluminum and Magnesium Alloys to Mechanical Surface Treatments: *Matthias Hilpert*[1]; Jens Wendt[1]; Lothar Wagner[1]; [1]Technical University of Brandenburg at Cottbus, Phys. Metall. and Matls. Tech., P.O. Box 101344, Cottbus, Brandenburg 03013 Germany

The effect of mechanical surface treatment on the HCF behavior of the automotive light-weight aluminum alloys 6082 Al and 6005 Al as well as magnesium alloys AZ80 and AZ31 was studied. For shot peening and roller-burnishing, the main process parameters Almen intensity and rolling force, respectively were widely measured to optimize fatigue behavior. The process-induced changes in surface layer properties were studied by profilometry, microhardness measurements and X-ray diffraction. The electrolytically polished condition served as a reference. It was found that the fatigue response of the magnesium alloys to shot peening depended strongly on Almen intensity, i.e., pronounced lifetime improvements were observed only in a range of very low Almen intensities. Higher intensities led to marked overpeening effects. In contrast, the aluminium alloys showed no loss in lifetime with increasing Almen intensity. Since both alloys responded with a similar lifetime improvement to increasing rolling forces in roller-burnishing (which generally leads to a smooth surface finish) it is argued that the shot peening-induced high surface roughness and microcracks are the main reason for the marked sensivity of the magnesium alloys to Almen intensity in shot peening.

10:40 AM

Cut Surface Quality of Trimmed Aluminium Alloy and Steel Automotive Sheet: *Tim Brian Hilditch*[1]; G. L. Kelly[1]; P. D. Hodgson[1]; [1]Deakin University, Eng. and Tech., Pigdons Rd., Waurn Ponds, Victoria 3217 Australia

As the use of aluminium alloys for automotive body panels becomes more widespread, it is increasingly important to understand the shearing behaviour of this material. Shearing defects such as slivers and burrs can cause considerable downtime in a press shop. Research into the shearing behaviour of steel has been on going since early last century, however the shearing of aluminium is still a relatively new field. This study looks at the differences in cut-surface quality of an aluminium alloy, and two different steel grades. Cut-surface quality is measured using rollover depths, burr heights, and observations of fracture surface 'cleanliness'. The aluminium alloy used is 6111-T4, whilst the two steels are a cold-rolled, critical drawing steel, and a hot-rolled, formable steel. The trimming variables analysed in this study include punch-die clearance, tool wear, tool geometry, and cutting angle.

11:00 AM

Natural Ageing Effect on the Bake Hardening Response in Al-Si-Mg Alloys: *Linzhong Zhuang*[1]; Joyce Janse[1]; Peter De Smet[2]; Jianghua Chen[3]; Henny Zandbergen[3]; [1]Corus RD&T, P.O. Box 10.000, CA, IJmuiden NL-1970 The Netherlands; [2]Corus Aluminium Rolled Products, B-2570 Duffel, Belgium; [3]Delft University of Technology, National Centre for HREM, NL-2628 AL Delft, The Netherlands

The heat treatable Al-Si-Mg based 6xxx alloys are becoming increasingly attractive for automotive skin panel applications where high formability and in-service dent resistance are basic requirements. The use of standard 6xxx alloys in the T4 condition results in inferior dent resistance in stamped panels because the strengthening potential of the alloys is not fully utilized due to a low temperature and a short duration in most commercial paint bake cycles. Recent progress in developing T4P (pre-aged) 6xxx alloys has substantially improved the paint bake response (PBR). However, the set-up of processing parameters can have a strong impact on the PBR of the final components. Therefore, in order to maximize the PBR in these alloys, further efforts should be made to optimize the entire processing route. The current work presents the results of laboratory simulation of natural ageing effect on the PBR of the T4P 6xxx alloys. The work focuses on: the natural ageing between the solid solution heat treatment (SSHT) and pre-ageing operation; the natural ageing in the pre-aged material; and their effect on the T4P strength and final strength after paint bake cycle. The differential scanning calorimetry, electrical conductivity measurement, and atomic resolution transmission electron microscopy are used to study the microstructural evolution in materials in different treatment conditions.

11:20 AM

Effect of Tensile Strain Aging on Uniform Elongation and n-Value in 6061 Al Alloy: *Masatoshi Sudo*[1]; Yoshiyuki Hattori[1]; [1]Kanazawa Institute of Technology, 7-1 Ohgigaoka Nonoichi, Ishikawa 921-8501 Japan

1. Introduction: 6061 commercial aluminum alloys are age hardenable alloys, and the sheets are being used as the outer panel for automobile cars. Several researches have been done on the effect of strain on the aging behavior of the alloys, however the strain was mainly given by cold rolling. So, the effect of uniaxial tensile prestrain on the change of tensile properties during aging will be examined. Attention will be especially paid to the behavior of uniform elongation and n-valueC known as controlling factors of stretchability. 2. Experimental Procedures: 6061 Al alloy sheets (1.58mass% Mg2Si, 0.07mass% excess Si, 1.0mm thick) were heat solution treated

at 803K for 40min in an atmospheric annealing furnace and quenched in iced-water. Pre-strain (0% and 5%) was given and tensile tests were carried out at the constant cross head speed of 10mm/min. Finally, they were aged at the temperature of 448K for 0 to 5600min. The change in 0.2% proof stress, tensile strength, fracture stress, 3% flow stress, total elongation, uniform elongation and n-value, and local elongation are measured during isothermal aging at 448K. 3. Results and Discussion: The strength begins to increase rapidly when increasing the aging time from 10 to 32 min, corresponding to the time of serration disappearance. The uniform elongation and n-value, on the contrary, decrease rapidly with the increase in aging time and then reach to a constant value. With increasing the aging time to 10min, however, the uniform elongation value and n-value increase for the stretched specimens and decrease for the unstretched specimens. A slight difference in the aging time beginning the decrease in uniform elongation is also observed between that for the unstretched specimens and that for the stretched specimens. The n value of the unstretched specimens in the as-quenched state is apparently higher by 0.15 than that of the stretched specimens. The relationship between the uniform elongation eu and n-value is described. The value of eu/n is slightly higher than 1 for the stretched specimens. The value is lower than 1 at the beginning of the aging, and becomes nearly 1 at the final stage of the aging for the unstretched specimens. A lot of discussions will be done concerning the method deriving n-value, the difference in the change in uniform elongation and in eu/n value between the unstretched and stretched specimens isothermally aged, and so on.

11:40 AM

Twin Roll Cast 5000 Series Aluminum Sheet for Automotive Applications: *Yucel Birol*[1]; Gokhan Kara[2]; Murat Dundar[2]; Osman Cakir[1]; A. Soner Akkurt[2]; Shaun Hamer[3]; Chris Romanowski[3]; [1]MCTRI, Marmara Rsch. Ctr., P.O. Box 21, Kocaeli, Gebze 41470 Turkey; [2]Assan Aluminum Works, E-5 Karayolu 32 Km., Tuzla, Istanbul 81700 Turkey; [3]FATA Hunter Inc., P.O. Box 5677, 6147 River Crest Dr., Riverside, CA 92507-0745 USA

Twin roll casting is well established as an economical method for producing all types aluminum foil and heat exchanger fin as well as various grades of building and construction sheet. Recently, there has been increasing interest in using twin roll casting as a method to produce low-cost/high-quality 5000 series aluminum sheet for automotive structural applications. Assan Aluminyum conducted a series of tests using a FATA Hunter caster to produce AA5052, AA5754 and AA5182. This paper compares the microstructure, mechanical properties, formability, age softening and corrosion resistance of this twin roll cast material with samples of similar alloys produced by DC casting. In all cases the twin roll cast material is shown to have equivalent, or superior, properties.

Carbon Technology: Anode Properties and Performance

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

Tuesday AM — Room: 215-216
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Markus W. Meier, R&D Carbon, Ltd., Sierre CH-3960 Switzerland

8:30 AM

Influence of Bath-Contamination on Anode Reactivity: *Marianne Aanvik Engvoll*[1]; Morten Sørlie[2]; Harald A. Øye[3]; [1]Norwegian University of Science and Technology, Inst. of Chem., c/o Elkem Res., P.O. Box 8040 Vågsbygd, Kristiansand N-4675 Norway; [2]Elkem ASA Research, P.O. Box 8040 Vågsbygd, Kristiansand N-4675 Norway; [3]Norwegian University of Science and Technology, Inst. of Chem., Trondheim N-7491 Norway

This work is a part of an ongoing effort to attain a fundamental knowledge of how bath-impurities, introduced via the addition of butts, influence the excess consumption of the carbon anodes. To avoid the disturbing and maybe masking effect of other impurities normally present in industrial anode materials, this work uses cokes with defined contamination profiles, made by carbonization of high purity coke precursors in a special pressurized lab-scale coke reactor. The calcined cokes are characterized by means of air and CO_2 reactivity, optical texture, crystalline structure and pore size distribution. NaF, Na_3AlF_6 and CaF_2 catalyze the air and CO_2 gasification reactions. AlF_3 on the other hand, acts as an inhibitor.

8:55 AM

The Effect of Aluminum-Contained Additives on the Reactivity of Pitch Binder in CO2: Yanqing Lai[1]; Jie LI[1]; *Yexiang Liu*[1]; Jianhong Yang[1]; [1]Central South University of Technology, Dept. of Metall. Sci. and Eng., Changsha, Hunan 410083 China

Selective oxidation of carbon anodes in aluminum electrolysis caused by reactivity differences between pitch coke and petroleum coke, is one of the main causes for excess carbon consumption. Impurities and additives in carbon anodes may substantially affect chemical activity of the anodes. In this investigation, coal tar pitch was doped with various aluminum containing additives, such as Al_4C_3, powder Al, $AlF_3.7/3H_2O$ and $AlF_3.7/2H_2O+CaF_2$. The doped pitch was carbonized and baked at 1150C, the powder samples of the same grain size were then subjected to the CO_2 reactivity test. Results showed that all the aluminum containing additives reduced the CO_2 reactivity of pitch coke to different extents. XRD indicated that Al based additives were converted into alumina after baking at 1150C, but CaF_2 remained. Results and the mechanism for the effect of the aluminum containing additives are discussed.

9:20 AM

Use of Coke Air Reactivity Testing for Predicting Anode Air Reactivity: *Jeffrey G. Rolle*[1]; Randy A. Czikall[2]; [1]A. J. Edmond Company, 1530 W. 16th St., Long Beach, CA 90813 USA; [2]Kaiser Aluminum and Chemical Corporation, Prim. Alum. Mats. Lab., 2107 E. Hawthorne Rd., Mead, Washington 99021 USA

In the last two decades there have been numerous studies examining the relationship between coke air reactivity and anode air reactivity. Today, many commercial coke calciners have coke air reactivity as a supply quality specification that must be met as a condition of sale. Our experimental work shows that coke air reactivity does not necessarily correlate with anode air reactivity. Further, it has been shown that it is possible to manufacture anodes with low anode air reactivity using high air reactivity coke. Conversely, it is possible to manufacture anodes with high anode air reactivity using low air reactivity coke. We therefore conclude that the use of coke air reactivity as criteria for selecting a particular coke can be misleading. However, coke air reactivity testing has been shown to be useful for internal quality control to monitor a single coke or a coke blend.

9:45 AM

Impact of Coke Calcination Level and Anode Baking Temperature on Anode Properties: *Bernard Samanos*[1]; Christian Dreyer[1]; [1]Aluminium Pechiney Research Center LRF, BP 114, 73300 Saint Jean de Maurienne, France

The impact on anode properties of the two factors mentioned in the title was tested at the laboratory scale. The main results are as follows: Increasing anode baking temperature decreases anode O_2 and CO_2 reactivities across the tested range below present typical commercial calcination temperatures. A coke calcination level increase has a negative effect on O_2 and CO_2 reactivity. Anode geometrical density increases as a function of coke calcination temperature but this effect vanishes at high anode baking temperature. The anodes with the highest thermal shock resistance are those baked at high temperature and made with cokes calcined at low temperature. These results show that it is interesting to further investigate the use of cokes calcined at a lower level than the typical industry levels. Furthermore, the industrial results agree well with the trends found in the laboratory.

10:10 AM Break

10:20 AM
Coke Blending at Anglesey Aluminum: *Les Edwards*[1]; John Wilson[2]; M. Franz Vogt[1]; [1]CII Carbon L.L.C., 1615 E. Judge Perez Dr. 4th Fl., P.O. Box 1306, Chalmette, LA 70044 USA; [2]Anglesey Aluminium Metal Limited, Penrhos Works, P.O. Box 4, Holyhead, Gwynedd, North Wales LL65 2UJ UK

For commercial and logistical reasons, the Anglesey Aluminium smelter has operated with two quite different coke sources over the last 15 years, switching regularly between high and low sulfur content cokes. Whilst anode performance has been satisfactory over this period, Anglesey initiated a study to investigate the benefits of blending high and low sulfur cokes to reduce overall process variation and improve anode performance. The paper presents the results of a laboratory anode study used to justify expenditure on coke blending equipment and compares the lab results to the improved plant anode results achieved after installation of the blending equipment.

10:45 AM
Impact of Anode Properties and Cover Materials on Cell Operation: Raymond Perruchoud[1]; Werner Karl Fischer[1]; *Wolfgang Schmidt-Hatting*[2]; [1]RDC, P.O. Box 362, Sierre, Valais 3960 Switzerland; [2]IS, Täusistr. 48, Rueti, Zurich 8630 Switzerland

About half of the heat loss from a cell occurs through the top via the anodes, the crust and the alumina cover on the anodes. In the ideal case, the top heat loss maintains the cell in thermal balance and allows an optimum profile of side freeze for stable operation and long cell life. The interpolar distance required to keep the cell in thermal balance should not be too small to introduce magnetic cell instabilities and should not be so high that it significantly increases energy consumption. Any changes in the heat flow through the anodes or the cover insulation will affect the thermal equilibrium of the cell. A quantitative review of the effects of the thermal conductivity of the anodes and of the alumina content in the cover are presented. Remedies and adaptations of the cell parameters for avoiding aluminium production problems and inefficiencies are discussed.

11:10 AM
Relative Contributions to Oxidation Impurity Levels in a Mode Anode Binder Matrix from the Binder and the Aggregate: *Nigel R. Turner*[1]; [1]Koppers UK Limited, Scunthorpe Works, Dawes Lane, Scunthorpe, North Lincolnshire DN15 6UR UK

The study investigates the influence of oxidation catalyst impurity levels in recycled anode carbon and the relatively low levels in anode binders, and their combined effect on the concentrations in the carbonised binder matrix. The results highlight the critical importance of butt-cleaning procedures. Binder pitch was mixed separately with graded aggregate containing several levels of bath impurity then co-carbonised to over 1000ºC in a simplified system that models an industrial anode. The graded aggregate was prepared either from anode butt material or from low impurity, unelectrolysed baked anode. Samples of the carbonised binder matrix were subsequently recovered for elemental analysis. Even after allowance for baking losses, oxidation catalyst impurity levels are much higher in the binder matrix after carbonisation compared with the original binder pitch. Impurity levels in the recovered binder matrix are sensitive to, and dominated by, the concentrations of the "key" catalyst elements in the aggregate.

Cast Shop Technology: Mathematical Modeling of DC 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

Tuesday AM Room: 208-210
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Ho Yu, Alcoa, Inc., Alcoa Tech. Ctr., 100 Tech. Dr., Alcoa Center, PA 15069 USA

8:30 AM
Influence of Surface Morphology on the Boiling Heat Transfer During DC Casting of Commercial Aluminum Alloys: *Dianfeng Li*[1]; *Mary A. Wells*[1]; [1]University of British Columbia, Met. and Mats. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

This study has attempted to clarify the influence of several important factors on the heat extraction during water cooling of industrially DC cast aluminum alloys, including: surface morphology, water flow rate and sample thermal history. The project involved experimental measurements, characterization of the as-cast sample surface morphology using a laser profilometer, and quantification of the sample surface temperature and heat extraction to the cooling water using an inverse heat conduction technique. The results from the study indicate that a variation in alloy surface morphology (machined versus as-cast), water flow rate and sample initial temperature all dramatically influence the calculated boiling curve. In particular, the intensity of the heat extraction was found to be enhanced as the surface of the sample became rougher; nucleation and growth of bubbles became easier.

8:55 AM
Secondary Cooling in DC Casting: Modelling and Experimental Results: *Jan Zuidema*[1]; Laurens Katgerman[1]; Ivo J. Opstelten[2]; Jan M. Rabenberg[2]; [1]Netherlands Institute for Metals Research/ Delft University of Technology, Lab. of Mats. Sci., Rotterdamseweg 137, Delft 2628 AL The Netherlands; [2]Corus RD&T, IJmuiden Technolgy Centre, P.O. Box 10000, IJmuiden 1970 CA The Netherlands

Cooling behavior in most situations can be described by applying a fixed heat transfer coefficient as a boundary condition. In secondary cooling of DC Casting, where a water jet is cooling the surface of a billet or slab, this approach cannot be used. The heat transfer coefficient is varying as function of distance from the impingement point of the water jet, as function of the surface temperature of the billet (slab) and as function of the water temperature. General relations for water-cooling were fitted for secondary cooling using data from a special experimental cooling set-up deviced to simulate secondary cooling in DC casting. These relations were then implemented in the Flow-3D source code, a finite volume based CFD code. The results show good comparison between Flow-3D calculations and experiments.

9:20 AM
Mathematical Modelling of Thermal Stress Evolution during the Start-Up Phase of the DC Casting Process for AA5182: *Joydeep Sengupta*[1]; Daan Maijer[1]; Mary A. Wells[1]; Steve L. Cockcroft[1]; Andre Larouche[2]; [1]University of British Columbia, Met. & Mats. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Alcan International, Ltd., Jonquiere, Quebec G7S 4K8 Canada

From the standpoint of defects and final aluminum ingot quality, the most critical stage of the Direct Chill (DC) casting process is the transient start-up phase. The varying rates of the primary and secondary cooling experienced by ingots during this phase can, under certain circumstances, give rise to unacceptable ingot defects. Math-

ematical finite element models predicting the thermomechanical behaviour of the ingot are emerging as a powerful tool to address this problem. This paper outlines the development of a 3D uncoupled thermal-stress model using the FEM code ABAQUS to predict the evolution of temperature, stress and inelastic strain in the ingot during the critical start-up phase. The geometry of the bottom block is included in the model and its role in the thermal stress evolution is examined. Model predictions of temperature and butt curl are compared to measurements made on an industrial DC casting machine.

9:45 AM

Prediction of Hot Tears in Aluminum DC Cast Billets: *Jean-Marie Drezet*[1]; Michel Rappaz[1]; [1]Ecole Polytechnique Federale de Lausanne, Laboratoire Metallurgie Physique, MX-G, Lausanne CH-1010 Switzerland

The appearance of hot tears, one of the most serious defects which a casting can suffer, represents a major limitation to the production of foundry cast parts and to the productivity of continuous casting processes such as the direct chill casting of aluminium alloys. As an example, the casting speed of the direct chill casting of billets is limited for some aluminium alloys because of their high propensity to develop hot tears which initiate at non zero liquid fraction at the bottom of the sump. In order to predict the occurrence of hot tears in solidifying parts, a hot tearing criterion based on the ability of the interdendritic flow of liquid to compensate for the thermally-induced deformation of the roots of the dendrites has been recently derived by Rappaz, Drezet and Gremaud [1]. Based upon a mass balance performed over the liquid and solid phases, this criterion accounts for the deformation of the solid skeleton and for feeding of the interdendritic liquid: it allows the calculation of the maximum strain rate that the roots of the dendrites can undergo without initiation and/or propagation of hot tears. The present paper gives a summary of the main features and assumptions of the new hot tearing criterion. The equations defining the hot cracking sensitivity index in the particular case of a uniform thermally-induced deformation rate in the mushy zone are presented. The influence of the solidification path of the alloy is studied. Then, the criterion is implemented in a FEM thermo-mechanical model of the DC casting of round billets of aluminum alloys. It is shown that the bottom of the sump is more sensitive to hot tearing than the primary cooling zone and that the casting speed has a large influence on the appearance of hot tears.

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10:20 AM

Application of a New Hot Tearing Analysis in Horizontal Direct Chill Cast Magnesium Alloy AZ91: *John F. Grandfield*[1]; Cameron J. Davidson[2]; John A. Taylor[3]; Arne Dahle[3]; [1]CSIRO Manuf. Sci. & Techn., Albert & Raglan Sts, Preston, Vic. 3072 Australia; [2]CSIRO, Brisbane, Qld Australia; [3]University of Queensland, Dept. of Min., Mins. and Mats. Eng., Brisbane, Qld. Australia

The horizontal direct chill (HDC) casting process is a potential production route for magnesium remelt ingot. The ingot may sometimes display surface and centreline cracking/tearing. In order to control these defects, an analysis of the hot tearing mechanisms based on crack propagation has been developed. The model builds on previous hot tearing models and calculates the pressure contributions acting on a nucleated void due to feeding, dissolved gas and capillary effects to determine if the void will propagate. The effects of columnar and equiaxed structures on tearing are predicted. This model is also applicable to aluminium and other metals.

10:45 AM

Quantification of Spatial Distribution of As-Cast Microstructural Features: *Philippe Jarry*[1]; Stephane Antoine[1]; Mathieu Boehm[1]; [1]Pechiney, Centre de Recherches de Voreppe, 725 rue Aristide Berges-BP 27, Voreppe 38341 France

A novel quantitative assessment of as cast micro-segregation has been developed and used for several years for aluminium based alloys in Pechiney Research Centre. It is based on a mathematical morphology algorithm performed on polished sections with an image analyser. The measurement is related to the spatial distribution of as cast intermetallic phases and is influenced by the average local composition as well as by the primary aluminium phase morphology. Whereas the sdas (secondary dendrite arm spacing) is not always defined, and is only a relevant parameter for continuous diffusion processes, such as homogenisation, governed by solute gradients within the solid solution, the description of the spatial distribution of as-cast intermetallic phases deals with information correlated with those singularities that govern discontinuous phenomena occurring downstream of casting in the transformation schedule, such as recrystallisation or damage build-up under plastic deformation (local or generalised). The adopted measurement method thus provides useful quantitative tools for the metallurgical engineer concerned with the heredity of solidification in final products and the optimisation of casting processes considered as an element of the transformation schedule. The usefulness of the method is illustrated by several examples.

11:10 AM

The Columnar to Equiaxed Transition in Horizontal Direct Chill Cast Magnesium Alloy AZ91: *John F. Grandfield*[1]; Cameron J. Davidson[2]; John A. Taylor[3]; Arne Dahle[3]; [1]CSIRO, Albert & Raglan Sts., Preston, Vic 3072 Australia; [2]CSIRO, Moghill Rd., Pinjarra Hills, Qld Australia; [3]University of Queensland, Dept. of Min., Mins. and Mats. Eng., Brisbane, Qld. Australia

The horizontal direct chill (HDC) cast HDC ingots of AZ91 alloy were cast at two sites. Ingots cast at one site exhibited columnar microstructures and were prone to formation of centreline cracks, whereas ingots cast at the other site had equiaxed microstructures and did not crack. In order to determine whether it was differences in the casting conditions or the melt preparation practices at the two sites that were responsible for the change in the microstructure, available models of the columnar to equiaxed transition (CET) were applied. Ingot microstructures were examined and thermal data was obtained by freezing thermocouples into the ingots during casting. CAFE (cellular automata finite element) modelling using the CalcoMOS® program was also used to predict grain structures. Results showed that while increased casting temperatures can contribute to a slightly increased tendency toward formation of columnar grain structures, a change in the concentration of nuclei concentration is a much more likely cause.

11:35 AM

A Marker Chain Front Tracking Method Adapted for Modelling Meniscus Dynamics in the Direct Chill Al Billet Casting Process: *Fionn Iversen*[1]; Jon Arne Bakken[1]; Stein Tore Johansen[2]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. and Electrochem., A. Getz V. 2B, Trondheim 7491 Norway; [2]SINTEF, Matls. Techn., A. Getz V. 2B, Trondheim 7491 Norway

In conventional direct chill (DC) hot-top casting of aluminium extrusion ingot with gas-slip poor surface quality can be a problem. In the worst cases pronounced surface wrinkling occurs coupled with periodic zones of reduced grainsize, macrosegregation and exudation. These surface characteristics are influenced by the dynamics of the meniscus. The meniscus dynamics are believed to be governed mainly by the gas volume flowrate, surface tension effects, and wetting at the meniscus contact points. A marker chain front tracking technique using cubic spline interpolation for surface reconstruction is applied in the multiphase model. The advantage of the model is its accuracy in the calculation of surface tension forces. Also the wetting can be implemented directly as a boundary condition. Modelling results will be compared with results from experiments and suggestions will be made on how to improve the process to achieve a higher cast ingot surface quality.

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

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

Tuesday AM Room: 222
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Russell H. Jones, Pacific Northwest National Laboratory, Matls. Scis. Dept., Richland, Washington 99352 USA; Richard P. Gangloff, University of Virginia, Mats. Sci. and Eng. Dept., Charlottesville, VA 22904-4745 USA

8:30 AM

Stress Corrosion Cracking of High Purity Iron-Carbon Alloys in Carbonate Solutions: *David J. Duquette*[1]; James A. Might[1]; [1]Rensselaer Polytechnic Institute, Mats. Sci. and Eng., Troy, NY 12180-3590 USA

Stress corrosion cracking (SCC)experiments have been performed on high purity iron-carbon alloys as functions of carbon content and heat treatment in ammonium carbonate solutions. Additionally some experiments were performed on single crystals of iron. The results of these experiments have shown that the critical electrochemical potential ranges that induce cracking are independent of either carbon content or microstructural distribution of cementite. However, in contrast to previously described results, these experiments have shown that the crack path can either be transgranular or intergranular depending on applied potentials and microstructural detail. The single crystal results indicate that active slip systems are preferentially dissolved in the carbonate solutions within the critical potential range. The results of these experiments indicate that, while grain boundary chemistry can be important to SCC of steels, preferential dissolution of active slip bands is an important phenomenon. A model will be proposed to include chemo-mechanical effects in IGSCC.

9:00 AM

Stress Corrosion of Iron Base Alloys in High Temperature Water Environments: *J. Congleton*[1]; E. A. Charles[1]; [1]University of Newcastle, Corros. Res. Cen., Old Forge Bldg., Newcastle upon Tyne, NEI 7RU, UK

Work performed at Newcastle relating to the conditions that induce stress corrosion cracking in iron base alloys when exposed to high temperature water environments will be presented. Such alloys can be made to crack in simulated light water reactor environments, but only if the conditions generate high (anodic) electrode potentials. Cracks often initiate at sulphide inclusions but can also be initiated at slip steps and/or corrosion pits in pure iron. Sulphate contamination of the water enhances cracking for low sulphur content alloys but has little effect if the alloy already contains many sulphide inclusions. Susceptibility to cracking is influenced by water temperature, the electrode potential, the applied strain and the crack tip strain rate. The usefulness of slow strain rate tests under applied potential control for assessing susceptibility to SCC and for studying the mechanism of cracking will be discussed.

9:30 AM

Crack Tip-dislocation Interactions in the Presence of Hydrogen: A. Taha[1]; *Petros Sofronis*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Theor. & Appl. Mech. & Mats. Res., 216 Talbot Lab., 104 S. Wright St., Urbana, IL 61801 USA

The effect of hydrogen on crack-dislocation interaction is studied under conditions where hydrogen is in equilibrium with local stresses. The dislocations are modeled as displacement discontinuities along a slip plane that is inclined at an angle to the crack surface. A variational statement is presented to couple the hydrogen effect with the elastic deformation at a crack tip which undergoes blunting through the emitted dislocations. Finite element analysis based on the Newton iteration method is used to solve the nonlinear boundary value problem and calculate the hydrogen distribution accounting for the stress relaxation associated with the hydrogen induced dilatation and the elastic moduli changes due to hydrogen. Interactions between the dislocations and the crack tip are calculated accounting for all the stress fields due to dislocations and hydrogen atmospheres. The plastic zone size and the crack tip opening displacement for different slip plane angles are estimated, and the results are compared with corresponding predictions from continuum plasticity calculations and experimental observations. In the absence of hydrogen, the crack opening displacement and plastic zone size results are in good agreement with the computations of McMeeking and Shih, as well as with experimental measurements.

10:00 AM

Theoretical Consideration on the Effetcs of Loading Modes on Environmentally Assisted Cracking: *Tetsuo Shoji*[1]; [1]Tohoku University, Fracture Rsch. Instit., 01 Aoba Aramaki Aoba-ku, Sendai, Miyagi 980-8579 Japan

Based upon a proposed theoretical formulation of environmentally assisted crack growth rate driven by slip dissolution (oxidation) mechanism, the effects of loading modes on growth rate are analysed where the crack growth rates under the conditions of 1)rising load, 2)constant load or constant stress intensity factor and 3)constant displacement(load decreasing type loading with crack advance). Numerical solution markedly delineated the effects of dK/dt on crack growth rate and also showed up effects of crack tip solution chemistry on growth rates. A strong interaction of dK/dt and da/dt is also demonstrated by numerical analysis in terms of the da/dt-K diagram. Some considerations on how to use the laboratory data for prediction in field incidences will be discussed from a difference in loading mode between a laboratory and field condition.

10:30 AM

On the Finite Element Modeling of Crack-Tip Fields in Stress Corrosion Cracking: *John E. Dolbow*[1]; Tomasz Hueckel[1]; [1]Duke University, Civil and Environ. Eng. Dept., P.O. Box 90287, Durham, NC 27708-0287 USA

Much attention has recently focused on specific electrochemical and mechanical crack-tip processes and their influence on stress corrosion cracking (SCC). A number of competing postulates have emerged to describe the hydrogen embrittlement of material ahead of the crack tip. While phenomenological models of various scenarios have proven useful, the majority have been one-dimensional and invoked several assumptions about crack-tip displacement and stress fields. This work represents a step towards developing a detailed description of the inherently coupled electrochemical and mechanical crack-tip fields. Specifically, a two-dimensional finite element model has been developed for quasi-static crack growth under the influence of external loads and local hydrogen diffusion. The importance of geometry is demonstrated by examining the coupling between mixed loading modes, hydrogen diffusion, and the cohesive law governing fracture. This work applies recent advances in finite element and coupled material modeling to describe the SCC process.

11:00 AM

Corrosion and Corrosion Fatigue in Perspective: *Robert P. Wei*[1]; [1]Lehigh University, Dept. Mech. Eng. and Mech., 327 Sinclair Lab., 7 Asa Dr., Bethlehem, PA 18015 USA

Corrosion and corrosion fatigue, or aging, of aluminum alloys has been shown to be dominated by localized corrosion in the beginning and by corrosion fatigue crack growth in the later stage. In recognition of Roger Staehle's interests and contributions, these processes are considered from the scientific and technological perspectives. In this paper, current understanding of the aging of airframe aluminum alloys is summarized. A possible resolution of the long-standing dichotomy between the nucleation and crack growth (and dissolution and hydrogen embrittlement) approaches to corrosion fatigue is discussed. The use of a simplified probabilistic model that integrates the individual processes is presented. Its application to the assessment of damage evolution and distribution and correlation

with long-term service data are discussed. The paper provides an overall perspective for a multidisciplinary approach for understanding and for integrating information for structural integrity and reliability analyses.*Research supported in part by AFOSR under Grant F49620-98-0198.

Computational Thermodynamics and Materials Design: Thermodynamic Modeling III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee, Jt. Computational Mats. Sci. & Eng., 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, France; Alan Dinsdale, National Physical Laboratory, United Kingdom; Mats Hillert, Royal Institute of Technology, Department of Materials Science and Engineering, Stockholm S-10044 Sweden; Gerhard Inden, Max-Planck Institute-Duesseldorf, Germany; Taiji Nishizawa, Tohoku University, Japan; Greg Olson, Northwestern University, USA; Gary Shiflet, University of Virginia, USA; John Vitek, Oak Ridge National Laboratory, USA

Tuesday AM Room: 201
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Mats Hillert, Royal Institute of Technology, Dept. of Mats. Sci. and Eng., Stockholm S-10044 Sweden

8:30 AM

Thermodynamic Assessments: Science or Art: *Bo Sundman*[1]; [1]KTH, MSE, Stockholm SE 100 44 Sweden

Thermodynamic assessments of experimental data is a skill mastered by few. The path from an initial collection of experimental data to a final set of models and parameters is not easy to describe. The author will review a number of cases and try to elucidate some common features.

9:00 AM

Thermodynamic Assessment of the Ternary System Al-Cr-Ni: *N. Dupin*[1]; I. Ansara[1]; B. Sundman[2]; [1]Institut National Polytechnique de Grenoble, Grenoble, France; [2]Royal Institute of Technology, Mats. Sci. and Eng., Stockholm 10044 Sweden

The good mechanical properties of the Ni-base superalloys are mainly due to the coherency between their constituting phases gamma (Al) and gamma prime (L1 sub 2). These phases have a crystallographic relation which is accounted for by using a single Gibbs energy function to describe their thermodynamic behaviour following previous studies [88Ans, 95Dup, 97Ansj]. For Ni base superalloys, the ternary system Al-Cr-Ni is an important sub-system. In this study, a new assessment based on Dupin's thesis work [95Dup] is presented taking into account new experimental liquidus temperature. A new modelling of the B2 phase is also introduced. It is considered as an ordered phase of the bcc (A2) phase. The existence of vacancies as defects in this structure is considered. The different models used is presented mainly focusing on the ordered phases L1 sub 2 and B2. The parameters describing the Gibbs energy of all the phases are given. Comparison of derived calculated behaviour with experimental data is presented.

9:30 AM

Thermodynamic Assessment of the La-Sr-O System: *A. Nicholas Grundy*[1]; Bengt Hallstedt[1]; Ludwig J. Gauckler[1]; [1]ETH Zurich, Dept. of Mats., Inst. of Nonme. Matls., Swiss Federal Institute of Technology, CH-8092 Zurich, Switzerland

We have started a new project to model the thermodynamical properties of the quaternary La-Sr-Me-O system (Me=Mn, Co, Fe, Ni) using the CALPHAD method. Particular emphasis will be laid on the La1-xSrxMeO3 perovskites, that are used as cathode material for solid oxide fuel cells (SOFC). Before this quaternary system can be addressed the lower order systems, i. e. La-O, Mn-O, Sr-O, La-Sr-O, Mn-Sr-O and La-Mn-O first have to be assessed to provide a consistent set of parameters for the quaternary LSM-system. The detailed assessment of the LSM-system will provide a sound platform, from which other more applied questions can be evaluated such as modelling of the reactions between La1-xSrxMeO3 Cathode and the Y2O3-stabilized ZrO2 based electrolyte or the defect chemistry and transport properties of La1-xSrxMeO3. All three binary systems have been previously modelled. Here we present the quasi binary La2O3-SrO system. As this system is of limited practical use, very little work has been previously done on it, also there exist conflicting reports as to which intermediate phases exist. Therefore an extensive experimental investigation was carried out on the system using DTA/TG, XRD and SEM equipped with EDS and WDS. The experimental results were used together with assessed literature data to model the thermodynamic properties of the system using the PARROT module ofthe Thermo-Calc software package.

9:50 AM

Thermodynamic Assessment and Experimental Study of the Zn-Fe-Ni System: *Nai-Yong Tang*[1]; Xuping Su[2]; Jim M. Toguri[3]; [1]Cominco, Ltd., Canada, Prod. Techn. Cen., Mississauga, Ontario, Canada; [2]Xiangtan University, Scl. of Mech. Eng., Hunan; [3]University of Toronto, Dept. of Metall. and Mats. Sci., Ontario, Canada

In galvanizing, a small amount of an alloying element is commonly added to molten Zn to control coating structure and properties. In general galvanizing, Ni, less than 0.1 wt%, is frequently added to baths to control the reactivity problem caused by the existence of residual Si in the steel while Al, frequently less than 0.2 wt%, is always added to a bath in continuous galvanizing. Since a production bath is always saturated with Fe due to the dissolution of the steel being galvanized, a Zn bath is, in fact, a ternary alloy. To understand the effects of minor additions to Zn baths on galvanizing processes and coating properties, a large-scale research program is currently being carried out at Cominco, Ltd., Product Technology Centre. This presentation reports the outcomes of thermodynamic assessments of the Zn-Fe and Zn-Ni systems. In these assessments, the homogeneity ranges of the solution compounds, such as the Gamma, Gamma 1 and delta phases in the Zn-Fe system and the beta, beta 1 and delta phases in the Zn-Ni system, were successfully modeled using sub-lattice models, and the solubility of Fe in molten Zn was accurately reproduced. Based on the assessments of the Zn-Fe and Zn-Ni systems and using the information on the Fe-Ni system available in the open literature, a preliminary assessment of the Zn-Fe-Ni system was carried out. To complement the phase diagram development, the Zn-rich corner of the Zn-Fe-Ni system was experimentally determined. It was found that, when the addition of Ni to the Zn bath saturated with Fe exceeded 0.055 wt%, the equilibrium intermetallic compound in the molten alloy changed from the zeta phase (Zn subscript 13 Fe with a small amount of Ni in solution) to a ternary compound of the Gamma 1 phase. This phase was believed by many to be an extension of the binary Gamma 1 phase in the Zn-Fe system. Work carried out in this study indicated clearly that Gamma 1 is a truly ternary compound.

10:10 AM Break

10:20 AM

Calculated Phase Diagrams of Aluminum Alloys and Their Utility in Microsegregation Studies: *X.-Y. Yan*[1]; Y. A. Chang[1]; F.-Y. Xie[1]; S.-L. Chen[2]; F. Zhang[2]; S. Daniel[2]; [1]School of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., 1509 University Ave., Madison, WI 53796 USA; [2]CompuTherm, LLC, 437 S. Yellowstone Dr., Suite 217, Madison, WI 53719 USA

One of the authors (Y.A. Chang) has been associating with Larry Kaufman ever since completing his doctoral degree. The authors take this happy occasion on Dr. Kaufman's 70th birthday to honor his pioneering work in the use of computer to calculate phase diagrams and his many contributions in our field. In this presentation, we will focus on (1) the importance of accurate phase boundary values in the study of microsegregation during the course of solidification of an alloy and (2) the advancements made in the calculation of complex phase diagrams. We will use aluminum alloys as ex-

amples of our discussion from binary Al-Cu to multicomponent commercial alloys. While the former may be considered as of academic interest, the importance of a good description of an important binary is a prerequisite in generating reliable descriptions of higher order systems.

10:50 AM

Thermodynamic Optimization of Aluminum Base Metallic Glass Systems: *R. E. Hackenberg*[1]; C. Gao[1]; G. J. Shiflet[1]; L. Kaufman[2]; [1]University of Virginia, Dept. of Mats. Sci. and Eng., 116 Engineer's Way, P.O. Box 400745, Charlottesville, VA 22904-4745 USA; [2]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., 77 Massachusetts Ave., Cambridge, MA 02139 USA

A thermodynamic and phase equilibria study was made of several systems known to form aluminum-based metallic glasses containing transition metal and rare earth elements. In particular, the Al-Fe-Gd and Al-Ni-Gd ternary systems were studied in detail, as was the quaternary Al-Fe-Ni-Gd system. Several alloys in each system were synthesized by melt spinning, each composition containing a minimum of 70 at% Al. X-ray and electron diffraction were employed to determine the solid state phase equilibria, while DTA was used to characterize the solidus and liquidus temperatures. Using these results combined with other published data, these systems were thermodynamically optimized in their Al-rich corners using the CALPHAD approach. The parameters describing the Gibbs free energy of each phase were optimized using Redlich-Kister polynomials. The relevance of this present work in furthering the understanding of metallic glass formation and its subsequent microstructural evolution upon heating will be discussed.

11:20 AM

Thermodynamic Modelling of Oxide Solid Solutions and Melt Processing of Bi-2212 Superconductors: *Bengt Hallstedt*[1]; Ludwig J. Gauckler[1]; [1]ETH Zurich, Dept. of Mats., Instit. of Nonmetall. Matls., Swiss Federal Instit. of Tech., Zurich CH-8092 Switzerland

Bi2Sr2CaCu2Oz (Bi-2212) and Bi2Sr2Ca2Cu3Oz (Bi-2223) are two of the most promising compounds of the ceramic superconductors for bulk applications. In order to produce dense single phase material it has turned out to be necessary to partially melt the material during processing. The resulting microstructure and superconducting properties are sensitively dependent on the processing conditions. Due to the complex phase relations around these phases very large efforts have been necessary to understand the basic reaction sequences during melt processing and, thus, to be able to control the final properties. As a substrate during processing Ag is usually used. Ag dissolves in the liquid phase but not in the solid phases. Thermodynamic modelling offers valuable help in understanding the reaction sequences during melt processing and the influence of various processing conditions on the resulting microstructure. We have modelled the Ag-Bi-Sr-Ca-Cu-O system using Calphad technique. In the present work we will concentrate on three topics: We will discuss thermodynamic modelling of oxide solid solution phases using the compound energy formalism. This will be illustrated by the modelling of the spinel phase in the MgO-Al2O3 system and the Bi2Sr2CaCu2O8+d phase. Several subsystems of the Ag-Bi-Sr-Ca-Cu-O system were experimentally completely unknown. We will use the Ag-Bi-O system as an example to show how the CALPHAD technique can be combined with experimental work to efficiently treat unknown systems. Using thermodynamic calculation and comparisons with experimental work we will discuss melt processing of Bi-2212 on Ag substrates.

11:40 AM

Phase Equilibria in the Fe-Co Binary System: *Ikuo Ohnuma*[1]; Hirotoshi Enoki[1]; Ryosuke Kainuma[1]; Hiroshi Ohtani[2]; Kiyohito Ishida[1]; [1]Tohoku University, Dept. of Mats. Sci., Aoba-yama 02, Sendai 980-8579 Japan; [2]Tohoku University, Center for Interdis. Res., Aoba-yama, Sendai 980-8578 Japan

BCC (α)/FCC (γ) phase equilibrium of the Co-rich portion in the Fe-Co binary system between 400 and 800°C have been determined from the lattice parameter measurements using thin film specimens. Bulk specimens were also used to compare the degree of attaining equilibrium. Thin film technique was found to have a great advantage to study phase equilibria at lower temperatures where solid state reactions are too slow to reach equilibrium structures to form in the conventional methods. It was confirmed that the width of the α+γ two-phase region has been extended below the A2/B2 order disorder transition temperature. Thermodynamic calculation of the Fe-Co binary system was also carried out. The ordering contribution due to B2 phase was described in the split two-sublattice compound energy description with the pair probability approximation of the short range ordering contribution. The extended α+γ two-phase equilibria at low temperatures can be explained as the effect of the B2 ordering contribution in accordance with the calculated results.

Cyanide: Social, Industrial, and Economic Aspects: Analysis and Control

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

Tuesday AM Room: 225
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Kevin Gering, Bechtel BWXT, Advisory Engineer, Idaho Falls, ID USA; Karen Tempel, Newmont, Lone Tree Mine, Valmy, NV 89438 USA

8:30 AM Invited

How to Analyze for Cyanide: *Emil B. Milosavljevic*[1]; Ljiljana Solujic[2]; [1]BioQuest, 4750 Longley Lane #202, Reno, NV 89502 USA; [2]Consultant, 4959 Talbot Lane #252, Reno, NV 89509 USA

Problems associated with distillation and other classical methods for analyzing operationally defined cyanide (CATC, WAD, Total and Free Cyanide) will be discussed. These methods often achieve incomplete, species dependent, cyanide recoveries and suffer from serious interferences. Depending on the relative concentrations of interferents and other matrix constituents, underestimation (ultimately Type II errors–probability of not detecting a constituent when it is actually present) or overestimation (ultimately Type I errors-probability of deciding a constituent is present when it is actually absent) of the cyanide levels present in a given sample may occur, causing in the first case false security and in the second case, excessive cyanide treatment costs. Many, if not all, enumerated problems can be solved by using novel flow injection (FI) gas-diffusion amperometric methods. A modification of the recently approved FI ligand exchange method for determination of available cyanide (USEPA Method OIA-1677) that obviates sulfide interference will be discussed. Nine laboratory round-robin data obtained for the FI method that determines total cyanide (Method OIA-1678) will also be presented. In addition, some methods for cyanide speciation will be presented. Finally, research needed for developing reliable methods for determination of operationally defined cyanide in solid samples will be discussed.

8:55 AM Invited

Cyanide Polluted Soils: A Combined Leachate and Solid Characterization: *Dorothée Proffit*[1]; Philippe Marion[1]; Marie-Claude Rouillier[1]; [1]Laboratoire Environnement et Minéralurgie, ENSG-INPL, 15, Avenue du Charmois, BP 40, Vandoeuvre les Nancy 54001 France

Characterization of solids containing cyanides is carried out according to recommendations and standard guidelines. Most of these procedures are based on the analysis of leachates collected either on site or after leaching in the laboratory of sampled polluted soils. Unfortunately, these sole results are often insufficient to identify the source of the pollution. To provide a more complete character-

ization of the polluted soils, optical and electron microscopy, XRD, Mössbauer spectroscopy, specular reflection and transmission infrared spectroscopies were combined to analyze leachates and raw and ground samples of polluted soils. The combining of such techniques allows to see what kind of cyanoferrate complexes are present at the solid state, different from Prussian blue as they do not have the same extraction behavior. The use of all these techniques to characterize solid samples combined to the usual analysis on solutions gives a better understanding of the behavior of cyanide.

9:15 AM Invited

Detector for Real-time Measurement of Aqueous Cyanide: *Kevin L. Gering*[1]; Jeffrey J. Rosentreter[2]; [1]Bechtel BWXT, INEEL, Idaho Falls, ID USA; [2]Idaho State University, Pocatello, ID USA

A detection method and apparatus have been developed to perform real-time measurement of aqueous free cyanide, CN. A laboratory prototype can measure cyanide ranging from 5 micrograms per liter to 100 milligrams per liter, where the time required for analysis is as short as 2 minutes per measurement. Sample size per measurement is typically less than a few ml, thus minimizing the waste volume produced from spent samples. The basis for this detection device is a quantifiable, stable affinity between the electrode material and free cyanide in the sample. This technology has experienced two generations of laboratory prototypes, and has been thoroughly tested to determine the operational sensitivity toward process variables such as pH, competing reactions, etc. The speed and accuracy of this method make it superior to conventional wet chemistry methods used for quantifying cyanide levels in water. A device based on this technology could be useful in areas involving process monitoring and environmental compliance. Interest for this detector has been shown by the gold miningindustry and others. Patent application is in progress.

9:45 AM Break

10:00 AM Invited

A Novel Method for the On-Line Analysis of Active Cyanide During the Cyanidation of Gold: *Michael J. Nicol*[1]; Kathryn Hindmarsh[1]; [1]Murdoch University, Min. Sci., South St., Murdoch, Perth, Western Australia 6150, Australia

The most common method for measuring cyanide concentrations in cyanidation leach circuits is based on the titration of cyanide ions with silver ions. This can either be done manually as a titration with a visual end-point or by using one of several commercial cyanide analysers which are based on the same titration but use a potentiometric end point. This method has its limitations in the case of some ores such as those containing high amounts of soluble copper and, more importantly, in terms of reflecting the concentration of cyanide which is available for the dissolution of gold. This paper will present the results of the development of a novel "active" cyanide analyser which is based on the reaction between gold colloids and cyanide. Gold colloids are a deep red colour (absorbance maximum at 520 nm) and can be very simply prepared and stored for extended periods of time when stabilized. Colloidal gold behaves similarly to metallic gold dissolving in sodium cyanide solutions to give the colourless Au(CN)2- ion. This allows the reaction between the colloids and cyanide to be monitored spectrophotometrically at 520 nm. An automated instrument has been developed around this concept which can analyse a filtered pulp stream every few minutes with periodic calibration using standard cyanide solutions. Results of testwork on synthetic solutions and solutions derived from several Australian gold plants will be presented as will the results of a comparative evaluation with a commercial titration analyser on a local gold plant.

10:30 AM Invited

Automated Cyanide Control at Newmont's Lone Tree Mine using Degussa's Cyplus® CCS Cyanide Control System: *Karen Tempel*[1]; Roy Norcross[2]; [1]Newmont, Lone Tree Mine, P.O. Box 388, Valmy, NV 89438 USA; [2]Degussa-Hüls, 4 Pearl Court, Allendale, NJ 07401 USA

Newmont's Lone Tree Mine is located 30 miles east of Winnemucca in Humboldt County, Nevada. Starting in February 1994 a pressure oxidation circuit was commissioned to treat refractory gold ores. Gold is leached and recovered from the oxidized slurry in a six-stage carbon-in-leach (CIL) circuit. Lone Tree has been able to maintain tighter control of cyanide addition and reduce cyanide consumption in its CIL circuit through the installation of Degussa's Cyplus® CCS Cyanide Control System in the spring of 2000. The Cyplus® CCS, along with additional inputs from plant sensors, has allowed for a more efficient and effective control strategy over the manual control method used in the past. This paper documents the key aspects of the Cyplus® CCS installation, the development of the control strategy, and the benefits achieved from enhanced control.

10:55 AM Invited

Cyanide Control in the Metallurgical Process of Gold Extraction in AngloGold (S.A.): B. J. Vorster[1]; S. R. Flatman[2]; AngloGold SA Metallurgical Technical Services; [2]AngloGold SA Ergo CIL Plant

AngloGold South Africa region currently consists of 12 Gold Plants. These plants use a combined total of $20 million of cyanide per annum. Of this the major portion (60%) is consumed at the two Ergo dump retreatment plants. Historically the primary motivation for cyanide control at Ergo has been one of leach/cost optimisation. However more recently with the increased public awareness of cyanide in the environment, a secondary but increasingly important motivation for control has been to ensure that only the minimum amount of cyanide is added to the process whilst not compromising on leach performance. Following a brief overview of the Ergo process the methodology in determining the amount of cyanide to be added is described. The paper then traces the developments in cyanide control from very basic manual systems to the current automated control system. In line with the increasingly sophisticated control systems, developments also took place as regards the method of cyanide analysis. The development of these analysers is discussed. Whilst cyanide is one of the major drivers if not the major driver of gold dissolution it cannot be viewed in isolation particularly in respect of the relationship between cyanide and oxygen derived from the well known Elsners equation. Consequently in order to control cyanide addition knowledge of the relative cyanide/oxygen profiles is necessary. Various means investigated at Ergo for pulp oxygenation have therefore also been included for the sake of completeness. Finally a comparison is made of the control achieved from the current system compared to the original manual system. The knowledge and experience gained at Ergo is now being used to draw up guidelines for the installation and optimisation work at the other AngloGold plants. The net effect of this will be a reduced cyanide consumption for the region, which also apart from the obvious economic benefit translates into a reduced environmental risk.

Defect Properties and Mechanical Behavior of H.C.P. Metals and Alloys: Dislocations, Twinning, and Deformation Behavior

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

Tuesday AM — Room: 211
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Michael I. Baskes, Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA; Craig S. Hartley, Air Force Office of Scientific Research, Air Force Research Lab., Arlington, VA 22203-1977 USA

8:30 AM Invited

Atomistic Simulation of Extended Defects in HCP Metals and Alloys: *Vaclav Vitek*[1]; [1]University of Pennsylvania, Dept. of Mats.

Sci. and Eng., 3231 Walnut St., Philadelphia, PA 19104 USA

Although the hcp structure is closed packed, similarly as the fcc structure, materials crystallizing in hcp lattice have much more variable mechanical properties than fcc materials. The latter are usually ductile with well defined slip systems while the former may be brittle or ductile with slip systems different in different materials and twinning may be an important deformation mode. The origin of this variability is hidden in the atomic structure of dislocations, twins, stacking faults etc. which may vary from one hcp metal to another. These properties originate in the atomic and electronic structure of these extended defects and can only be investigated by atomic level simulations. The precursor of such calculations is a description of atomic interactions appropriate for a given material and in this presentations we first discuss various approaches to this problem. These range from pair potentials, through many-body central force potentials of the EAM type to quantum mechanics based bond-order potent ials (BOPs). Dislocations and other extended defects in hcp metals were studied using all these approaches and we discuss both successes and failures of these investigations. Finally, we concentrate on BOPs which were constructed for titanium and we present their application to dislocation studies. Furthermore, BOPs were recently developed for TiAl with $L1_0$ structure and we demonstrate that they are also applicable Ti_3Al with hexagonal DO_{19} structure.

9:00 AM

Atomistic Calculations of the Energies and Structures of Dislocations and Planar Faults in HCP Metals: *James R. Morris*[1]; Man H. Yoo[2]; Kai-Ming Ho[1]; [1]Ames Laboratory, Ames, IA 50011 USA; [2]Oak Ridge National Laboratory, Mets. and Cer. Div., Oak Ridge, TN 37831-6115 USA

We are performing atomic scale calculations to explore the structure and energetics of defects associated with <c+a> deformation in hcp materials, in particular Zr, Ti and Mg. Ab initio calculations have provided new information on twin boundary energies and stacking fault energies, associated with compression twinning and slip on the {11-22 }plane. Edge dislocation core structures associated with slip on this plane, calculated using classical potentials (embedded atom models), show two distinct geometries: a glissile, "type II" geometry, dissociated on the {11-22} plane, and a sessile, "type III" geometry that is dissociated on the basal plane that nucleates a {11-21} tension twin. In this talk, I will review these results, and present more recent work on other dislocation structures for b=<c+a>.

9:20 AM

Effect of Temperature and Shear Direction on Yield Stress by {1122}<1123> Slip in HCP Metals: *Hideki Tonda*[1]; Shinji Ando[1]; [1]Kumamoto University, Mech. Eng. and Matls. Sci., 2-39-1 Kurokami, Kumamoto City 860-8555 Japan

The yield shear stress of the τ_y due to {1122}<1123> second-order pyramidal slip system (SPCS) in cadmium, zinc and magnesium HCP crystals increased with increasing temperature. This result is interpreted by two thermally activated processes; the dissociation of a SPCS <c+a> perfect edge dislocation into <c+a> sessile dislocation and an a glissile basal dislocation, and the immobilization of the <c+a> edge dislocation as a result. Consequently, double cross slip of <c+a> screw dislocations must be activated thermally by an increment of applied stress to increase propagation velocity of slip band width. Moreover, τ_y due to SPCS in zinc and cadmium is affected strongly by a direction of applied shear force. The anomalous behaviors of yielding would be caused by nonsymmetrical core structure of <c+a> dislocation because of lattice heterogeneity in HCP metals.

9:40 AM

Molecular Dynamics Simulation of <c+a> Dislocation Core Structure: *Shinji Ando*[1]; Hideki Tonda[1]; Takushi Goto[2]; [1]Kumamoto University, Dept. of Mech. Eng. & Mats. Sci., Faculty of Eng., 2-39-1 Kurokami, Kumamoto 860-8555 Japan; [2]Kumamoto University, Grad. Sch. of Sci. and Techn., 2-39-1 Kurokami, Kumamoto, 860-8555 Japan

The <c+a> edge dislocation has two types of core at 0K; one is a perfect dislocation (Type-A) and the other is two <c+a>/2 partials (Type-B). Type-A transforms to Type-B by abruptly increasing temperature from 0K to 293K, while Type-B is stable in whole temperatures. In contrast, Type-A extends parallel to (0001) at 30K and this extended core is still stable at 293K. These results suggest that the <c+a> edge dislocation glides on the {1122} as two <c+a>/2 partial dislocations and becomes sessile due to changes of the core structure. The <c+a> screw dislocation spreads over two {1011} at 0K. The core transforms to unsymmetrical structure at 293K, which is spread over {1122} and {1011}, and to a core spread parallel to {1122} at 1000K. The dependence of the yield stress on the shear direction can be explained form these core structures.

10:00 AM Break

10:20 AM Invited

Dislocation Core Structure and Glide Mechanisms in HCP Metals and Alloys: *Alain Couret*[1]; Marc Legros[2]; Daniel Caillard[1]; [1]CEMES-CNRS, 29 Rue J. Marvig, BP 4347, Toulouse, Cedex 4 31 055 France; [2]LMP-CNRS-INPL, Ecole des Mines, Parc de Saurupt, Nancy Cedex, 54 402 France

Titanium and the intermetallic alloy Ti3Al glide primarily in the prismatic plane. The corresponding glide mechanisms have been studied by in situ straining experiments performed inside the transmission electron microscope at various temperatures. In both cases, screw dislocations are submitted to a frictional force which is due to the core spreading out of the glide plane. For the case of titanium, dislocations move by jumps between locking positions. The mechanical properties as the variation of the activation area with the stress are interpreted from this dynamic behaviour. Two different antiphase boundary energies and two different frictional forces have been evidenced for the a dislocations gliding in the prismatic plane of Ti_3Al. They are interpreted as resulting from different cutting planes, in agreement with several theoretical estimates.

10:50 AM

Alloying Effects on Non-Basal Slip Behavior of HCP Metals: *M. H. Yoo*[1]; S. R. Agnew[1]; [1]Oak Ridge National Laboratory, Mets. and Ceram. Div., Oak Ridge, TN 37831-6115 USA

In polycrystalline hcp metals, a critical situation arises where the c-axis of a grain is oriented parallel to the uniaxially applied stress axis. In this so-called "hard orientation," non-basal <c+a> pyramidal slip and/or deformation twinning play important roles in generalized plastic deformation. In this work, effects of alloying elements on the pyramidal slip process are analyzed in light of the recently proposed source mechanism for this non-basal slip mode. In the case of Mg, Li additions lower the elastic stiffness, as would raising the temperature, and Cd increases the elastic anisotropy and enhances the source operation energetically. In the case of Co, Ni additions could assist the source operation kinetically because the increase in (0001) stacking fault energy promotes cross slip of <a> dislocations into the (1-100) prism plane. Other possible effects, e.g., Cu or Ni addition in Be, will be also discussed.

11:10 AM

Plasticity of Mg and HCP Mg-Li Alloys: The Role of <c+a>{11-22} Dislocations: *S. R. Agnew*[1]; J. A. Horton[1]; M. H. Yoo[1]; [1]Oak Ridge National Laboratory, Mets. and Cera. Div., Oak Ridge, TN 37831-6115 USA

The limited number of independent deformation modes is frequently blamed for the typically poor ductility of polycrystalline hcp metals. Increased activity of nonbasal, e.g. <c+a>{11-22}, dislocations could allow for significant improvements in ductility. The activity of the various slip modes has been identified both by deformation texture modeling, as well as by direct observation using a TEM. The main component in plane strain and uniaxial compression textures is "off-basal" for magnesium alloys, and in particular for Mg-Li. Based on texture simulations, this is due to <c+a> slip. Mg-Li a-solid solutions have improved room temperature ductility, as compared to pure magnesium and its common alloys. Increased <c+a> dislocation activity is likely responsible, as opposed to prismatic <a> dislocations cited in earlier works. TEM observation has verified significant <c+a> activity. Furthermore, direct evidence is presented for a collinear dissociation of <c+a> –> η <c+a> + (1-n)

η <c+a> partial dislocations formerly predicted by atomistic simulation.

11:30 AM

Dislocation Processes in Single Colony Alpha/Beta Titanium: *Michael F. Savage*[1]; Joseph Tatalovich[1]; Michael J. Mills[1]; [1]The Ohio State University, Dept. Mats. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Observations have been made of significant anisotropy in the room temperature creep response and constant strain rate behavior in single colonies of a two phase alpha(hcp)/beta(bcc) titanium alloy. Much of the observed anisotropy is attributed to the near-Burgers orientation relationship (OR) formed upon growth of the alpha laths from prior beta grains. The Burgers OR provides for easy slip transmission of one of the hcp a/3<11-20> slip systems through the beta phase, while the other slip systems require significant residual dislocation formation. To understand the deformation mechanisms controlling room temperature plasticity in these alloys, detailed Transmission Electron Microscopy investigations into the dislocation processes active in single alpha/beta colonies of Ti-6Al-2Sn-4Zr-2Mo-0.2Si and single alpha crystals of Ti-6Al will be presented. Mechanisms for slip transmission through the alpha/beta interfaces will be discussed as well as investigations into the structure of a/3<11-20> dislocations on basal and prism planes.

11:50 AM

Determination of Dislocation Densities in HCP Metals from XRD Line-Broadening Analysis: *M. Griffiths*[1]; D. Sage[1]; R. A. Holt[1]; C. N. Tome[2]; [1]Atomic Energy of Canada, Ltd., Chalk River, Ontario, Canada; [2]Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545 USA

The determination of bulk dislocation densities in metals is typically undertaken by analysis of X-ray diffraction (XRD) lines, relating the measured line-broadening to the dislocation density. In the case of HCP Zr-2.5Nb alloy two types of dislocations are present, e.g. those arising from <a>-slip and those arising from <c+a>-slip. Since during irradiation creep and growth the physical effects of different dislocation structures are different, individual determinations of dislocation densities are required. As a consequence, the contributions to XRD line-broadening have to include the resolved contribution to the strain from each type of dislocation. In this work this assessment has been achieved by tensile deformation of a highly textured, recrystallized Zr-2.5Nb alloy with a preponderance of either <a>-type or <c+a>-type dislocations. XRD line-broadening analysis of specimens deformed between 0 and 15% has been performed, and an iterative deconvolution method has been applied to extract the dislocation broadening function using the 0% deformed specimen as a standard for instrumental broadening. The average strains for both prism and basal plane line-broadening due to either <a>-type or <c+a>-type dislocations are calculated for each specimen and compared with the results of Fourier analyses of the broadened diffraction lines. The results are discussed in terms of the accuracy of the calibration factors experimentally determined by TEM and also in terms of the effect of intergranular stress distributions on the accuracy of the line-broadening analyses using single crystals as standards.

Hume Rothery Award Symposium - Electronic Structure and Alloy Properties:Theory

Sponsored by: Electronic, Magnetic & Photonic Materials Division; Structural Materials Division

Program Organizers: Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Tuesday AM — Room: 202
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: J. Sam Faulkner, Florida Atlantic University, FL 33431 USA; Ole Krogh Andersen, Max-Planck-Institut fuer Festkoerperforschung, D-70569 Stuttgart, Germany

8:30 AM Invited

On The Quasi-Paricle Spectra of Superconducting Random Alloys: *Balazs Gyorffy*[1]; [1]University of Bristol, Dept. of Phys., H.H.Wills Phys. Lab., Tyndall Ave. Bristol, UK BS8 1Tl UK

A description of disorder is central to the theory of Superconductivity.Although the pioneering works of Abrikosov and Gorkov(AB)[1] and Anderson[2] explains the principle puzzle of why it does not lead to finit resistance they do not add up to a complete Mean-Field Theory of disordered Superconductors. In this talk I will review recent progress in combining the Hartree-Fock-Gorkov and Coherent Potential Approximations to provide such theory[3]. The emphasis will be on the cases of exotic, d- and p-wave, pairing relavent to recent experiments and the novel consequences of random alloy type of disorder has on the superconducting state in these interesting systems. [1]A.A.Abrikosov and L.P.Gorkov, Sov.Phys. JETP8,1090(1959) [2]P.W.Anderson J.Phys.Chem.Solids 11,26 (1959) [3]A.M.Martin et al Phys.Rev.B60,7523(1999)

9:10 AM Invited

Muffin Tin Orbitals of Arbitrary Order: *Ole Krogh Andersen*[1]; [1]Max-Planck-Institut fuer Festkoerperforschung, Heisenbergstrasse 1, D-70569 Stuttgart, Germany

We have derived orbital basis sets from scattering theory. They are expressed as polynomial approximations to the energy dependence of a set of partial waves, in quantized form. The corresponding matrices, as well as the Hamiltonian and overlap matrices, are specified by the values on the energy mesh of the screened scattering path operator and its first energy derivative. These orbitals are a generalization of the 3rd-generation linear MTOs and should be useful for electronic-structure calculations in general. Examples for cuprate high temperature superconductors are given.

9:30 AM Invited

Ab Initio Theory of the Interlayer Exchange Coupling in Random Metallic Multilayers: *Josef Kudrnovsky*[1]; Vaclav Drchal[1]; Ilja Turek[2]; [1]Institute of Physics AS CR, Na Slovance 2, Prague CZ-182 21 Czech Republic; [2]Institute of Physics of Materials AS CR, Zizkova 22, Brno CZ-616 62 Czech Republic

We present ab initio formulation of the interlayer exchange coupling (IEC) between two, in general non-collinearly aligned magnetic slabs embedded in a non-magnetic spacer whereby both the spacer, the magnetic slabs as well as their interfaces may be random. This approach is based on the spin-polarized surface Green function technique within the tight-binding linear muffin-tin orbital method, the Lloyd formulation of the IEC, and the coherent potential approximation using the vertex-cancellation theorem. The temperature dependence of the IEC will be also discussed. The periods, amplitudes, and phases are studied in terms of discrete Fourier transformations, the asymptotic behavior of the IEC is briefly discussed within the stationary-phase method. The relation of the IEC and the giant magnetoresistance will be also mentioned. Numerical results illustrating the theory are presented.

9:50 AM Invited

Relativistic Calculation of Magnetic Linear Response Functions Using the KKR-Green's Function Method: *Hubert Ebert*[1]; Ming Deng[1]; Harald Freyer[1]; [1]University of Munich, Depts. of Chem. and Phys. Chem., Butenandtstr. 5-13, Munich D-81377 Germany

The use of the KKR (Korringa-Kohn-Rostoker) Green's function method to calculate magnetic linear response functions as e.g. the magnetic susceptibility has been suggested nearly 20 years ago. Also the extension of the formalism to include relativistic effects has been worked out in the past, but first applications could be presented only some few years ago. Several new developments of this very flexible approach will be presented. Among others the VanVleck orbital susceptibility is calculated in a fully relativistic way and the enhancement of the orbital susceptibility is accounted for by Brooks orbital polarization mechanism. Very closely connected to the susceptibility is the Knight shift that essentially probes the induced magnetization at the nuclear site. The magnetic form factor on the other hand represents the induced magnetization in a more global way. Results for these response quantities will be presented for pure transition metals, compounds and disordered alloys.

10:10 AM Invited

The Effects of Magnetic Annealing Transition Metal Alloys Deduced from Ab-Initio Electronic Structure Calculations: *Julie B. Staunton*[1]; S. S.A. Razee[1]; B. Ginatempo[2]; E. Bruno[2]; F. J. Pinski[3]; [1]University of Warwick, Phys. Dept., Coventry CV4 7AL UK; [2]Universita di Messina, Dipartimento di Fisica and Unita INFM, Messina, Italy; [3]University of Cincinnati, Dept. of Phys., Cincinnati, OH 45221-0011 USA

A theory is presented for describing the effects of annealing magnetic alloys in magnetic fields. The approach has an ab-initio spin-polarised relativistic KKR-CPA electronic structure basis and uses the framework of concentration waves. Alloys which would otherwise be soft magnets are found experimentally to develop directional chemical order and significant uniaxial anisotropy when annealed in magnetic fields. Our theory is able to provide a quantitative description of these effects together with the underlying electronic mechanisms. We describe several applications to transition metal systems in detail including NiFe, FeCo and CoPt.

10:30 AM Break

10:50 AM Invited

The Mathematics of the Polymorphous Coherent Potential Approximation: *J. S. Faulkner*[1]; [1]Florida Atlantic University, Dept. of Phys., Boca Raton, FL 33431 USA

The original coherent potential approximation (CPA) for calculating the electronic states in substitutional solid-solution alloys contains the implicit assumption that the alloy is isomorphous. That is, all of the atoms of a given chemical type are assumed to be identical. The extension of the CPA philosophy to treat an alloy model in which all of the atoms are allowed to have distinct charges and potentials is called the polymorphous CPA (PCPA). This extension requires some interesting changes in the mathematical formalism that is used to develop the CPA equations. Aspects of the mathematical formalism of the PCPA will be discussed. In particular, the ergodic theorem from measure theory will be invoked to justify the new equations for the average Green's function.

11:10 AM Invited

Universal Screening Atomic Sphere Net Charges in Metallic Alloys: *Andrei Vladimirovich Ruban*[1]; Hans L. Skriver[1]; [1]Danish Technical University, Phys. Dept., Lyngby DK-2800 Denmark

The locally self-consistent Green's function (LSGF) method is used in supercell calculations to establish the distribution of the net charges in the atomic spheres of the alloy components in metallic alloys with different compositions and degrees of order. This allows one to determine a contribution to the one-electron potential and total energy in the single-site model for the Poisson equation. We show that in the single-site approximation for the electronic structure calculations it gives a consistent and practically exact solution of the electrostatic problem, i.e. the electronic structure, total energy and their conditional averages obtained in the usual single-site CPA-DFT method becomes identical to whose in the super-cell calculations by the single-site LSGF-CPA method. We demonstrate that the basic mechanism which governs the charge distribution is the local screening of the net charges of the alloy components. Such a screening appears to be almost universal in the single-site approximation for the electronic structure part. As a consequence a unique expression for the electrostatic shift of the one-electron potential and the corresponding contribution to the total enengy in the single-site CPA-DFT method is propose d which provides a very accurate description, relative to the single-site LSGF, of the electronic structure and the total energy of metallic random alloys independently of their composition, volume, and crystal structure.

11:30 AM Invited

Physical Properties of Technological Alloys: First-Principles Simulations: *Igor Abrikosov*[1]; [1]Uppsala University, Phys. Dept., Box-530, Uppsala S-75121 Sweden

The physical properties of intermetallic alloys and compounds are attractive for technological applications, for example, in high strength superalloys, magnetic materials, refractory compounds, superconductors, and so on. A deep understanding of their behavior at different temperatures, compressions, and compositions is essential for the design of new materials. Practically all technological materials contain deviations from an ideal three-dimensional periodicity, and the most common type of crystal defects is a substitutional disorder. The purpose of the present paper is to show that in the framework of the first-principles electronic structure theory a deep understanding of the behavior of off-stoichiometric intermetallic alloys can be derived, which allows one to predict a priori the technological properties of real materials. We discuss the recent development of the theoretical tools within the alloy theory for first-principles simulations of properties of intermetallic alloys, their surfaces and interfaces. Applications of theoretical methods for studying properties of industrial materials (Fe-Ni invar alloy, Newsilver (Cu2NiZn), Al-Zn,high-temperature intermetallics, important catalysts, etc.) are presented.

11:50 AM Invited

Ab Initio Angle-Resolved Photoemission in Multiple Scattering Formulation: M. Lüders[1] A. Ernst[1,2]; W. M. Temmerman[1]; Z. Szotek[1]; P. J. Durham[1]; [1]Daresbury Laboratory, Daresbury, Warrington, WA4 4AD, Cheshire, UK; [2]Max Planck Institut für Mikrostrukturphysik, Halle, Germany

The theory of ab initio semi-relativistic angle-resolved photoemission calculations is formulated within the real-space multiple scattering theory and the single-particle approximation. It has the flexibility and simplicity to study systems with layered structures and more general complex geometries. For layered structures the layer-resolved potentials are obtained self-consistently with the Korringa-Kohn-Rostoker (KKR) method. The advantage of the present approach is that both the self-consistent potential and the photocurrent are treated on the same footing and calculated within the same formalism. The approach is illustrated through a study of the angle-resolved photoemission for real space, layered systems with two-dimensional periodicity, with the specific application to Cu.

12:10 PM Invited

Relativistic Theory of Photoemission from Magnetic Surfaces: *Paul Strange*[1]; Matthew Woods[1]; Arthur Ernst[2]; Walter Temmerman[2]; [1]Keele University, Phys. Dept., Sch. of Chem. and Phys., Keele, Staffordshire ST5 5BG UK; [2]Daresbury Laboratory, Theore. and Comput. Phys., Daresbury, Warrington, Cheshire WA4 4AD UK

A fully relativistic theory of photoemission from magnetic surfaces has been developed. It is based on density functional theory and implemented using multiple scattering theory using a real space cluster method. Interpretation of the results yields information about the effect of relativity on the band structure. We illustrate the theory with results for the surfaces Ni(001), Ni(100), Co(001), Fe(001), and Fe(110).

Emerging Technologies for Metals Production I

Sponsored by: Extraction & Processing Division, Light Metals Division, Aluminum Committee, Process Fundamentals Committee, TMS Young Leaders Committee

Program Organizers: Samuel A. Davis, TIMET, Henderson, NV 89009 USA; Toni Marechaux, US Department of Energy, Office of Industrial Technology, Washington, DC 20585-0121 USA; Thomas P. Robinson, US Department of Energy, Office of IndustrialTechnology, Washington, DC 20585-0121 USA

Tuesday AM Room: 221
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Sam Davis, TIMET, Process Eng., Henderson, NV 89009 USA

8:30 AM Opening Remarks

8:45 AM Invited

A Comparison of Some of the New Titanium Metal Technologies with DuPont R&D and Commercial Results Through the Early 1960's: *James W. Reeves*[1]; [1]3 R Associates, 8 Wollaston Rd, Wilmington, DE 19810 USA

DuPont was the titanium metal pioneer who put the greatest effort into sponge and powder R&D during the period 1945-1963. Commercial results were use of ilmenite for $TiCl_4$ production, an improved Kroll sponge process, an improved Hunter powder process and a direct powder metallurgical process. DuPont pursued powder metallurgy because of the long term potential and because they were not a fully integrated producer using sponge. This effort failed because the powder chloride content could not be lowered to the desired level even though the fabrication techniques were very successful. This remained a trade secret until published by E. H. Mahla in NMAB-392 in 1983. Other processes were explored, but the currently promising electrolysis route was not because DuPont is a producer of merchant sodium and $TiCl_4$.

9:15 AM

Economics and Production of Primary Titanium by Electrolytic Winning: *Marco V. Ginatta*[1]; [1]GTT, C. M. D'Azeglio 21, Torino, Italy

Current world production, 60,000 ton/y, is exceedingly too small for titanium's extraordinary combination of favorable properties; it should be 1,000,000 ton/y (7% of stainless-steel).Prices that competitively sustain that sales volume are achievable only with electrolytic production, as it is for all other commercial nonferrous metals. But titanium does not have its commercial electrolytic plants yet, because of producers decisions and strategies, scientists works, industrial problems with chloride process, lack of consumers sponsors. Fluoride high temperature process has the advantages of aluminum electrolysis, plus other favorable characteristics specific to titanium and its feed material. One electrolytic titanium potroom replaces several different plants used for sponge production. Production of titanium ingots with zero defects is achieved. The solidified cathode rectangular slabs are suitable for direct rolling.

9:45 AM

Low Cost Titanium-Myth or Reality: *Paul C. Turner*[1]; Alan D. Hartman[1]; Steven J. Gerdemann[1]; Jeffrey S. Hansen[1]; [1]US Department of Energy, Albany Research Center, 1450 Queen Ave., SW, Albany, OR 97321-2198 USA

Titanium has been proven an excellent material for the aerospace industry; however, its cost has prevented its outstanding properties from being utilized in non-aerospace applications, including the automotive and heavy vehicle industries. Over the past few years, a number of new, innovative processes have claimed the potential to significantly reduce titanium production costs. This manuscript will review a number of these processes. The Department of Energy's Albany Research Center (ARC) located in Albany, Oregon, has been involved in the reduction and processing of titanium and its alloys for over 50 years. Over the past 10 years, ARC scientists have focused on the development of processes to reduce the cost of titanium for non-aerospace applications both through in-house research and cooperative efforts with others in the field. As such, ARC is uniquely suited to review these emerging, innovative technologies.

10:15 AM Break

10:35 AM

Plasma Quench Production of Titanium Powder: *Alan D. Donaldson*[1]; [1]Idaho Titanium Technologies, Inc., 101 Technology Dr., Idaho Falls, ID 83401 USA

Plasma Quench reduction of titanium tetrachloride is the direct heating of the tetrachloride in an arc plasma to 5000 K, where it dissociates to titanium and chlorine atoms, and cooling the reaction by expansion through a Delaval nozzle. Expansion through a nozzle accelerates the gas to supersonic speed converting the thermal energy to kinetic energy. Cooling rates can be as high as 10^7 Kelvins per second. Cooling this rapidly prevents back reactions between the chlorine and titanium. The titanium condenses into a nano-powder; the chlorine combines with injected hydrogen to form HCl gas. We have produced titanium powder at 20 kg/h. At large enough scale energy requirements will be less than Kroll, and costs will be much lower. The main sources of cost reduction are the elimination of magnesium and greatly reduced capital requirements. Fabrication technology development is needed.

10:55 AM

A Process for Continuous Titanium Production from Titanium Oxide: *Katsutoshi Ono*[1]; Ryosuke O. Suzuki[1]; [1]Kyoto University, Dept. Engy. Sci. and Techn., Yoshida-Honmachi, Sakyo-ku, Kyoto 606-8501 Japan

A new refining process for titanium is proposed. The characteristic feature is a continuous operation and a direct reduction from TiO_2 using Ca. The molten salt electrolysis supplies a two-phase mixture of $Ca+CaCl_2$, and this is mixed and reacted with TiO_2. The reduced Ti particles with average size of 0.5 mm and the reduction product CaO form the slurry with $CaCl_2$ and they can be continuously taken out of the reduction reactor, because the $CaO+CaCl_2$ mixture melts partially above 1200K. The extracted product is leached by water under Cl_2 gas blowing (a product of $CaCl_2$ electrolysis). Ti powders are compacted for VAR melting, and the aqueous solution is condensed to $CaCl_2$-$2H_2O$, anhydrated to $CaCl_2$, and charged for the electrolysis. The oxygen level in Ti powder varied from 200 to 3000 ppm depending on the reducing conditions.

11:15 AM

Selective Extraction of Titanium from Titaniferrous Minerals via a Novel Fluoride Route: Matthew D. Stephens[1]; *Nancy F. Levoy*[1]; William T. Nachtrab[1]; [1]Starmet Corporation, R&D, 2229 Main St., Concord, MA 01742 USA

A new one-step process for extracting titanium from titaniferrous materials has been demonstrated. In the process, depleted uranium tetrafluoride (DUF_4), a solid, is mixed with ilmenite ($FeTiO_3$) and heated to 900°C. Titanium is extracted in the vapor phase as titanium tetrafluoride (TiF_4) and the DUF_4 is converted to uranium-iron oxide. The process has been found to selectively fluorinate the titanium in ilmenite and is capable of producing high purity TiF_4 with very low iron content. TiF_4 can be collected by condensation or through reaction with a fluoride salt such as NH_4F, KF, or NaF. The TiF_4 produced from the process can be used to make titanium metal, titanium oxide, or fluorotitanate compounds. Since the fluorination process is highly selective for titanium, it avoids many of the separations and waste issues common to other titanium extraction processes.

11:35 AM

Reduction of Titania and Ilmenite by Methane Containing Gas: *Oleg Ostrovski*[1]; Guangqing Zhang[1]; [1]The University of New South Wales, Sch. of Mats. Sci. and Eng., Sydney, NSW 2052 Australia

Reduction of titania and ilmenite ores by CH_4-H_2-Ar gas mixture was investigated in a laboratory fixed bed reactor. At temperatures 1200-1500°C, titania was reduced to titanium oxycarbide. At 1400-1500°C, the extent of titania reduction achieved more than 85% in

90 min, equivalent to about 70 wt% of TiC in the TiO-TiC solid solution. Optimum conditions for titania reduction include temperature 1300-1450°C, methane content 8vol% and hydrogen content above 35vol%. Ilmenite ore was reduced to metallic iron and titanium oxycarbide. Metallic iron catalyzed methane cracking and solid carbon deposition. Optimum temperature and methane content for ilmenite reduction are 1200°C and 8vol%, respectively. Increasing hydrogen content enhanced both rate and extent of reduction. At 1200°C, reduction of ilmenite by gas containing 5vol% CH_4, 75vol% H_2 and 20vol% Ar was completed in 60min. Reduction of titanium oxides to oxycarbide followed by chlorination may be an efficient alternative technology for processing of titanium minerals.

High Temperature Coatings - IV: Thermal Barrier Coatings

Sponsored by: Materials Processing and Manufacturing Division, ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee, Surface Engineering Committee
Program Organizers: Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Janet Hampikian, GA Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Tuesday AM — Room: 219
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: James A. Nesbitt, NASA Glenn Research Center, Cleveland, OH 44135 USA; Janet M. Hampikian, Georgia Institute of Technology, Mats. Sci. and Eng., Atlanta, GA USA

8:30 AM Keynote
Accelerated Durability Testing of Coatings for Gas Turbines: M. J. Stiger[1]; F. S. Pettit[1]; G. H. Meier[1]; R. Handoko[2]; J. L. Beuth[2]; [1]University of Pittsburgh, Mats. Sci. Dept., Pittsburgh, PA 15261 USA; [2]Carnegie Mellon University, Dept. of Mech. Eng., Pittsburgh, PA USA

Oxidation resistant and thermal barrier coatings for components in the hot sections of gas turbine engines are desired to have lifetimes on the order of tens of thousands of hours. This presents a problem in evaluating new coatings and modifications to existing coatings because tests, which completely replicate operating conditions, could take years to complete. Therefore, a reliable accelerated testing protocol is needed. In this paper efforts directed toward developing a mechanism-based protocol for evaluating the lifetimes of oxidation resistant coatings under thermal cyclic and hot corrosion conditions and thermal barrier coatings under thermal cyclic conditions will be described. The cyclic lifetimes of oxidation resistant and thermal barrier coatings are determined by spalling behavior. Spallation is a function of oxide thickness and stress level, which control the elastic energy available to drive spallation, and the structures and morphologies of the various layers and interfaces in a given system, which control the fracture toughness at possible planes of weakness. Efforts to evaluate these quantities in relatively short duration tests will be described. Specific techniques include acoustic emission studies, indentation techniques, and detailed metallographic observations. The extrapolation of results from high temperature tests, where failure can be achieved in relatively short times, to lower temperatures, which are characteristic of service conditions, will also be described. The hot corrosion lives of high temperature coatings depend on a variety of factors including temperature, deposit composition, deposition rate, and gas composition. An approach to control these variables in a manner to produce accelerated failures under conditions, which allow estimation of lifetimes under typical operating conditions, will be described and preliminary results will be presented.

9:00 AM
Synthesis of alpha-Al2O3 Template on Ni Superalloy Surface by CVD: *Woo Young Lee*[1]; Y. F. Su[1]; [1]Stevens Institute of Technology, Dept. of Chem., Biochem., and Mats. Eng., Burchard Bldg. 308, Hoboken, NJ 07030 USA

Prior research suggests that the cyclic oxidation life of thermal barrier coatings can be improved by placing a thin layer of alpha-Al2O3 at the metal-ceramic interface region. However, it is known that alpha-Al2O3 is not an easy material to prepare as a thin-film, particularly for complex substrate materials like Ni-based alloys. Also, our knowledge, as how such an alpha-Al2O3 layer can alter the oxidation mechanism of the Ni alloy surface, is fundamentally lacking to guide further exploration of the Al2O3 interlayer concept for practical TBC development. In this work, the morphology and phase nature of CVD-Al2O3 coatings deposited on the surface of a single crystal Ni alloy were examined with and without a Pt interlayer. The extent of morphological tailoring, that is possible via control of deposition variables and alloy surface modifications, will be discussed along with the effects of morphology on subsequent oxidation behavior.

9:20 AM
Oxidation Behavior of EB-PVD TBC Systems with CVD (Ni,Pt)Al Bond Coatings: *J. Allen Haynes*[1]; Michael J. Lance[1]; Bruce A. Pint[1]; Karren L. More[1]; Ian G. Wright[1]; [1]Oak Ridge National Laboratory, Mets. & Ceram. Div., P.O. Box 2008, Oak Ridge, TN 37831-6063 USA

The specific degradation mechanisms of thermal barrier coating (TBC) systems continue to be the subject of intense study and debate. Failure of TBCs deposited by electron beam-physical vapor deposition (EB-PVD) is closely associated with the oxidation and deformation behavior of the metal-ceramic interface. The present study investigated oxide formation within commercial EB-PVD TBC systems with chemical vapor deposition (CVD) platinum aluminide bond coatings and single-crystal superalloy substrates with varying sulfur and reactive element contents. The effects of bond coat grit-blasting, substrate sulfur and substrate reactive element content on oxide phases, oxide stress and TBC thermal cycle life will be described. The impact of bond coat grit-blasting on premature TBC failure will be addressed. Finally, recent observations that provide further insight into the influences of Pt and S on bond coat oxidation behavior will be discussed. Research sponsored by the U.S. Department of Energy, Advanced Turbine Systems Program under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

9:40 AM
Interfacial Microstructure for As-Deposited and Cycled-to-Failure Thermal Barrier Coatings: *Altaf H. Carim*[1]; Tabbetha A. Dobbins[1]; Merrilea J. Mayo[1]; Lucille A. Giannuzzi[2]; [1]The Pennsylvania State University, Dept. of Mats. Sci. and Eng., 118 Steidle Bldg., University Park, PA 16802 USA; [2]University of Central Florida, Dept. of Mech., Mats., and Aeros. Eng., OTC 305, Orlando, FL 32816 USA

Interfacial morphology and reaction products have been investigated in thermal barrier coating systems consisting of yttria-stabilized zirconia (YSZ) deposited onto NiCrAlY bond coat layers. Both as-deposited materials and samples thermally cycled to failure were examined. Scanning electron microscopy and transmission electron microscopy (TEM) were utilized, including energy-dispersive x-ray spectroscopy (EDS) and diffraction analysis; TEM specimens were fabricated using a focused ion beam (FIB) lift-out technique. In particular, the development of oxides of the bond coat constituents at the interface was explored. In failed samples, interfacial protrusions several microns in size contain grains of elemental Ni intermixed with Ni(Al,Cr)2O4 spinel, (Al,Cr)2O3, and other oxides. Grain size and microstructure in the YSZ vary substantially with deposition conditions.

10:00 AM Break

10:15 AM Invited
Advanced Thermal Barrier Coating Systems-Research and Development Trends: *Christoph Leyens*[1]; Uwe Schulz[1]; Klaus Fritscher[1]; Manfred Peters[1]; Wolfgang A. Kaysser[1]; [1]DLR-German

Aerospace Center, Inst. of Matls. Res., Linder Hoehe, Cologne D-51147 Germany

Thermal barrier coatings (TBCs), typically comprised of a ceramic coating deposited onto a bond coated superalloy substrate, are currently used for lifetime improvement of highly loaded turbine blades and vanes in aeroengines and land-based gas turbines by reducing the average metal temperature and mitigating the effect of hot spots. However, the increasing demands placed on the high-tempeature capabilities of turbine components require so called 'designed-in' TBC solutions, i.e. the TBC system is integral part of the component and vital for its safe operation. 'Prime-reliant' coatings are necessary which performance has to go beyond that of state-of-the-art TBC systems. Based on the extensive body of research available from practical applications and laboratory tests, the present paper highlights research and development trends devoted to future generation TBC systems, predominantly fabricated by electron beam physical vapor deposition, with required significant performance improvements and reliability. The overview includes consideration of both the bond coat and the ceramic coating. Compatibility with the substrate and improved oxide scale spallation resistance at higher temperatures than currently employed are important issues for bond coat development, including environmental resistance (oxidation and hot corrosion) and mechanical aspects such as coefficient of thermal expansion and creep behavior. For the ceramic coating, improved sinter resistance, phase stability and lower thermal conductivity are major areas of interest. Since material properties are closely linked to processing conditions, the paper addresses important relationships where appropriate. Examples are g iven of the significance of single layer property interactions with regard to overall coating system performance. Finally, the paper briefly addresses recent advances in non-destructive evaluation techniques and life-prediction methodologies.

10:40 AM

Effect of Hf Additions to Pt Aluminide Bond Coats on EB-PVD TBC Life: *James A. Nesbitt*[1]; Ben Nagaraj[2]; Jeffrey Williams[2]; [1]NASA Glenn Research Center, MS 106-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [2]General Electric Engine Company, One Neumann Way, Cincinnati, OH 45215 USA

Small Hf additions were incorporated into the Pt aluminide coatings during chemical vapor deposition (CVD) of single crystal Rene N5 substrates. Standard yttria-stabilized zirconia top coats were subsequently deposited onto the coated substrates by electron beam-physical vapor deposition (EB-PVD). The coated substrates were then furnace cycle tested at 2125F (1-hr cycles) until spallation of the thermal barrier coating (TBC). The Hf content in the bond coat clearly had a significant effect on the TBC life. Overdoping with Hf reduced the TBC life to less than half that of the baseline which contained no Hf. Smaller Hf additions resulted in TBC lives of 2-3 times that of the baseline. Scanning electron microscopy of the spalled surfaces indicated that small Hf additions increased the adherence of the thermally grown alumina to the Pt aluminide bond coat.

11:00 AM Invited

Thermal Cycling Induced Damage Initiation In Thermal Barrier Coatings: *Vladimir K. Tolpygo*[1]; *David R. Clarke*[1]; [1]University of California, Mats. Dept., Santa Barbara, CA 93106-5050 USA

A variety of damage mechanisms has been identified during the course of a systematic study of the effects of thermal cycling on the life and spallation behavior of electron beam deposited thermal barrier coatings. These range from interface separation between the TBC and the bond-coat, associated with "rumpling" of the bond-coat surface, to the development of sub-surface cavitation in the bond-coat, to inhomogeneous "rumpling" of the TBC itself. The propensity of the individual damage mechanisms depends on both the temperature and the thermal cycling profiles used. Some of these mechanisms are correlated with changes in the stress in the thermally grown oxide with thermal cycling whereas others are independent. This unexpected variety of damage mechanisms further demonstrates that although final failure is generally by buckling and subsequent spallation of the TBC, the underlying mechanisms can be far more complex.

11:25 AM

Modeling Thermal Stresses and Measuring Thin Film Cte in MoSi2 and MoSi2+Sic Composite Coatings on Mo: Earl C. Hixson[1]; *C. Suryanarayana*[1]; Graham G. W. Mustoe[1]; John J. Moore[1]; [1]Colorado School of Mines, Dept. of Metall. and Mats. Eng., Adv. Coat. and Surf. Eng. Lab. (ACSEL), Golden, CO 80401-1887 USA

Non-linear stress analysis utilizing finite elements has been employed to study the thermally induced stresses in the MoSi2 and MoSi2+SiC coating systems. These analyses considered four different coating architectures: a sharp interface between MoSi2 and Mo, a sharp interface between MoSi2+50wt% SiC composite and Mo, and two architectures identical to the above, but including a diffusion barrier layer (DBL) between the Mo and the coating. These models examine the effect of varying the DBL thickness and coefficient of thermal expansion (CTE). The results show that the largest axial thermal stresses are in the coating or the DBL. The DBL CTE strongly influences the stress in the DBL, but only weakly affects the stress in the Mo or the coating. Changing the CTE from 4.0x10-6/°C to 10.0x10-6/°C in a 50 nm DBL changed the DBL stress from 1.8 GPa tension to 2.6 GPa compression. By comparison, the stress in the composite coating changes from only 12 MPa compression to 12 MPa tension. The stress in the DBL decre ases with increasing DBL thickness. The CTE of the DBL was measured using a Netsch dilatometer. The paper will outline the experimental procedure used to measure the CTE of free-standing thin films.

International Symposium on Deformation and Microstructure in Intermetallics: Deformation and Fracture

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

Tuesday AM	Room: 220
February 13, 2001	Location: Ernest N. Morial Convention Center

Session Chairs: C. T. Liu, Oak Ridge National Laboratory; Patrick Veyssiere, LEM, CNRS-ONERA, France

8:30 AM Invited

Operative Slip Systems and Anomalous Strengthening in Ni3Nb Single Crystals with the D0a Structure: *Yukichi Umakoshi*[1]; Kouji Hagihara[1]; Takayoshi Nakano[1]; [1]Osaka University, Dept. of Matls. Sci. and Eng., Grad. Sch. of Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan

Temperature and orientation dependence of operative slip systems and yield stress in Ni3Nb single crystals were examined in tension and compression. Four slip systems of (010)[100], (010)[001], (001)[100] and {201}<10 -2>, and three twinning systems of {211}<-107 13>, {011}<0-11> and {012}<0-21> were operative depending on the crystal orientation and temperature. Deformation substructures were observed by TEM. The CRSS for (010)[100] and (001)[100] slips increased with increasing temperature and reached a maximum around 1100K. The anomalous strengthening for the (010)[100] slip is due to the Kear-Wilsdorf locking based on the cross slip of [100] screw dislocations from (010) onto (100) plane. Strong hardening just after yielding was observed. The hardening is due to exhaustion of the Frank-Read source. The anomalous strengthening for the (001)[100] slip is also discussed. Effects of V and Rh additon on the plastic deformation behavior in Ni3Nb-based single crystals will be also presented.

9:00 AM Invited

Comparing Deformation Mechanisms of NiAl and Ni3Al by Stress Relaxation: *Yong Qian Sun*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mats. Sci. and Eng., 1304 West Green St., Urbana, IL 61801 USA

The plastic deformation characteristics of NiAl and Ni3Al are different in many aspects. A most remarkable difference is in their yield strength: the yield strength of Ni3Al increases rapidly with temperature, while for NiAl the yield strength decreases with temperature. In this work, stress relaxation experiments coupled with in situ electrical resistance measurement have been conducted to reveal how the dislocation density evolves with plastic strain in these two antithetical intermetallics. The changes in the electrical resistivity is used to probe changes in the dislocation content of the single crystal specimens. The results show that in NiAl dislocation density increases with the relaxation plastic strain, whereas in Ni3Al the dislocation density decreases with the relaxation plastic strain. This suggests clearly the importance of dislocation annihilation in the plastic deformation process of Ni3Al. The effects of dislocation density evolution in the deformation process are discussed in relation to the general mechanical properties of these two intermetallics, including the temperature dependence of the yield strength and the work-hardening rate.

9:30 AM

Stability and Cross-Slip in [101] Superdislocations in Gamma TiAl: *Zhijie Jiao*[1]; *S. H. Whang*[1]; Z. Wang[1]; [1]Polytechnic University, Dept. of Mech. Eng., 333 Jay St., Six Metrotech Center, Brooklyn, NY 11238 USA

The cross-slip behavior in Gamma TiAl is complex in that many cross-slip planes for both ordinary and superdislocations are available and each cross-slip may be operative at a certain temperature range depending on the activated state. The cross-slip of <101] type superdislocations occurs at relatively moderate temperatures, accompanied by moderate anomalous hardening while (010) type cross-slip by the dislocations becomes dominant at high temperatures where the hardening rises exponentially with temperature. To describe the anomalous hardening for the entire temperature range, an analytical expression was developed to take into account both cross-slip mechanisms. In addition, the stability of the superpartial dislocations was examined in each cross-slip plane to understand cross-slip behavior and dislocation glide behavior. An attempt will be made to develop a coherent picture of cross-slip activities, and to explain both macro- & microscopic results.

9:50 AM

Exploratory Study into the Effects of an Electric Field and of Electropulsing on the Plastic Deformation of TiAl: Di Yang[1]; *Hans Conrad*[1]; [1]North Carolina State University, Dept. of Matls. Sci. and Eng., P.O. Box 7907, Raleigh, NC 27695-7907 USA

The effects of a concurrent external electric field (2kV/cm) and of electric current pulses($2x10^4$ A/cm^2), 60μs duration and 20 pulses per second) on the stress-strain curves in compression of cast TiAl specimens were determined at 600°C. The electric field produced a 25-47% reduction in the yield stress, which was followed by an increase in the subsequent strain hardening. However, the flow stress at a strain of 10% with the field was still significantly below that without. In contrast, electropulsing increased both the yield stress and strain hardening so that the flow stress at ε=10-20% was higher than without electropulsing. The effects of the electric field and electropulsing on the flow stress occurred during the early stages of plastic deformation(ε<1%), but were retained upon subsequent straining even when the field or current pulsing was shut off. This suggests that the effects of the electric field and electric current on the flow stress may result from changes in the stacking fault energy and/or antiphase boundary energy in the γ-TiAl lamellae, thereby influencing deformation twinning. The present results suggest that the formability of TiAl may be improved by the application of an electric field and that the creep resistance may be enhanced by electropulsing.

10:10 Break

10:25 AM Invited

Fracture and Fatigue of Refractory Metal Intermetallic Composites: *John J. Lewandowski*[1]; Deneesh Padhi[1]; Sergey Solv'yev[1]; [1]Case Western Reserve University, Dept. Matls. Sci. and Eng. Cleveland, OH 44106 USA

The fracture and fatigue behavior of refractory metal intermetallic composites are being determined under a variety of test conditions. Composites based on the binary Nb-Si system as well as multi-component systems are being tested to determine the balance of properties attainable in such systems. Fracture toughness experiments have been conducted over a range of test temperatures and loading rates. Fatigue crack growth experiments have been conducted to determine the effects of changes in R-ratio and test temperature on both the threshold for fatigue as well as the Paris Law slope. Quantitative fracture surface analyses have been conducted in order to determine the effects of changes in such test conditions on the operative fracture modes. The effects of such changes in test conditions on the balance of properties will be presented and compared to similar work conducted on monlithic Nb.Partial support provided by AFOSR-F49620-96-1-0164 and AFOSR-F49620-00-1-0067.

10:50 AM

Crack Tip Plasticity,Alloying Effects and Fracture Toughness of Cubic Titanium Trialuminide Intermetallics: *Robert A. Varin*[1]; Les Zbroniec; [1]University of Waterloo, Mech. Eng., 200 University Ave.W., Waterloo, Ontario N2L 3G1 Canada; National Institute of Materials and Chemical Research, 1-1 Higashi, Tsukuba, Ibaraki 305-8565 Japan

The $L1_2$-ordered titanium trialuminides derived from $D0_{22}$-ordered Al_3Ti by alloying with fourth-period transition elements such as Cr,Mn,Co,Ni,Cu and Zn attracted much attention as potential high temperature structural materials. It was expected that due to their cubic lattice structure a noticeable improvement of room temperature tensile ductility and/or fracture toughness could be achieved. Unfortunately, their fracture toughness still remains quite low. In this work the results of fracture studies of cubic $(L1_2)Al_3Ti$ alloys stabilized with Mn will be presented. It has been observed that either very localized and planar plastic process zones or pseudo-bifurcated process zones form at the crack tips in cubic $(L1_2)Al_3Ti$ (9at%Mn) trialuminides. Surprisingly, a combination of increased Ti concentration (up to≈30at%) and boron doping improves fracture toughness at room temperature by a whopping 100%. In addition, at elevated temperatures up to 1000°C the increase of Ti concentration suppresses intergranular failure mode.

11:10 AM

Optimization of Toughness and Strength in Multiphase Intermetallics: *Ronald Gibala*[1]; Amit Misra[2]; Ronald D. Noebe[3]; [1]University of Michigan, Mats. Sci. & Eng., 2300 Hayward St., 2026 H. H. Dow Bldg., Ann Arbor, MI 48109-2136 USA; [2]Los Alamos National Laboratory, Los Alamos, NM 87545 USA; [3]NASA Lewis Research Center, Cleveland, OH 44135 USA

We have examined effects of fine precipitation in the matrix phase on strength and toughness of two NiAl-based alloys. In Ni-33Fe-21Al alloys, the B2 matrix is reinforced with ductile fcc-based second phases. Spinodal decomposition leads to fine-scale bcc matrix precipitates, producing a two-fold increase in strength, but with reduced ductility and toughness compared to materials without the strengthening phase. The high strength limits matrix plasticity prior to cleavage crack initiation, but some slip transfer occurs from the fcc-based phase to the B2+bcc matrix. The dendritic microstructure also accounts for lower toughness. In NiAl-31Cr-3Mo materials alloyed with Hf and Si and reinforced with bcc Cr(Mo) second phases, the precipitation of a cuboidal G-phase in the matrix causes significant strengthening. Reduced toughness relative to unalloyed NiAl-Cr(Mo) is attributed to lack of plasticity in the precipitate-strengthened matrix and partial loss of the aligned lamellar microstructure by Hf and Si alloying. Deformation mechanisms in these alloys are used to discuss microstructural design of multiphase intermetallics for optimized strength and toughness.

11:30 AM

Slip Transmission and Fracture Initiation in Rolled Ti-45Al-2Cr-2Nb Notched Tensile Specimens: *Boon-Chai Ng*[1]; Tom R.

Bieler[1]; Martin A. Crimp[1]; [1]Michigan State University, Mats. Sci. and Mech., 3504 Eng. Bldg., East Lansing, MI 48824-1226 USA

Dislocations and twins have been examined in specimens designed to study crack initiation of bulk equiaxed TiAl in an effort to examine the nature of grain to grain deformation transfer and to characterize the conditions which lead to slip transfer or crack initiation. Specially designed crack initiation specimens, cut in different orientations from a textured hot rolled sheet of TiAl, were deformed in-situ in an SEM. Electron backscattered patterns (EBSP), were used to characterize the micro-texture conditions in the notched area where microcrack initiation occurred, and electron channeling contrast imaging (ECCI) was used to directly image dislocations and twins in order to identify deformation conditions at grain boundaries. These examinations have been carried out either under the conditions of static loading or following the release of the load. Microtexture conditions favorable to enhanced toughness andductility are being identified with an aim to guide microstructure design.

11:50 AM

Impact Damage Effects on Threshold-Based Models of Fatigue Behavior of Two γ-TiAl XD™ Alloys: *Ryan M. Smith*[1]; Trevor S. Harding[2]; J. Wayne Jones[1]; [1]University of Michigan, Dept. of Mats. Sci. and Eng., 2300 Hayward St., 3062 H.H. DOW Bldg., Ann Arbor, MI 48109-2136 USA; [2]Kettering University, IMEB Dept., 1700 West 3rd Ave., Flint, MI 48504 USA

Gamma titanium aluminides have received significant attention as potential materials in aerospace applications such as turbine blades. Their high specific strength and stiffness and comparatively low density point to potential weight savings when compared to current materials. However, titanium aluminides exhibit relatively low ductility and limited damage tolerance. Fatigue crack growth rates are highly sensitive to changes in stress intensity range. The resulting short propagation lifetime is especially important where foreign object damage could eliminate the fatigue initiation lifetime. In this scenario, a damage tolerance approach would require more frequent inspections and significant increases in the life cycle cost of a gas turbine engine. A threshold-based design approach to fatigue of γ-TiAl may be a preferable choice. This work explores impact damage geometry and microstructural effects using a threshold-based model of fatigue behavior in two near-fully lamellar γ-TiAl XD™ alloys: a Ti-46.8Al-2.1Nb-1.1Mn-0.1Si-1.4B-0.01C-0.17O (at%) alloy and a Ti-45Al-2Nb-2Mn-0.8(vol%)TiB2 (at%) alloy. The shapes of cracks resulting from impact damage are quantified through fractography. Correlations between impact location, velocity and severity of damage will be shown in comparison with finite element analysis on a similar TiAl alloy. An estimate of the threshold stress for fatigue growth of impact damage will be compared with calculations using long-crack fatigue crack growth rate data for these two materials.

International Symposium on Shape Casting of Aluminum: Science and Technology: Advances in Process Simulation

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

Tuesday AM — Room: 224
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Jon A. Dantzig, The University of Illinois, Dept. of Mech. Eng., Urbana, IL 61801-2906 USA; Elwin L. Rooy, Rooy and Associates, Aurora, OH 44202 USA

8:30 AM Invited

Flow Visualization of the Lost Foam Casting Process: *David D. Goettsch*[1]; Mike Walker[1]; William Harper[2]; [1]General Motors Powertrain, 30003 Van Dyke Ave. mc 480-723-602, Warren, MI 48090 USA; [2]General Motors Powertrain, 1629 N. Washington, Saginaw, MI 48605 USA

The lost foam casting process is gaining acceptance for producing parts low in cost, high in quality and complex in geometry. Wider acceptance will require further understanding of the complex interaction between the metal, foam and coating during the metal filling process. Direct observation of molten aluminum front progression and foam regression using real-time x-ray and neutron radiography has been used to investigate these interactions. The results of casting A356 plates with varying thickness, inlet orientation and coating type will be presented. Experiment results indicated that the buoyancy effect on foam decomposition products can significantly alter the metal fill pattern and increase the metal-to-foam gap in thick sectioned foams. These visualization results should support the advancement of lost foam casting mathematical models.

9:00 AM Invited

Study on Numerical Simulation of Mold Filling and Solidification Processes of Aluminum Shape Casting under Pressure Conditions: *Baicheng Liu*[1]; Shoumei Xiong[1]; [1]Tsinghua University, Dept. of Mech. Eng., Tsinghua Garden, Beijing 100084 China

Numerical simulation of mold filling and solidification processes for high and low pressure die castings (HPDC & LPDC) was studied. A mathematical model considering the turbulent flow and heat transfer phenomenon during the HPDC process was established and the parallel computation technique was used to speed up the the mold filling simulation. The laminar flow characteristics of the LPDC process was studied and a simplified model of the mold filling process for aluminum alloy wheel hub casting was developed. The cyclic characteristics and the complicated boundary conditions were considered and the techniques to improve the computational efficiency were discussed. The verification and application of the simulation systems for both HPDC and LPDC processes were presented.

9:30 AM

Using Simulation to Solve Aluminium Casting Problems: *Mark R. Jolly*[1]; [1]The University of Birmingham, IRC in Matls., Edgbaston, Birmingham, West Midlands B15 2TT UK

The Castings Centre at the University of Birmingham, UK, has been using a combination of practical rules and computer simulation to supply running system solutions to industry over the past 5 years. The practical rules have come from a distillation of the work carried out by the Castings Research Group directed by Prof. John

Campbell using real time x-ray techniques to observe real filling systems. Despite this, specific detailed geometry of the running systems have to be developed by trial and error methods which in this case are performed using casting simulation software. The group now has access to over 10 commercial software packages for solving casting problems. This paper gives detailed descriptions of the development of a gravity die cast running system is described for an automotive power-train component in Al-Si-Cu. A second example gives the history of the development of the process for an Al wheel casting.

10:00 AM Break

10:30 AM

Using Computer Simulation to Optimize Boundary Conditions and Determine Processing Parameter Sensitivity in Aluminum Alloy Castings: *Joy Adair Hines*[1]; Ravi Vijayaraghavan[1]; [1]Ford Motor Company, Mats. Sci., MD 3182, 2101 Village Rd., Dearborn, MI 48124 USA

Increasingly, foundries and designers are turning to computer simulation to determine ways to optimize casting designs and improve casting quality and efficiency. However, optimization tends to focus on a narrow group of conditions to produce the best casting, whereas in real production situations foundries must contend with a large range of conditions. Computer simulations offer a tool with which to investigate such variations. In this study, the boundary conditions for a small permanent mold were first optimized using a proprietary program and experimental cooling curve data from the casting. A matrix of computer simulation experiments was run to determine the effect of variations in processing parameters on the casting. In particular, mold temperature, melt temperature, pressurization profile, and mold cooling were examined. Some of the results were compared with experimental data.

11:00 AM

Modeling and Measurement of Quenching Residual Stresses in W319: M. L. Newman[1]; *J. A. Dantzig*[1]; X. Y. Zhang[1]; H. Sehitoglu[1]; [1]University of Illinois, Mech. & Indust. Eng., 1206 West Green St., Urbana, IL 61801 USA

A study of the development of residual stresses in aluminum alloy w319 is presented. Experiments are described where a beam is quenched from solid solution to room temperature by water cooling on one side. The deformation of the beam is continuously monitored during cooling, and the residual stresses are measured by a groove removal technique. A model is also presented for the alloy in solid solution state. Rapid tension tests are performed to determine the mechanical response at a range of temperatures and different strain rates. These results are used to develop a constitutive model for W319. The model is implemented in ABAQUS, and is demonstrated to correlate well with the results of the beam quenching experiment.

Lead-Free Solder Materials and Soldering Technologies III: Fundamentals, Phases, Wetting, Surface Tension, Mechanics

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

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

Tuesday AM Room: 227
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Gautam Ghosh, Northwestern University, Dept. Mat. Sci. Eng., Evanston, IL 60208 USA; Ronald W. Smith, Materials Resources International, North Wales, PA USA

8:30 AM Invited

Thermodynamic Calculation of the Phase Equilibria: *Xing Jun Liu*[1]; Cui Ping Wang[1]; Ikuo Ohnuma[1]; Ryosuke Kainuma[1]; Kiyohito Ishida[1]; [1]Tohoku University, Grad. Sch. of Eng., Dept. of Mats. Sci., Aoba-yama 02, Aoba-ku, Sendai 980-8579 Japan

The phase equilibria and liquidus surface in the Cu-Sn-Sb system are important for the design of the Pb-free high-temperature solders and understanding of the interface reaction between Cu substrate and SnSb solder. However, the phase equilibria in the Cu-Sn-Sb system have not been well established, and the thermodynamic assessment of this system has not been carried out so far. In the present paper the phase equilibria of the Cu-Sn and Cu-Sb binary systems are re-assessed because the new experimental data are available for the Cu-Sn system. The thermodynamic assessment of the Cu-Sn-Sb system are carried out on the basis of the experimental results including the data of thermodynamic properties and phase equilibria. A consistent set of optimized thermodynamic parameters have been arrived at for describing the Gibbs energy of each phase in this system leading to a better fit between calculation and experiments. On the basis of the optimized parameters of the liquid phase, the surface tension of the liquid phase is also estimated and discussed in the present work.

8:55 AM

Computer Simulation of Time Dependent Wetting Behavior in the Wetting Balance: *Jae Yong Park*[1]; Jung Whan Han[2]; Hwang-gu Lee[2]; Dong Heun Kam[2]; Seung Boo Jung[1]; Choon Sik Kang[3]; Chul woong Yang[1]; Jaepil Jung[4]; [1]Sungkyunkwan University, Metall. & Mats. Eng., Chunchun-dong, Jangan-gu, Suwon, Kyunggi-do 440-746 Korea; [2]Inha University, Matls. Sci. and Eng. Dept., Yonghyun-dong, Younsu-ku, Incheon Korea; [3]Seoul National University, Mats. Sci. & Eng., Shillim-dong, Kwanak-gu, Seoul 151-742 Korea; [4]University of Seoul, Mats. Sci. & Eng., Jeonnong-dong, dongdaemun-gu, Seoul, Korea

Wetting balance test is known to be the most versatile method for wettability evaluation, because it can provide quantitative information and time dependent wetting behavior. With this method, two major results can be obtained: wetting time and wetting force. These two indices can be combined as a wetting curve. As to wetting force, analytical solution exists using Young's and Laplace equation, and static meniscus can be calculated using public domain code called "surface evolver". Mostly, surface tension of a solder and contact angle between a solder and a substrate are the key variables which determine the wetting force. However, as for wetting time, there is no theoretical background nor mathematical calculation; only experimental deduction exists. In this study, the wetting time is focused as a wettability index. Wetting curve which shows time dependent wetting behavior will be calculated using computer simula-

tion. The mechanism of meniscus rise will be analyzed. Also, based on this calculation, the major properties or variables for wetting time determination will be studied.

9:15 AM

Growth Rate and Morphological Instability: *Alexander R. Umantsev*[1]; [1]Saint-Xavier University, Chem./Phys., 3700 West 103rd St., Chicago, IL 60655 USA

Experimental observations show that growth of intermetallic compounds from the molten solders demonstrates complex dynamical behavior: the layer grows in the form of scallops and whiskers, as opposed to planar interface morphology in bimetallic solid-state couples. Up-to-date the question: "Why solder joints exhibit scallop-type growth mode?" remains unresolved. The main thrust of the current paper is to find the driving force of such instability. Theoretically intermetallic growth may be described within the framework of a three-phase Stefan problem. In the paper such problem is posed and solved taking into account temperature and concentration gradients that develop in the solidifying system. Analysis of the constitutional supercooling principle as a possible cause of the morphological instability is made. Comparison with the experimental results is carried out, and the estimate of the Cu-Sn interdiffusion coefficient is made. An experiment to elucidate the mechanism of morphological instability is suggested.

9:35 AM

Surface Tension Measurements of the Bi-Sn and Sn-Ag-Bi Liquid Alloys: *Zbigniew Stanislaw Moser*[1]; Wladyslaw Gasior[1]; Janusz Pstrus[1]; [1]Polish Academy of Sciences, Inst. of Metall. and Mats. Sci., Reymonta 25, Krakow 30-059 Poland

As the preliminary step in our studies on Pb-free solders there were performed surface tension, viscosity and density measurements of liquid Pb-Sn alloys,considering the fact, that the properties of each new soldering material are compared with those of Pb-Sn. Next, we have started with surface tension measurements of the Ag-Sn alloys at the entire range of concentrations including pure components and on the change of surface tension by small additions of Zn and In to the eutectic alloy Sn-3.8 at.% Ag. The main aim of this report is to summarize the influence of Bi additions on the surface tension of the eutectic alloy Sn-3.8 at.% Ag. In addition, there are presented surface tension data of pure Bi and liquid Bi-Sn alloys at the entire range of concentrations. Experimental data of Bi-Sn are compared with Butler's model and a very good agreement has been obtained. Surface tension measurements by the maximum bubble pressure method of the pure Bi and Bi-Sn alloys were determined at the temperature range from about 500K to 1150K and compared with previously reported data. Similarly, there were investigated ternary alloys, adding to the eutectic (Ag-Sn 96.2 at.%) 3, 6, 9 and 12 at.% Bi at the similar temperature range as in the case of Bi-Sn alloys. It has been confirmed that the additions of Bi to liquid Sn and to the eutectic alloy Sn-3.8 at.% Ag markedly reduces the surface tension.

9:55 AM

Tin Pest in Sn-0.5mass%Cu Lead-Free Solder: *Yoshiharu Kariya*[1]; Naomi Williams[1]; Colin Gagg[1]; William J. Plumbridge[1]; [1]The Open University, Mats. Eng. Dept., Walton Hall, Milton Keynes, Buckinghamshire MK7 6AA UK

Tin undergoes an allotropic transformation of white-tin into grey-tin (termed Tin Pest) at temperatures below 286K. The allotropic change is accompanied by an increase in volume of 26 per cent; this could have serious repercussions when considering solder joint lifetime. As tin pest has not been found in lead-tin alloys, it has not become the subject of any reliability screening of lead-free solders. Recently, we have revealed that tin pest can occur in tin-copper alloys. From an economic viewpoint, tin-copper eutectic may well become an important lead-free solder. Tin pest could have major ramifications when considering service lifetime of electronic assemblies using this solder. Therefore, a fundamental understanding of its allotropic transformation behavior will be required before any widespread implementation of the alloy can commence. In this paper, the nature and degree of the allotropic transformation behavior in Sn-0.5mass%Cu will be presented.

10:15 AM Break

10:30 AM

Use of Thermodynamic Data to Calculate Surface Tension and Viscosity of Sn-Based Soldering Alloy Systems: *Jong Ho Lee*[1]; Dong Nyung Lee[1]; [1]Seoul National University, Sch. of Mats. Sci. and Eng., College of Eng., Seoul 151-742 Korea

Thermodynamic database for the Pb-free soldering alloy systems, which include Sn, Ag, Cu, Bi, and In has been developed using CALPHAD method. The resulting thermodynamic properties of the Sn-based binary alloy systems are used to determine the surface tensions and viscosities. The surface tensions can be calculated using Butler's monolayer model, and viscosities by Hirai's model and Seetharaman's. Butler's model can also be used to determine the surface active element. The segregation of surface active elements gives the understanding of the de-wetting phenomenon after soldering. The results for binary systems have been extended to the Sn-based ternary systems(Sn-Ag-Cu, Sn-Ag-Bi, Sn-Bi-In). The surface tensions are measured by the sessile drop method, and these values are compared with calculated data.

10:50 AM

Studies of the Ag-In Phase Diagram and Surface Tension Measurements: Zbigniew Moser[2]; Wladyslaw Gasior[2]; Janusz Pstrus[2]; Wojciech Zakulski[2]; *Ikuo Ohnuma*[1]; Xing Jun Liu[1]; Yasuo Inohana[1]; Kiyohito Ishida[1]; [1]Tohoku University, Grad. Sch. of Eng., Dept. of Mats. Sci., Aoba-yama 02, Aoba-ku, Sendai 980-8579 Japan; [2]Polish Academy Science, Inst. for Met. Res. and Mats. Sci., Reymonta St. 25, Krakow 30-059 Poland

The phase boundaries of the Ag-In binary system were determined by diffusion couple method, DSC and metallographic techniques. The results show that region of the ζ (hcp) phase is narrower than that reported previously. Thermodynamic calculation of the Ag-In system is presented by taking into account the experimental results obtained by the present and previous works including the data of the phase equilibria and thermochemical properties. The Gibbs energies of liquid and solid solution phases are described on the basis of sub-regular solution model, and that of the intermetallic compounds are based on the two-sublattices model. A consistent set of thermodynamic parameters has been optimized for describing the Gibbs energy of each phase, which leads a good fit between calculated and experimental results. The maximum bubble pressure method has been used to measure the surface tension and densities of liquid In, Ag and five binary alloys at the tempertaure range from 227 to about 1227°C. On the basis of the thermodynamic parameters of the liquid phase obtained by the present optimization, the surface tensions are calculated using the Butler's model. It is shown that the calculated values of the surface tensions are in reasonable agreement with the experimental data.

11:10 AM

Application of an Asymmetrical Four Point Bend Shear Test to Solder Joints: *Ozer Unal*[1]; Iver E. Anderson[1]; Joel L. Harringa[1]; Robert L. Terpstra[1]; Bruce A. Cook[1]; James C. Foley[1]; [1]Ames Laboratory-USDOE, Metall. and Ceram. Prog., 207 Mets. Dev., Ames, IA 50011 USA

Determination of shear properties of solder joints is critical for design and reliable use of components in service. Since component testing is expensive and requires extensive experimental time, theoretical models are used for life prediction based on failure criterion in a given condition. These models require material properties obtained under a pure stress-state. However, most of the test methods used for solder joints generate a mixed-mode shear stress-state and thus, the values obtained from these tests may not be representative. In this study, application of an asymmetrical four-point bend (AFPB) test, which provides a pure shear condition to the solder alloy microstructure in the middle of a simple joint, will be shown. Test development efforts involving finite element modeling, specimen preparation and testing will be discussed. Preliminary shear strength, stress-relaxation and creep test results from Sn-Ag-Cu solders alloy will be presented. Support received from USDOE-BES, Materials Science Division (contract no.W-7405-Eng-82).

11:30 AM Invited

An Investigation of 58Bi-42Sn Solder Paste Wetting Behavior on Pb-free Metal Surface Finishes of Printed Circuit Boards (PCBs): *Valeska Schroeder*[1]; Fay Hua[1]; [1]Hewlett Packard Company, Electr. Sys. Techn. Ctr., 1501 Page Mill Rd., M/S 6U-A, Palo Alto, CA 94304 USA

Eutectic 58Bi-42Sn solder is considered a low melting point alternative to lead-based solders, particularly for consumer electronics applications. In such applications, the metallic bond between 58Bi-42Sn solder and the surface finish should be optimized for increased reliability. In this investigation, the spreading behavior of 58Bi-42Sn solder paste was evaluated on printed circuit boards with six metal surface finishes: organic coated copper (OCC), immersion silver, electroless nickel/immersion gold, two types of immersion tin, and hot air solder leveled tin-lead as a control. On these surfaces three no-clean and three water-clean flux chemistries were evaluated in a nine-zone convection reflow oven, flowing with air or nitrogen. These solder paste spreading tests were conducted on the as-received surfaces and after aging 5 and 10 days at 100°C with low relative humidity and at 85°C with relative humidity of 85% to simulate accelerated storage conditions. In addition to the spreading tests, the surface finishes were analyzed before and after aging with scanning electron microscopy. Wetting angles on all of the surfaces were calculated from measurements of the height and diameter of the solder cap. These results suggest superior spreading on immersion tin surfaces, less spreading on average compared to 63Sn-37Pb paste on all but the tin surfaces, a limited effect of nitrogen gas, and significant variation with flux chemistry. In addition, reflowed solder was aged at three temperatures in order to analyze accelerated intermetallic growth rates at the interface between 58Bi-42Sn solder and the metal surface finishes.

Lightweight Alloys for Aerospace Applications: Processing and Properties-I

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

Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Nack J. Kim, Center for 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

Tuesday AM — Room: 213
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Eui W. Lee, Naval Air Systems Command, Pax River, MD USA

8:30 AM Invited

Processing and Properties of Gamma Titanium Aluminides and their Potential for Aerospace Applications: J. Paul[1]; M. Oehring[1]; F. Appel[1]; and H. Clemens[1]; [1]GKSS Research Center, Institute for Matls. Res., Max-Planck-Str., D-21502 Geesthacht, Germany

The development of high-temperature materials is the key to technological advancements in engineering areas where materials have to withstand extremely demanding conditions. Examples for such areas are the aeroengine and aerospace industry. Intermetallic g (TiAl) based alloys offer many attractive properties for use in various hypersonic space and aerospace vehicles. These properties include high melting point, low density, good oxidation and burn resistance, good creep properties, and high specific strength up to application temperatures which are considered to be in the range of 700ºC to 900ºC. This paper reviews the present status in alloy development of g (TiAl) based alloys and thermomechanical processing on industrial scale. The progress achieved in forging of large ingots, rolling of sheets from forged ingot and powder compacts as well as single and multistep extrusion of ingots will be presented. The mechanical properties of sheets, extruded rods in as-processed condition and after subsequent heat-treatments will be compared. For further manufacture of semi-finished products to final TiAl components secondary processing steps are required. Examples for conventional and superplastic forming, machining and joining will be given. Finally, the fabrication of components from forged, rolled and extruded g (TiAl) material will be described and the results derived from component tests will be presented.

9:00 AM Invited

Hot Working, TMP and Superplasticity in Aluminum Alloys: *Hugh J. McQueen*[1]; Michael E. Kassner[2]; [1]Concordia University, Mech. Eng., 1455 Maisonneuve W, Montreal, QC H3G 1M8 Canada; [2]Oregon State University, Mech. Eng., Corvallis, OR 97331 USA

Hot forging and extrusion traditionally produce Al alloy components for aircraft, providing suitable shaping capabilities combined with creation of beneficial subgrain, grain, preferred orientation and fiber microstructures. Hot rolling produces plate for direct milling and strip for accurate finishing on cold mills followed by sheet-forming processes. The hot shaping can be controlled and integrated into thermomechanical processing (TMP) to produce microstructures with improved service properties or capable of superplastic behaviour. Superplastic forming is mainly applied to manufacturing hard-to-press sheet products, but it could be used for isothermal forging. Discussion of the above processes includes consideration of net strength and ductility, constitutive equations, microstructural evolution, restoration mechanisms and product properties. The study will include primarily 2XXX, 7XXX and 8XXX aerospace alloys but also some Al-Mg, Al-Mg-Mn and Al-Fe alloys. The consolidation and shaping of RSP and MA alloys is also considered.

9:30 AM

The Application of a Novel Technique to Examine Thermomechanical Processing: *Martin Jackson*[1]; R. J. Dashwood[1]; H. M. Flower[1]; Leo Christodoulou[1]; [1]Imperial College of Science and Technology and Royal School of Mines, Dept. of Mats., Prince Consort Rd., London SW7 2BP UK

A novel specimen design and testing strategy has been exploited to determine the effect of thermomechanical processing on the microstructural development of titanium alloys. A double truncated cone test geometry is isothermally deformed at near transus temperatures, to obtain microstructural information for a range of strains within a single specimen. A finite element modelling (FEM) package, is then employed to produce strain profiles, which readily correspond to the equivalent microstructural profiles of the test specimens. A parametric study of the effects of process (e.g. friction) and material (e.g. strain rate sensitivity) parameters on the strain distributions obtained during the test are also investigated. Such convergence of information can provide the basis of a constitutive equation to predict microstructural evolution. The effectiveness of this testing strategy is illustrated with a qualitative description of the microstructural evolution with strain, for various strain rates, at sub transus forging temperatures for different classes of titanium alloys.

9:55 AM

Microstructural Evolution During Hot Working of Ti-6Al-4V at High Strain Rates: *T. Seshacharyulu*[1]; Steve C. Medeiros[1]; William G. Frazier[1]; Y.V. R.K. Prasad[2]; [1]Air Force Research Laboratory, AFRL/MLMR, 2977 P St. Bldg. 653, Wright-Patterson AFB, OH 45433 USA; [2]Indian Institute of Science, Metall. Div., Bangalore 560012 India

Microstructural conversion from lamellar to equiaxed is an important step in the thermomechanical processing sequence of Ti-6Al-4V and is conventionally achieved by cogging in the alpha-beta phase field. Since hot working at higher strain rates (>0.1 s^{-1}) in the two phase field produces adiabatic shear bands and cracking, cogging is performed at slow speeds. Also, the occurrence of strain-induced porosity at lower strain rates and lower temperatures (<850°C) demands higher temperature control during cogging to obtain defect-

free products. In view of these difficulties, an effort has been made to find an alternative process for conversion. Isothermal compression tests conducted at high strain rates (1-100 s^{-1}) close to the beta transus revealed the evolution of an equiaxed microstructure consisting of alpha grains surrounded by a thin beta case. The new microstructure is found to be thermally stable and exhibited better mechanical properties over the conventional globularized structure. The equiaxed microstructure has been successfully reproduced under industrial manufacturing conditions using extrusion and subscale turbine-engine disk forging experiments at high speeds. The temperature-strain rate limits for the occurrence of the new microstructure are established by correlating the disk forging experimental results with FEM simulations.

10:20 AM

Novel High Temperature Aluminum (HTA) Alloys for Aerospace Applications: *Shihong Gary Song*[1]; [1]United Technologies Research Center, Components, 411 Silver Lane, East Hartford, CT 06108 USA

The strength of conventional aluminum alloys is sensitively dependent on temperature. They are rarely used above 150°C due to a sharp reduction in strength with temperature. In most cases, titanium and nickel based alloys are used for structural applications in the temperature regime of 150 ~ 350°C. They are, however, considered overkill in many instances and inevitably lead to high costs and weight penalties. Aluminum materials or composites can be used in place of titanium and superalloy, provided that the strength of the former is enhanced at the temperature regime. To this end a strengthening mechanism other than precipitation has to be employed, which should be less temperature sensitive. In this light, new dispersion strengthened aluminum alloys are being developed, which have demonstrated promising properties for elevated temperature applications.

10:45 AM

High Ductility Cast Aluminum Beryllium Alloys: *Nancy F. Levoy*[1]; William T. Nachtrab[1]; [1]Starmet Corporation, Res. and Dev., 2229 Main St., Concord, MA 01742 USA

Cast aluminum beryllium alloys have recently been introduced in the market for high performance aerospace applications where high stiffness and low density are critical properties. During solidification of cast aluminum beryllium alloys, a two phase composite microstructure develops in which the primary beryllium phase forms within an aluminum matrix. These alloys typically contain more than 60 weight percent beryllium and are approximately 20% lighter and 3 times stiffer than conventional aluminum alloys. However, these high beryllium alloys are limited by low ductility and the high cost of beryllium. New alloys with lower beryllium content have now been developed, which optimize mechanical properties such as strength and ductility while still providing high specific stiffness. This paper will describe how alloy composition can be tailored to provide different combinations of properties based on application requirements. Results will be discussed in the context of property development in metal matrix composites.

11:10 AM

On the Crystallographic Texture of the 2195, C458, 2090, 2297 Aluminum-Lithium and 7249 Alloys: A. Zahmehr[1]; Y. Ren[1]; D. Hamilton[1]; J. Foyos[1]; J. Ogren[1]; E. W. Lee[1]; H. Garmestani[1]; *O. S. Es-Said*[1]; [1]Loyola Marymount University, Res. Exper. for Undergrad. Prog., Los Angeles, CA 90045 USA

As-received and heat treated and processed samples of 2090, 2297, 2195, C458 aluminum-lithium, and 7249 alloys were studied in this experiment. The Schultz Reflection method of x-ray diffraction in Scintag X1 diffraction system was used to obtain the generic scans and pole figures of these alloys. The intensity of deformation, shear and recrystallization components are determined by Orientation Distribution Functions. All samples were cold worked by rolling to 50% reduction in thickness, and were tested. Another set of rolled samples was annealed at 900ºF for 1 hour, and was also tested. Some samples were cut from half of the thickness of the plates and the others from a quarter of the thickness of the plates.

11:35 AM

Observations of the Effect of Crystal Orientation on Cavitation in Hot Tensile Deformation of Ti-6Al-4V: T. R. Bieler[1]; S. L. Semiatin[2]; [1]Michigan State University, Dept. of Mats. Sci. and Mech., 3536 Engineering Bldg., MI 48824-1226 USA; [2]Air Force Research Laboratories, Mats. and Manuf. Direct., AFRL/MLLM, Wright Patterson AFB, OH 45432 USA

Cavitation during hot tensile deformation in Ti alloys is commonly observed. Prior work indicated that cavities are often located in grain boundaries perpendicular to the tensile axis where the lamellae appear nearly parallel to the tensile axis. Since colony interfaces tend to include the c-axis, it has been hypothesized that cavities develop preferentially in boundaries that have the hard c-axis orientation parallel to the tensile axis. Two deformed specimens were investigated to evaluate this hypothesis using orientation imaging microscopy, by measuring the crystal orientations surrounding nucleated cavities. In a tensile specimen deformed at 815°C and 0.1/s strain rate, small cavities observed far from the fracture surface were in agreement with the hypothesis. In regions with more strain, more cavity growth was apparent when a non-c-axis grain was present as a minority orientation at or near a cavity. Similar orientation relationships around cavities were observed in hoop tensile regions of an upset forged cylindrical sample deformed with the same conditions. The role of crystal orientation and boundary misorientation as parameters to predict cavitation behavior is discussed.

11:55 AM

The New Aluminum Alloy 6069: S. C. Bergsma[1]; M. E. Kassner[2]; [1]Northwest Aluminum Company, The Dalles, OR 97958 USA; [2]Oregon State University, Corvallis, OR 97331 USA

AA 6069, a new aluminum alloy, has been developed for application in hot and cold extrusion and forging. The nominal composition is 0.85%Si, 0.25%Fe, 0.70%Cu, 1.35%Mg, 0.20%Cr, and 0.15%V. Average T6 properties of the ingot without hot or cold deformation are 410 MPa ultimate tensile strength, 375 MPa yield strength and 12% elongation. Average properties after hot and/or cold extrusion range from 395-475 MPa ultimate tensile strength, 350-450 MPa yield strength, and 14-20% elongation. This alloy also has favorable fatigue, corrosion-fatigue and stress-corrosion and sustained load cracking properties due to a combination of composition, high solidification rate, thermal and mechanical processing, and T6 practice. Careful fracture toughness testing revealed that 6069-T6 is comparable or superior to that of 6061-T6 with identical ingot prepa-

ration and subsequent forming procedures. Careful TEM, SEM, optical microscopy and EDS were used to characterize the precipitation features and the basis for improved mechanical properties over alloys such as 6061-T6. The recrystallization and quench sensitivities have also been characterized and compared with 6061.

Magnesium Technology 2001: Casting and Solidification

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

Tuesday AM
February 13, 2001
Room: 203-205
Location: Ernest N. Morial Convention Center

Session Chair: David Creber, Alcan International, Ltd., Kingston R&D Center, Kingston, Ontario K7L 5L9 Canada

8:30 AM
Magnesium Alloy Sheet Produced by Twin-Roll Casting: *Daniel Liang*[1]; Daniel R. East[1]; Tracey J. Johnson[1]; Ross V. Allen[1]; Wendy E. Borbidge[1]; [1]CSIRO, Div. of Manuf. Sci. and Techn., Private Bag 33, Clayton South MDC, Clayton, Vic 3168 Australia

Twin-roll casting has been used to produce near-net shape sheet of Mg-3 to 9%Al based alloys, followed by thermo-mechanical processing of the as-cast sheet by hot-rolling and heat treatment. The microstructures of both the as-cast and hot-rolled magnesium alloys have been characterized so as to investigate the effects of near-rapid solidification from twin-roll casting and of the subsequent thermo-mechanical processing on the morphology, size and distribution of the microstructural components. Compared to similar alloys processed by conventional casting and rolling technology, these alloys produced via twin-roll casting have been found to exhibit homogeneity of microstructure, refined grain size, reduced segregation and increased solid solubility. Mechanical properties have also been evaluated and have been shown to largely increase as a result of the associated improvements in microstructure.

8:55 AM
Solidification Behavior of Commercial Magnesium Alloys: *Qingyou Han*[1]; Edward A. Kenik[1]; Sean R. Agnew[1]; Srinath Viswanathan[1]; [1]Oak Ridge National Laboratory, Met. and Cera. Div., P.O. Box 2008, Oak Ridge, TN 37831-6083 USA

The solidification behavior of magnesium AZ91D, AM60B, AS41 and AS21 alloys have been simulated using ThermoCalc. Phase fractions and the temperature at which each phase precipitates have been predicted. Comparison of the predictions with optical and scanning electron micrographs from permanent mold cast samples show good agreement. The segregation of solute elements in the primary magnesium phase has also been calculated. The simulation shows that aluminum and zinc are highly segregated in between dendrites and near the grain boundaries of the primary magnesium phase. Based on the segregation of solutes, the homologous temperature distribution within a primary magnesium grain, defined as the ratio of the use temperature to the local solidus temperature, has been calculated. The simulation results indicate that the homologous temperature near the grain boundary is much higher than that in the center of the grain. This suggests that creep deformation may occur mainly near the grain boundary.

9:20 AM
The Effect of Aluminium Content and Grain Refinement on Porosity Formation in Mg-Al Alloys: Paul L. Schaffer[1]; *Young C. Lee*[1]; *Arne K. Dahle*[1]; [1]CRC for Cast Metals Manufacturing, Dept. of Min., Min. and Mats. Eng., The University of Queensland, Brisbane, Qld 4072 Australia

Porosity is detrimental to the mechanical properties, surface finish and pressure tightness of castings and it is therefore important to understand the mechanisms that control porosity formation. A significant amount of research has been performed on the relationship between composition and porosity formation in aluminium alloys, however little work has been performed on magnesium-based alloys. Size and morphology of primary phase and eutectic, permeability and solidification range are influenced by alloy composition and grain refinement and their impact on porosity formation has been studied in the present work. Castings of varying aluminium content from pure magnesium to Mg-33%wt Al alloy were produced and the sample density was analysed using Archimedes principle to determine the effect of alloying content on porosity formation. The porosity location and morphology was then characterised by optical microscopy. Grain size of the same alloys was then refined by several different methods, depending on alloy content, to investigate the effect of grain size on porosity.

9:45 AM
Effects of Beryllium Content in Thixomolding® of AZ91D: *D. Matthew Walukas*[1]; [1]Thixomat, Inc., 620 Technology Dr., Ann Arbor, MI 48108 USA

A comparison of the Thixomolding® and die cast processes is presented. Be is added to magnesium alloys to help prevent oxidation and improve fluidity. AZ91D for die cast applications typically contains 5-15 ppm Be. AZ91D was molded using the Thixomolding® process with Be levels of 0, 3, 7, 15, 20 ppm at three different solid fractions, indicating a reduced need for Be in Thixomolding® compared to die casting. Oxygen content was measured in the feed material and the resulting parts using the neutron activation method, showing limited increases in oxygen levels in the Thixomolding® process. Microstructural comparisons and mechanical testing results are presented.

10:10 AM Break

10:20 AM
The Influence of Primary Solid Content on the Tensile Properties of a Thixomolded AZ91D Magnesium Alloy: *Frank Czerwinski*[1]; Pierre J Pinet[1]; J. Overbeeke[1]; [1]Husky Injection Molding Systems Limited, Thixosys., 480 Queen St., Bolton, Ontario L7E 5S5 Canada

Thixomolding®, an emerging semisolid technology, was used to process an AZ91D magnesium alloy under experimental conditions designed to yield from 5% to 60% of the primary solid particles. The thixotropic microstructures obtained were characterized in detail and linked to the corresponding tensile properties. An increase in primary solid content was accompanied by its larger microchemical and microstructural inhomogeneity expressed by Al and Zn segregation, sub-micron precipitates of Mg17Al12 and eutectic islands. At the same time, the size of Mg grains within the eutectic mixture was reduced. For the volume fraction of the primary solid up to about 20%, the tensile strength and elongation remained at the level of 240 MPa and 4.5%, respectively. A further increase of the primary solid caused a reduction in both strength and ductility. The fractographic analysis revealed a correlation between the primary solid content and the morphology of the decohesion surface. It is concluded that for alloys with a solid fraction below approximately 20%, the internal structure of the primary solid and the eutectic mixture control the properties. For a large volume of unmelted fraction, the interface between the primary solid and the eutectic mixture is a key factor which controls the tensile properties of the thixomolded alloy. Thixomolding and Thixomolded are registered trademarks of Thixomat Inc., Ann Arbor, MI.

10:45 AM

Welding of Magnesium Alloys: *Ulrich Draugelates*[1]; Antonia Schram[1]; Christian Kettler[1]; [1]Technical University Clausthal, Inst. of Weld. and Mach., Agricolastrasse 2, Clausthal-Zellerfeld D-38678 Germany

The scope of the presentation focuses on the Nd:YAG laser beam welding and the friction welding of conventional magnesium alloys of the AZ- and AM- classification as well as alloys containing rare elements. The base materials used for the experiments are cast-alloys as well as wrought-alloys. A description of the process and also quality relevant process parameters and resulting joining properties are discussed. The mechanical properties and the grain structures of the various areas of the welding are subjects of major interest. Another focus is put on the proof of the use of statistical test methods for welding applications to lower the amount of test runs and to get a detailed description of the influences and interactions between process parameters and marks of quality. With regards to the results of friction welding a newly defined factor for the weld seam quality will be introduced which describes the ratio between energy input and deformation energy.

Materials Processing Fundamentals III

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

Tuesday AM — Room: 218
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Mark E. Schlesinger, University of Missouri, Dept. of Metal. Eng., Rolla, MO 65409-0001 USA

8:30 AM

Development of a Mathematical Model Using Abaqus to Simulate Industrial Hot Tandem Rolling of AA5XXX Aluminum Alloys: Mary A. Wells[1]; Daan Maijer[1]; M. R. Van der Winden[2]; *Simon P. Jupp*[1]; [1]University of British Columbia, Mets. and Matls. Eng., 6350 Stores Rd., Vancouver, British Columbia, Canada; [2]Corus Group

A 2D coupled thermal-mechanical model was developed to simulate hot tandem rolling of AA5xxx aluminum alloys using the commercial finite element software package, ABAQUS. The model was used to predict the temperature, strain rate and strain distribution in the strip at any position in the roll bite as well as the temperature through the thickness of the strip at any position in the interpass region. The paper describes the set-up of the model in ABAQUS including, the boundary conditions used in the roll bite and interstand regions as well as the material constitutive behaviour. The model was validated through comparisons with literature and industrial data. Preliminary microstructure equations have been incorporated into the model using literature data and an initial sensitivity analysis to processing parameters has been completed.

8:55 AM

Combined Thermodynamic and Kinetic Models for Processing of Materials: *Pertti S. Koukkari*[1]; Karri Penttilä[1]; Klaus Hack[2]; Gunnar Eriksson[2]; [1]VTT, Chem. Techn., P.O. Box 1404, Espoo 02044 Finland; [2]GTT Technologies, Kaiserstrasse 100, Herzogenrath D-52134 Germany

Computer simulation has become an everyday practice in thermodynamic and materials research. Advanced thermochemical algorithms take into account heat transfer effects and salient reaction kinetics when applied for process models. In particular, the combination of overall reaction rates with multi-component Gibbs energy minimisation provides an effective tool for simulation of industrial thermochemical processes and processing of materials. In this work the combination of reaction kinetics and heat transfer with the Gibbs energy technique was studied by using the novel program ChemSheet, which operates through Microsoft's Excel® spreadsheets. Such processes as: CVD process model for pure silicon production; thermochemical process model for titania pigment manufacturing; vapour pressure of Mercury in amalgams (Hg-lamp manufacturing); melting behaviour of low-temperature alloys have been modelled. With the thermochemical data available simulations can be done with any thermodynamic system. The calculation can be performed as an affinity study without a particular reactor model or with a specific reactor scheme.

9:20 AM

Use of Surface Evolver to Predict Liquid Breakthrough Pressures in Packed Spheres: Jon L. Hilden[1]; *Kevin P. Trumble*[1]; [1]Purdue University, Mats. Eng., 1289 Mats. and Elect. Eng. Bldg., West Lafayette, IN 47907-1289 USA

The displacement of one fluid by another in particulate solids is a necessary step in a wide variety of processes including filtration of liquid metals, drying of ceramics, and infiltration of metals into porous ceramics to produce composites. Such processes are typically characterized by a breakthrough capillary pressure, which depends on the contact angle, pore (particle) size, and shape. However, even for regular-packed spheres, solutions for the breakthrough pressure have required approximations of the actual pore geometry. The Surface Evolver numerical analysis program has been used to calculate general solutions for the capillary (breakthrough) pressure of liquid in pores defined by packed spheres (e.g. tetrahedral and octahedral pores, etc.). The results are discussed relative to the many approximate solutions, as well as experimentally measured breakthrough pressures and satiation pressures in packed spheres over a wide range of contact angles.

9:45 AM

Stability of Hall-Heroult Cells: *Kjell Kalgraf*[1]; [1]Elkem Research, Box 8040, 4675 Kristiansand, Norway

The motion and stability of the metal bath interface is determined by Navier-Stokes equation. This equation may be decomposed into 2 independent equations by taking the divergence and the curl of Navier-Stokes equation. The divergence describes the motion and stability of gravitational waves, and leads directly to a criterion of stability that is a modification of Sele's criterion of stability. The curl of Navier-Stokes equation leads to an equation containing only velocity and magnetic field and not gravity, and tells that the magnetic field is driving a circulation pattern inside the pot. This curl equation has stability criteria of its own, related only to magnetic field and geometry. The waves associated with this equation are called Alfven waves. In this paper we develop both the modified Sele criterium and the criterion of stability of Alfven waves, and present stability data from real pots.

10:10 AM Break

10:20 AM

Atomistic Simulations of Defect Formation Processes during Crystallization of Melted Silicon: *Manabu Ishimaru*[1]; [1]Osaka University, The Instit. of Sci. and Indust. Rsch., Mihogaoka, Ibaraki, Osaka 567-0047 Japan

Reduction of defects in crystal silicon (c-Si) wafers is strongly required as device sizes are decreased in Si integrated circuits. These wafers are mostly produced from the liquid phase, therefore, understanding the defect formation mechanisms from liquid Si is of technological importance to reduce defects in c-Si. In the present study, we have performed molecular-dynamics calculations to examine microscopic structures of the defects and their formation processes in Si grown from the melt. The findings of this investigation are as follows: (i) The [110] bonds at the solid-liquid interface induce the eclipsed configurations or hexagonal Si structures which stabilize microfacets composed of the {111} planes. (ii) Defect formation during crystal growth processes is due to misorientations at the {111} interfaces which result in an 'elementary' grown-in defect structure including five- and seven-member rings. (iii) The 'elementary' grown-in defect migrates in c-Si by bond-switching motions during further crystal pulling or annealing.

10:45 AM

A New Numerical Model for Predicting Carbon Concentration during RH Degassing Treatment: *Young Geun Park*[1]; Kyung Woo Yi[1]; [1]Seoul National University, Sch. of Matls. Sci. and Eng., SAN 56-1 Shinrim-dong, Kwanak-ku, Seoul 152-742 South Korea

A new decarburization model which are coupled with three dimensional numerical flow simulation program for the RH process was constructed. Shapes of the Ar plumes which are formed by agron blowing at the wall of up-snorkel are calculated in order to estimate driving force for melt circulation and reaction site area. In this model, Ar bubble surface, bath surface, and inner sites were considered as decarburization sites. Using this numerical model, carbon and oxygen concentration were calculated transientely during RH process for various operating parameters. The calculated results were verified by comparison with real RH operation data. At initial stage, decarburizaiton at inner site was dominant but bath surface decarburization became dominant at last stage (less than 100 ppm carbon). Therefore, in order to accelerate decarbuization rate in the RH process, the reation rate in the vessel surface must be increased.

11:10 AM

Distributions of Various Structural Species in Alkali Borate Melts: *Zhijing Zhang*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Dept. of Metall. and Mats. Eng., P.O. Box 870202, Tuscaloosa, AL 35487-0202 USA

A thermodynamic structure model for alkali borate melts is developed to describe the distribution of structural groups in the alkali borate melts as a function of compositions. Fractions of threefold coordinated boron atoms and fourfold coordinated boron atoms in the potassium borate melt are calculated for the entire composition range and compared with results reported in the literature. The model has been used for structural interpretation for the borate anomaly in viscosity, which regards the borate anomaly as a manifestation of the various antithetical effects caused by the introduction of alkali oxide into boric oxide. It has been found that the viscosities of alkali borate melts increase or decrease according to the changes in rigidity and spatial connectivity of the glass network structure, which is a function of temperature and composition.

11:35 AM

Coupling of Thermal Grooving and Migration of Inclined Grain Boundaries: Huifang Zhang[1]; *Harris Wong*[1]; [1]Louisiana State University, Mech. Eng. Dept., Baton Rouge, LA 70803 USA

Grain boundary migration is a fundamental process governing grain growth. The motion of a grain boundary is significantly affected by the presence of a free surface because of the formation of a groove at the triple junction. The interaction of grain boundary migration and thermal grooving has not been studied in detail. We have coupled thermal grooving and grain-boundary migration for slightly inclined grain boundaries, and obtained free-surface and grain-boundary profiles. We assume that thermal grooving results from surface diffusion whereas grain boundary migration obeys a curvature-driven law of motion. A range of length and time scales are needed to describe the coupled motion. It is found that the grain boundary is never pinned. We will present these results and discuss the implications.

Materials & Processes for Submicron Technology: Metallization 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

Tuesday AM — Room: 226
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Ashok Kumar, University of South Florida, Tampa, FL 33620 USA; Bart Van Schravendijk, Novellus Systems, Inc., 4000 N. First St., San Jose, CA 95134 USA

8:30 AM Invited

Nickel Silicide Formation with a Presilicide Nitrogen Implantation: *Pooi See Lee*[1]; Dominique Mangelinck[2]; Kin Leong Pey[3]; Jun Ding[1]; Alex See[4]; [1]National University of Singapore, Dept. of Mat. Sci. (NUS), 10 Kent Ridge Crescent 119260 Singapore; [2]Institute of Materials Research and Engineering, Microelec., IMRE, 3 ResearchLink 117602 Singapore; [3]National Univerity of Singapore, Dept. of Electr. Eng., (NUS), 10 Kent Ridge Crescent 119260 Singapore; [4]Chartered Semiconductor Manufacturing, R&D, 60 Woodlands, Industrial Park D St. 2 738406 Singapore

The key feature of our study is to incorporate N_2^+implant prior to Ni sputtering on the poly-Si gate and source/drain regions. Effects of N_2^+implant on the silicide formation on Si (100) and undoped poly-Si substrates were studied using X-Ray Diffraction, micro-Raman Spectroscopy, Secondary Ion Mass Spectroscopy, Scanning Electron Microscopy and Rutherford Backscattering Spectroscopy. Our study shows that N_2^+ implant is able to suppress agglomeration in the Ni-silicide films up to 900°C and enhanced the phase stability of NiSi on Si(100) up to 750°C. Stable and low sheet resistance (4.3 ohms/sq) was achieved on the silicided undoped poly-Si up to 700°C due to reduced poly-inversion. Poly-inversion is driven by grain boundary energy and surface energy of the poly-Si. The reduction of poly-Si inversion might be caused by the segregation of nitrogen at the grain boundaries.

9:00 AM

Nickel Silicide as a Contact Material for Deep Sub-Micron CMOS Devices: *Chi Dongzhi*[1]; D. Mangelinck[1]; A. S. Zuruzi[1]; S. K. Lahiri[1]; [1]Institute of Materials Research and Engineering, 3 Research Link 117602 Singapore

Nickel monosilicide (NiSi) is an attractive alternative to the currently used silicides for the coming generations of deep sub-micron CMOS devices. This silicide material has a resistivity, which is comparable to that of TiSi2 or CoSi2, but consumes less silicon for this formation. The silicide-silicon interface is relatively planar, and unlike TiSi2 its resistivity does not change with the line width for narrow lines. However, the thermal stability of NiSi is relatively poor at the currently used temperatures during process integration. Recent investigations have shown that the stability of such films could be increased substantially through small additions of alloy elements, which do not increase the silicide formation, and thus the leakage current, can be minimized significantly through appropriate selection of rapid thermal annealing temperatures. In the paper the details of these experimental results will be presented and discussed.

9:20 AM Invited

Dissociation Behavior of Dilute Immiscible Copper Alloy Thin Films: *James M. E. Harper*[1]; K. Barmak[2]; G. Lucadamo[3]; C. Cabral[1]; C. Lavoie[1]; [1]IBM T.J. Watson Research Center, Rm. 12-254, P.O. Box 218, Yorktown Heights, NY 10598 USA; [2]Carnegie Mellon

University, Pittsburgh, PA USA; [3]Lehigh University, Bethlehem, PA 18015 USA

Copper interconnections continue to shrink along with silicon device dimensions, placing increased demands on the properties of the copper wiring itself. A possible strategy for extending copper wiring well beyond the 100 nm device generation is to add dilute additive concentrations, however, these concentrations must remain very low to retain the high conductivity advantage of copper. Supersaturated alloys of elements immiscible in copper undergo precipitation and microstructure evolution during the first heating. Here, we describe the dissociation behavior of dilute, immiscible copper alloy thin films and show that it falls into three broad categories that correlate with the form of the Cu-rich end of the binary alloy phase diagrams. Eight alloying elements were selected for these studies, with five elements from groups 5 and 6, two from group 8, and one from group 11 of the periodic table. They are respectively V, Nb, Ta, Cr, Mo, Fe, Ru and Ag. The progress of precipitation in approximately 500 nm thick alloy films, containing 2.5-3.8 at.% solute, was followed with in situ resistance and stress measurements as well as with in situ synchrotron x-ray diffraction. Texture analysis and transmission electron microscopy were also used to investigate the evolution of microstructure of Cu(Ta) and Cu(Ag). For all eight alloys, dissociation occurred upon heating, with the rejection of solute and evolution of microstructure often occurring in multiple steps that range over several hundred degrees between approximately 100 and 900ºC. However, in most cases, substantial reductions in resistivity of the films took place below 400ºC, at temperatures of interest to copperinterconnection schemes for silicon chip technology.

9:50 AM

Electrochemical Deposition of Copper on PVD-W2N Liner Materials for ULSI Devices: *Michael J. Shaw*[1]; Stephan Grunow[2]; David J. Duquette[1]; [1]Rensselaer Polytechnic Institute, Troy, NY USA; [2]State University of New York at Albany, NY USA

Electrochemical deposition of copper is the preferred metallization scheme for filling on-chip interconnections in ULSI semiconductor devices. As device features approach 70 nm, with 5-10 nm barrier coatings, application of a copper seed layer by conventional PVD or CVD techniques becomes severely limited, and a different strategy is needed. This presentation reports results obtained from electrochemical studies designed to identify process and chemical parameters, which provide adequate nucleation and thin film growth directly on ultra-thin, PVD-tungsten nitrade diffusion barriers. It is shown that very thin copper films can be nucleated directly on a conducting PVD-W2N liner surfaces. The nucleation and growth mechanism, film resistivity, and film adhesion are dependent on both ECD process parameters and electrolyte chemistry. Nucleation process and film quality are also dependent on the spatial relation of the electrical contact and on barrier film thickness.

10:10 AM Break

10:30 AM Invited

Challenges in Cu-Low k Dual Damascene Integration: *Bart Van Schravendijk*[1]; [1]Novellus Systems, Inc., 4000 N. First St., San Jose, CA 95134 USA

To enable modern logic devices to sustain the device speed improvements that come with scaling of transistors, device manufacturers are rapidly implementing new materials, such as Cu and low k dielectrics in the production line. A selection of the resulting challenges in the process integration will be discussed in this paper. Cu metallization requires not only excellent diffusion barriers, but extremely careful management of the thermal budget to prevent void formation and hillocks and even to ensure the expected low resistance and high electromigration resistance in narrow lines. The compatibility of Cu with dielectrics is dependent on reducing the effects of oxidation: Cu requires specific treatments to ensure adhesion of dielectric layers. For dielectrics the lowering of the dielectric constant has meant reduction of the mechanical integrity compared to SiO_2 and FSG. In addition low k materials interact differently with lithography, etch and CMP than conventional dielectrics. Although tradeoffs must therefore be made between the resulting integration complexity and performance, optimization of dielectric deposition of PECVD SiOCH films can minimize the cost and complexity increases and permit successful Dual Damascene integration with a conventional 'Via First' strategy.

11:00 AM Invited

Effects of Deposition Conditions of Al and Ti Underlayer on Electromigration Reliability for Deep-Submicron Interconnect Metallization: *Young-Bae Park*[1]; H. H. Ryu[1]; W. G. Lee[1]; [1]Hyundai Electronics Industries Company, Ltd., L15 Proc. Dev. Team, System IC R&D Cen., 1, Hyangjeong-dong, Hungduk-gu, Cheongju-si 361-725 Korea

Dependence of the microstructures, electrical resistances and electromigration reliability of Ti/Al-0.5%Cu/Ti/TiN metal stack interconnections on the deposition conditions of Ti underlayer and Al alloy are investigated. Effects of Ti underlayer are compared for the conventional, collimated, and IMP(Ionized Metal Plasma) sputtering deposition methods. And the effects of Al deposition power and temperature variations are also investigated. Structural characterizations are performed using XRD, AFM and cross-sectional TEM. And electrical characterizations are performed by the measurements of sheet resistance from blanket films and Kelvin resistance from multi-level Al metallization structures. Also, EM(electromigration) lifetimes of metal line pattern are compared using wafer level EM testing method which uses highly accelerated test conditions over conventional package-level EM test. At last, the effects of deposition conditions of Ti underlayer and Al alloy on EM reliability and electrical resistances of Al stack in terconnections can be well explained from Al (111) texture analysis and Ti/Al reaction results.

11:30 AM

Plastic Deformation of Thin Al and Cu Films During Thermal Cycling: *Indranath Dutta*[1]; *M. W. Chen*[1]; [1]Naval Postgraduate School, Dept. of Mech. Eng., 200 Dyer Rd., Monterey, CA 93943 USA

Due to differences in thermal expansions between thin metallic films and silicon substrates in microelectronic devices, high stresses can develop during thermal excursions experienced in processing steps or service, which may induce plastic deformation of the thin films accompanied by creep and interfacial sliding. These stresses and deformation processes can have a pronounced effect on the reliability of microelectronic devices and components. Even though various methods have been proposed to display the thermal stresses, how to measure the plastic deformation of thin films is still an unsolved issue. Here, we report our results on the plastic deformation of thin Al and Cu films on Si substrates during thermal cycling. The cross profiles of pattern-grown Al and Cu films with thickness of ~250nm and a size of ~0.005 mm X 0.005 mm were measured before and after thermal cycling by employing an atomic force microscope. With the assistance of statistical analysis, the size changes of the thin films induced by thermal cycling were determined. Combining microstructural analysis and finite element calculations, the plastic deformation of the thin films constrained by the Si substrates is attributed to the diffusion-controlled interfacial sliding.

11:50 AM Invited

Nanogravimetry Measurements in Advanced Materials: *Dentcho Ivanov*[1]; [1]New Jersey Institute of Technology, Microelect. Cen., 161 Warren St, Newark, NJ 07101 USA

Achieving an understanding and control over the properties of wide range of materials at the nanometer scale has become a major task in materials research. So far, scanning-tunneling microscopy (STM) studies and atomic force microscopy (AFM) have been the main characterization techniques used in the study of nanomaterials. These techniques are excellent tools for static studies of nanscale materials. However, for dynamic studies such as particle diffusion, ion transport during charging and discharging processes of redox reactions, stoichiometry determination during ion intercalation, phase transitions and various interface processes in metal ceramic thin film structures, STM and AFM do not offer very useful information. In such processes, nanogravimetric-time measurements could provide more information about detailed changes in a nanoscale structure. Polymer electrolytes are considered to be the basic material of lithium polymer batteries. Nanogravimetry study of the lithium transport process in ion conducting polymers provides im-

portant information about the lithium ion movement in the separator as well as about the ion intercalation process in the battery's cathode. A nanobalance is a particularly useful tool in the study of transport processes in mixed conductors. In this paper, we analyze the capabilities of a quartz-resonator nanobalance as a mass-sensitive detector in nanogravity electrochemical experiments. We show that in experiments where the nanomaterial film is involved in some chemical reaction, the resonance-frequency shifts are due not only to mass-related changes in the film, but also to changes in the film density, mechanical structure, and electrical properties. The change in resonance frequency due to changes in film-density or mechanical properties may, under some circumstances, provide information concerning the chemical structure of the nanomaterial thin film. Metal coating by Zn phosphatation is analyzed.

Properties of Nanocrystalline Materials: Deformation & Fracture

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

Tuesday AM Room: 223
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Horst Hahn, University of Darmstadt, Darmstadt D-64295 Germany; A. K. Vasudevan, Office of Naval Research, VA USA

8:30 AM Invited
Ductility and Superplasticity in Nanostructured Materials: *Carl C. Koch*[1]; [1]North Carolina State University, Matls. Sci. and Eng. Dept., P.O. Box 7907, 2401 Stinson Dr., Raleigh, NC 27695 USA

The fundamental mechanisms for deformation and fracture of nanocrystalline (nc) materials-at least in the range of the finest grain sizes (< 30nm)-are not well defined. The mechanical property of ductility is probably least well understood. Ductility-plastic deformation limited by the onset of failure mechanisms-can be considered with respect to two broad classes of failure criteria. These are: 1; a force instability in tension, ie. the necking instability, and 2; a crack nucleation or propagation instability where the imposed stress concentration at an existing flaw exceeds the critical toughness value of the material. This paper will review evidence for ductility, including superplasticity, of nc materials from the recent literature and the author's laboratory with regard to the two failure criteria cited above. Authors research supported by DMR-NSF, grant number 9871980 with Dr.Bruce MacDonald as program monitor.

8:55 AM Invited
Surface and Interfacial Energy Effects in Nanocrystalline Materials: Some Consequences for Materials Processing and Properties: *Merrilea J. Mayo*[1]; [1]Pennsylvania State University, Dept. Mats. Sci. & Eng., Rm. 115 Steidle, University Park, PA 16802 USA

Grain boundary and surface energies in solid materials are often not large–on the order of 1 J/m^2. However, in a 10 nm grain or particle, the boundary tension results in an internal compressive stress that can be on the order of 200 MPa. These large pressures increase the free energy of the system, thereby skewing the thermodynamic equlibria that depend on free energy. This presentation links the thermodynamic free energy changes, derived from fundamental relations, to macroscopic phenomena such as increased solubility for nanoparticles in suspension and shifts in phase stability towards denser phases. The practical consequences of the skewed thermodynamics are also presented, using nanocrystalline zirconia as an example system: ceramic nanoparticles that dissolve in water, unexpected phases obtained on synthesis, and factors of 5 change in fracture toughness.

9:20 AM
Role of Interstitials in Deformation of Nanocrystalline Nickel: *S. H. Whang*[1]; *W. M. Yin*[1]; [1]Polytechnic University, Dept. of Mech. Eng., Six MetroTech Cen., Brooklyn, NY 11201 USA

Nanocrystalline metals with grains of 30 nm or less may be characterized by their high tensile strength, low elongation and low temperature creep, which is a typical exhibition of dominant grain boundary deformation in this material. Consequently, the plastic deformation is mainly carried out by grain boundary migration and sliding. In addition, the deformation is sensitive to strain rate and temperature, which means an important role of dynamic deformation in this class of materials. The creep in nanocrystalline nickel at room temperature was identified as having a Coble type mechanism while the creep above room temperature is no longer a simple Coble type. A small amount of interstitials such as boron, carbon, sulfur, etc has a great influence on the tensile and creep behavior. In this presentation, we will review some of the results on deformation, creep and grain growth in nano-nickel containing B, C, S, etc. An attempt will be made to analyze the effect of interstitials on the deformation based on a phenomenological approach.

9:40 AM
Mechanical Properties and Deformation Modes of Bulk Nanophase Iron: D. Jia[2]; K. T. Ramesh[2]; *Evan Ma*[1]; [1]Johns Hopkins University, Mats. Sci. and Eng., 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Johns Hopkins University, Mech. Eng., Baltimore, MD 21218 USA

Full density Fe with grain sizes in the nanophase to submicron range has been consolidated from mechanically milled powders. The deformation behavior of such materials, as a function of grain size, strain rate, and temperature, have been studied using quasi-static and high strain rate (Kolsky bar) tests. With ultrafine grain sizes, Fe exhibits high strength, little work hardening, and plastic strains localized in shear bands. Shear banding appears to be the dominant mode from the onset of the plastic deformation in our consolidated materials. Little strain rate sensitivity of the flow stress is observed over a wide strain rate range (up to 5E5/s). These behaviors are contrasted with those of conventional bulk Fe, which shows uniform deformation, significant work hardening, and strong strain rate sensitivity. The underlying deformation and failure mechanisms are discussed based on these observations. With increasing grain sizes and/or temperature, the yield strength decreases and plastic strain increases. These findings are compared with the Hall-Petch relationship, and examined to derive the validity range, in terms of grain size, temperature, strain rate, and particle bonding strength, of different deformation mechanisms (including grain boundary mechanisms). In addition, we comment on the potential advantage of related bcc alloys in military kinetic energy penetrators where the shear banding mode and self-sharpening capability during high rate deformation are desired.

10:00 AM Break

10:15 AM
R-Curve Characterization of the Fracture Toughness of Nanocrystalline Nickel Thin Sheets: *R. A. Mirshams*[2]; C. H. Xiao[1]; S. H. Whang[3]; W. M. Yin[3]; [1]Southern University and A&M College, Baton Rouge, LA 70813 USA; [2]University of North Texas, Denton, TX 76203 USA; [3]Polytechnic University, Six Metrotech Ctr., Brooklyn, NY 11201 USA

The fracture resistance curves of nanocrystalline nickel and carbon doped nanocrystalline nickel in different annealing temperatures have been generated and studied. The results indicate that crack growth resistance of pure nanocrystalline nickel is very sensitive to annealing temperatures. The crack growth resistance decreased with increasing annealing temperature for the nanocrystalline nickel. Carbon doping greatly reduces crack growth resistance of nanocrystalline nickel. However, the crack growth resistance of carbon-doped nanocrystalline shows improvement through annealing

processing. A cluster model was effectively used to explain the crack growth resistance behavior of nanocrystalline nickels.

10:35 AM

Anelastic Deformation in Nanostructured Nickel: *W. M. Yin*[1]; S. Y. Kim[1]; *S. H. Whang*[1]; [1]Polytechnic University, Dept. of Mech. Eng., Six MetroTech Center, Brooklyn, NY 11201 USA

When the grain size in nanostructured nickel decreases to near single digit nano-sizes, a considerable anelastic deformation has been observed probably due to a significant volume fraction of intercrystalline components. Also, such anelasticity is not only a function of time, but also sensitive to temperature. To understand the anelasticity in this material, cyclic tension tests have been conducted with different loads and cyclic periods. In each cyclic deformation, an elastic hysteresis has shown a relatively large anelasticity. In addition, an anelastic recovery was observed from the interrupted creep test at 373K. The results showed that the anelastic strain of nanostructured nickel recovered gradually after unloading during the steady state creep. But, the anelastic strain is negligible at room temperature. Furthermore, polycrystalline nickel did not show any time-dependent recovery of creep strain. The deformed specimens from the various tests will be investigated by TEM and the results will be reported. The anelasticity mechanism in nanostructured nickel will be discussed.

10:55 AM

Strength and Fracture of Ni/Cu Laminated Nano-Structures: *Alirio J. Liscano*[1]; Fereshteh Ebrahimi[1]; [1]University of Florida, Matls. Sci. and Eng. Depts., P.O. Box 116400, 141 Rhines Hall, Gainesville, FL 32611 USA

In this study Cu/Ni laminated nano-structures were produced by electrodeposition method. Deformation and failure of the Cu/Ni nanolaminated structures were evaluated by tensile testing. The multilayers produced in this investigation broke without gross plasticity. The load-displacement curves of some of the specimens demonstrated linear elastic behavior, while others exhibited slow crack growth prior to final failure. The study of fracture surfaces using SEM revealed the presence of two distinct zones: one brittle and the other ductile. A detailed analysis of the microstructure of deposits in cross-section revealed that the brittle fracture zones are associated with porous regions. Microprobe analysis established that these regions have much lower copper concentrations than the dense regions, suggesting a low efficiency during copper deposition. It is concluded that owing to hydrodynamic effects, copper ions become depleted near the deposition front and cause hydrogen evolution, which forms nano-size bubbles at Ni/Cu interfaces and twin boundaries.

11:15 AM

Nanophase Aluminide Intermetallics: *Robert A. Varin*[1]; Tomasz Czujko[2]; Jerzy Bystrzycki[2]; [1]University of Waterloo, Dept. Mech. Eng., 200 University Ave.W., Waterloo, Ontario N2L 3G1 Canada; [2]Military University of Technology, Dept. of Matls. Tech., Kaliskiego 2, Warsaw 00-908 Poland

Nanophase (nanocrystalline)intermetallics have increasingly attracted interest with the expectation that some of their detrimental characteristics such as lack of ductility/fracture toughness could be alleviated by nanograin size while hardness/strength could increase according to the Hall-Petch relatinship. In this work, nanophase intermetallic powders of B2 FeAl and $L1_2Al_3Ti$(9at%Mn)(iron aluminides and titanium trialuminides, respectively)were processed by ball milling under low energy shearing mode. Also, bulk intermetallic compacts were fabricated by both hot compaction and shock-wave consolidation. The microstructure of powders and bulk compacts, their lattice parameter and degree of long-range order (LRO), were studied by optical/scanning microscopy and X-ray diffraction. Mechanical properties were studied by Vickers microhardness. Iron aluminides exhibited diverse magnetic behavior depending on their microstructural condition which was studied by the measurements of the force of magnetic attraction. The indentation fracture toughness testing was conducted on bulk compacts. The results of these studies will be presented and discussed.

11:35 AM

Mechanical Properties of Nano-Scale Copper Thin Films on Silicon Substrates: *Seyed Allameh*[1]; A. Butterwick[1]; Zhigang Suo[1]; B. S.H. Royce[1]; A. G. Evans[1]; W. O. Soboyejo[1]; [1]Princeton University, Princeton Mats. Inst., Dept. of Mech. & Aerosp. Eng., Olden St., Princeton, NJ 08544 USA

This paper presents experimental approaches to the measurement of thin film mechanical properties of the nano-scale thin films. These include: cantiliever bending techniques for the measurement of elastic modulus; cantilever vibration methods for the measurement of the elastic moduli of thin films, and micro-/nano-indentation methods for the characterization of strength. The length-scale issues associated with the measurement of modulus and strength are examined within the context of mechanics models. The relationships between the microstructure and mechanical properties are discussed before exploring the implications of the results for the multi-scale modeling of deformation between the nano- and macro-scales.

11:55 AM

Microstructure and Properties of Ultrafine-Grained Pure Ti Processed by ECAP and Cold Deformation: *Yuntian T. Zhu*[1]; Vladimir V. Stolyarov[2]; Terry C. Lowe[1]; Ruslan Z. Valiev[2]; [1]Los Alamos National Laboratory, Mats. Sci. and Techn. Div., MS G755, Los Alamos, NM 87544 USA; [2]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Mats., K. Marksa 12, Ufa 450000 Russia

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

Reactive Metals - General Sessions

Sponsored by: Light Metals Division, Reactive Metals Committee

Program Organizers: John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Sean M. McDeavitt, Argonne National Laboratory, Chemical Technology Division, Argonne, IL 60439-4837 USA

Tuesday AM	Room: 212
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Session Chair: TBA

8:30 AM

The Properties of Phosphide in Rare Earth Silicide Alloy Prepared by Carbon Thermal Reduction of RE Concentration: *Zhao Qun*[1]; [1]Northeastern University, Sch. of Mat. and Metall., Shenyang, Liaoning 110006 PRC

The carbon thermal reduction process of rare earth concentration was briefly introduced and the behavior of P in the process was generally investigated. SEM and other micro-method studied the microstructure and composition of the product-rare earth silicide alloy, especially the phosphide in the alloy and its contribution to the disintegration of the alloy in detail. The waterized experiment of the alloy was done and the gases escaped from the alloy was collected and analyzed by quality and quantity method. As a result, we

found that only a little phosphide in the alloy can react with water and the gas of PH3 was given off, the microstructure of the alloy can greatly affect its disintegration property.

8:55 AM

Electro-Deoxidation of Metal Oxides: George Z. Chen[1]; *Derek J. Fray*[1]; [1]University of Cambridge, Dept. of Mats. Sci. and Metall., Pembroke St., Cambridge CB2 3QZ UK

Many metals, especially those in groups 4-6 of the periodic table, are prepared by the reduction of a compound of the metal by a more reactive element. In this paper, we report a method whereby many of the oxides of these metals can be reduced by making the the oxide the cathode in a fused bath of calcium chloride. On the application of a voltage, below the decomposition voltage of the salt, the favoured cathodic reaction is the ionisation of the oxygen to form an oxygen ion which dissolves in the salt. The cathode gradually transforms to the pure metal. Results are presented for the reduction of many of oxides of the group 4-6 elements. The general form of the product is a high purity porous solid of particle size around 12 micron,depending upon the temperature of reduction and the particle size of the starting material.

9:20 AM

A Comparison of the Sintering Behavior Titanium Powder Produced by International Titanium Powder and Commercially Available Titanium Powders: *Stephen James Gerdemann*[1]; David Alman[1]; [1]Albany Research Center-DOE, Therm. Treat. Tech., 1450 Queen Ave SW, Albany, OR 97321 USA

The sintering behavior of CP Ti and Ti 6-4 alloy powder produced by International Titanium Powder (ITP) was characterized. Green specimens were die pressed into cylindrical compacts and vacuum sintered. The influence of green density, sintering temperature and sintering time on the final density and microstructure was evaluated. These results are compared to commercially available -325, -200, -100 mesh titanium powder and Ti 6Al 4V alloy powder. Near-net-shape tensile bars were pressed and consolidated using the determined optimal sintering conditions, and room temperature tensile properties were measured. The results are compared to the ASTM standards and to the same material melted into buttons and machined into tensile bars.

9:45 AM

Compatibility of Molten Beryllium With Ceramics: *Robert Joseph Hanrahan*[1]; [1]Los Alamos National Laboratory, MST-6, TA3 MS G770, Los Alamos, NM 87545 USA

A series of experiments was performed in order to test the compatibility of molten beryllium with various ceramic materials (other than BeO). The materials tested include Al2O3, Y2O3 MgO, ZrO2, SiC and fused silica. We have evaluated the reaction products and to some extent the mechanism of reaction of beryllium with each of these materials. In the case of alumina, the apparent compatibility may be attributed to the formation of an intermediate BeO-Al2O3 phase. In the case of magnesia, yttria, and zirconia the oxides may be reduced by beryllium resulting in formation of intermetallic phases and beryllium oxide. The SiO2 and SiC are reduced to silicon. The significance of these results with regard to the published thermodynamic data will be discussed.

10:10 AM Break

10:25 AM

An Experimental and Numerical Investigation of the Initiation: *Robert Joseph Hanrahan*[1]; [1]Los Alamos National Laboratory, MST-6, TA3 MSG770, Los Alamos, NM 87545 USA

An experimental and numerical investigation was undertaken to evaluate the initiation of uranium hydride. It has been found that the nucleation and growth rate of the hydride depends on: the local texture, the local strain field, hydrogen diffusion, and the strength of the material. The growth of hydride requires that the uranium rupture to allow the hydride to expand. Hydride expansion work-hardens the surrounding matrix thereby constraining lateral growth. A 2-dimensional finite volume model was used to incorporate the features observed in experiments. The hydride nuclei were randomly placed in the computation domain with each nucleus having either a fixed or variable activity. Local hydride growth was constrained by using a hydride concentration-stress relationship to account for strength and work-hardening effects. Initial strength and work-hardening have a dramatic effect, shifting the hydride formation from localized pitting only to a near uniform attack, which parallels the results seen in experiments.

10:50 AM

Mechanical and Magnetostrictive Properties of (Tb,Dy)Fe2-(Tb,Dy) In-situ Composites: *Won Je Park*[1]; Zin Hyoung Lee[1]; [1]Korea Advanced Institute of Science and Technology, Mats. Sci. and Eng., Gusong-dong 373-1, Yusong-gu, Taejon 305-701 Korea

The giant magnetostrictive material Terfenol-D is very brittle and its application is limited by the brittleness, despite of its superior magnetostrive properties. To enhance the toughness of this smart material, composites of brittle RFe2 phase and ductile pure RE phase was fabricated by directional solidification. The ingot of desired composition was cast in a rod with 11mm in diameter and 100mm in length and was sealed in a quartz ampoule in Ar gas atmosphere. By varying the amount of RE phase and the solidification conditions such as growth rate and temperature gradient, solidification microstructures were investigated. Solidification parameters for the coupled eutectic growth were also investigated. Mechanical properties was evaluated by tensile test at room temperature and magnetostrictive properties by LVDT and fluxmeter. The relations of mechanical and magnetostrictive properties with microsturctures were investigated.

11:15 AM

Benefication of Rare Earth Ore in China: Ru'an Chi[1]; *Shengming Xu*[1]; Guocai Zhu[1]; Jingming Xu[1]; Xin Qiu[1]; [1]Tsinghua University, Inst. of Nuc. Engy. Techn., Beijing 102201 China

The paper briefly introduces the characteristics and the distribution of rare earth resources in China, the existing states of rare earth in ore and the main deposits and minerals. It centers on discussing of the recent development and the practical application of beneficiation and extraction technology on Chinese rare earth minerals, and finally put forward the problems of Chinese rare earth exploit and the countermeasures to solve those problems.

Sampling, Sensors & Control for High Temperature Metallurgical Processes: Ferrous Processing, Cupola Operation, and Vacuum Arc Remelting

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

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

Tuesday AM — Room: 230
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Derek Fray, University of Cambridge, Dept. of Mats. Sci. and Metall., Cambridge CB2 3QZ UK; Markus Reuter, TU Delft, Delft, The Netherlands

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8:30 AM

Sensing of Sulphur in Molten Iron Using Strontium Beta Alumina: Mark A. Swetnam[1]; R. Vasant Kumar[1]; *Derek J. Fray*[1]; [1]University of Cambridge, Dept. of Mats. Sci. and Metall., Pembroke St., Cambridge CB2 3QZ UK

A sulphur sensor based upon strontium beta alumina was developed and tested in molten iron. The strontium beta alumina sold

electrolyte was fabricated using injection molding and optimised for thermal shock resistance and toughness by including up to 20 wt % monoclinic zirconia within the electrolyte phase. A mixture of molybdenum and molybdenum sulphie was used as the reference electrode, while strontium sulphide, allowed to form in-situ during sulphur sensing, formed the auxiliary electrode. The sensor behaviour in terms of response time, life time to failure and possible reaction mechanisms are discussed. The results were in good agreement with the LECO analysis.

8:55 AM

Infrared and Laser-Based Sensors and Systems to Monitor Accurately 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, 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.

9:20 AM

Balanced Blast Application for Cupola Operation Improvement: *Robert Allen Medower*[1]; [1]The Foxboro Co., Mets. & Min. Proc., 6887 Stonewood Court, Eden Prairie, MN 55346 USA

This paper will explore the potential benefits of balancing cupola blast. Reported benefits include reduced channeling, higher pour temperature, reduced emissions and improved control of blast rate.

9:45 AM Break

10:05 AM

Optimal Filtering Applied to the Vacuum Arc Remelting Process: *Rodney L. Williamson*[1]; Joseph J. Beaman[2]; David K. Melgaard[1]; [1]Sandia National Laboratories, Dept. 01835, Mail Stop 1134, P.O. Box 5800, Albuquerque, NM 87185-1134 USA; [2]University of Texas, Mech. Eng. Dept., Austin, TX 78712 USA

Optimal estimation theory has been applied to the problem of estimating process variables during vacuum arc remelting (VAR), a process widely used in the specialty metals industry to cast large ingots of segregation sensitive and/or reactive metal alloys. Four state variables were used to develop a simple state-space model of the VAR process: electrode gap (G), electrode mass (M), electrode position (X) and electrode melting rate (R). The optimal estimator consists of a Kalman filter that incorporates the model and uses electrode feed rate and measurement-based estimates of G, M and X to produce optimal estimates of all four state variables. The filter provides estimates that have error variances between one and three orders of magnitude less than estimates based solely on measurements, allowing for significantly improved process control as demonstrated by both laboratory and industrial test results.

10:30 AM

The Use of Data Reconciliation as a Soft Sensor for the Control of Metallurgical Reactors: *Markus A. Reuter*[1]; [1]TU Delft, Mijnbouwstraat 120, Delft 2628 RX The Netherlands

The use of data reconciliation is discussed for the modelling and control of metallurgical reactors and processes. The results of the data reconciliation are applied to (i) calibrate black-box models such as ARMAX models and neural nets, (ii) calibrate grey-box models, (iii) create soft sensors for the prediction of various relevant process variables for process control, and (iv) create models for the prediction of waste and intermediate streams in view of environmental control. Various industrial examples will be discussed including Zn, Pb, Sn, Cu, P, Mn and steel production.

10:55 AM

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

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

Second Global Symposium on Innovations in Materials Processing & Manufacturing: Sheet Materials: Properties and Applications

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

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

Tuesday AM — Room: 228
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Jian Cao, Northwestern University, Evanston, IL 60208 USA; Andy Sherman, Ford Motor Company, Dearborn, MI USA

8:30 AM

Ultra High Strength and Stainless Steels for Automotive Applications: *Mahmoud Y. Demeri*[1]; [1]Ford Motor Company, Ford Res. Lab., 2101 Village Rd., P.O. Box 2053, MD 3135, Dearborn, MI 48121 USA

The need to address environmental concerns, abide by government regulations, increase crash safety and improve customer satisfaction has placed new emphasis on improving energy efficiency and fuel economy of cars and trucks produced for the US auto market. Fuel economy improvements could be achieved through the combined effects of weight saving, aerodynamic streamlining, friction reduction and powertrain enhancements. Significant weight saving appears to be the major factor in improving fuel economy and reducing carbon dioxide emissions. Reduction of vehicle weight can be achieved by replacing parts made from mild steel with those made from thinner gages of high strength steels. This paper reviews the various types of Ultra High Strength & Stainless Steels, discusses the advantages and limitations of using them and lists vehicle parts that can be targeted for safe and lightweight applications in the automotive industry.

8:50 AM

Using Stainless Steel for Energy Absorbing Components in Automotives: *Roger Andersson*[1]; Claes Magnusson[2]; [1]Luleå University of Technology, Manufact. Sys. Eng. Div., Luleå SE-971 87 Sweden; [2]Volvo Car Corporation, Body Comp. Div., Olofström SE-293 80 Sweden

To increase the crash performance in automotives it is necessary to use new techniques and materials. To produce energy absorbing components the material should have high yield strength, high elongation to fracture and strong work hardening. The total work a component absorbs during an impact is the area under the stress-strain curve for unit material volume. This has lead to an interest in high strength stainless steels as crash safety components in automotives due to their excellent material properties. The plastic performance and crash impact behaviour of different stainless grades has been evaluated through intrinsic and simulative tests. A stainless steel bumper beam has been optimised for a VOLVO car and comparisons have been made with the present application. Simulations have been done and verified by experiments.

9:10 AM

Forming Behaviour of Isotropic Steel Sheets: *Klaus Siegert*[1]; Necdet Dogan[1]; Klaus Freier[2]; [1]University of Stuttgart, Instit. for Met. Form. Techn., Holzgartenstrasse 17, Stuttgart 70174 Germany; [2]Salzgitter AG, Res. & Dev., Eisenhuettenstrasse 99, Salzgitter 38239 Germany

The demand of the automotive industries to reduce the fuel consumption and therefore the weight of vehicles are becoming more and more significant. Reduction of vehicle weight can be realized, among other factors, by building lighter car bodies. Particularly, the reduction of the sheet metal thickness in car bodies can provide a significant contribution to solving this problem. In this case, the use of high-strength steel is necessary. Isotropic high-strength steels, the so-called I-Steels (e.g. St250i), have been developed in Germany. Such steel sheets showed direction-independent properties and exhibited a very good deep drawing behavior. With these I-Steels, interior components as well as exterior panels can be produced. In this paper, the isotropic characteristics of high-strength I-Steel sheets are compared with a reference deep drawing quality material (DC04) having a similar strain hardening coefficient(n) and microsurface structure.

9:30 AM

Production and Engineering Use of Magnesium Sheet: *Gerald S. Cole*[1]; [1]Ford Motor Company, Ford Res. Lab., P.O. Box 2053, MD 3135, Dearborn, MI 48121-2053 USA

Only 5% of the 90,000 MT of magnesium used in NA is not die cast; there are no sheet products used. If sheet cost could be reduced, formability increased and corrosion-protection solved, there could be new applications, especially for the new hybrid vehicles being developed, where mass is a critical functional attribute. This paper analyzes current sheet cost and will examine the potential for cost-reduction compared to other light weight materials, polymers, steels and aluminum. Current magnesium corrosion-protection modalities will be examined in terms of their applicability to formed sheet products. Finally, new manufacturing methods to form sheet products will be described that could significantly improve the commercial applicability of magnesium sheet in the automotive industry.

9:50 AM Break

10:10 AM

Integration of Optical Coordinate Measuring System into Sheet Metal Manufacturing: *Mumin Song*[1]; *Frank Chen*[1]; [1]Ford Research Laboratory, Dept. of Manuf. Sys., 2101 Village Rd., MD3135, Dearborn, MI 48121 USA

Various coordinate measuring systems were used to measure dimensional quality in stampings. Such systems were considered to have a complementary rather than an essential role in the overall manufacturing of sheet metal components. This role resulted from performance limitations of the coordinate measuring systems and from the lack of efforts to integrate them into the manufacturing environment. Attempts have been made to develop efficient coordinate measuring systems and to integrate them into every aspect of the manufacturing cycle from manufacturing feasibility to part quality. This paper will first review the historical development of the various coordinate measuring systems and will discuss the pros and cons of using them. It will also present the current efforts in developing an efficient optical coordinate measuring system. Finally, it will introduce an integrated manufacturing system layout with the optical measuring system performing essential rather than complementary roles.

10:30 AM

Speed Effects in Drawing Al-Zn Alloy Sheets: *A. Sherif El-Gizawy*[1]; *Tai-Kun Yeh*[1]; *Herwin Ont*[1]; [1]University of Missouri-Columbia, Mech. and Aero. Eng., E3412 Eng. Bldg. E., Columbia, MO 65211 USA

Punch speed in deep drawing operations has direct influence on the process behavior and the quality of sheet products. These effects are due to changes in process dynamics and the local interface condition between the sheet and the tool. In the present work, a finite element simulation model is developed to characterize the effects of speed on the process behavior and on the post forming shape distortion. An industrial scale experimental model is also established to verify the results of the numerical simulation. The effects of six different punch speed profiles with same peak value are investigated.

10:50 AM

Constitutive Relationships for the Hot Working of AA 3004 (A1-1. OMn-1.2Mg): *Hugh J. McQueen*[1]; J. Belling[1]; [1]Concordia University, Mech. Eng. Dept., 1455 Maisonneuve W., Montreal, Quebec H3G 1M8 Canada

Recrystallized plate of 3004 (0.96%Mn, 1.23%Mg, 0.37Fe) was subjected to hot torsion tests in the ranges 250 to 500°C and 0.1 to $10s^{-1}$. The flow curves strain hardened to a broad peak and softened slightly towards a steady state regime. At higher temperature, T and lower strain rate ε the maximum stress σ and the softening were lower and the fracture strains were higher. The exponential law was found satisfactory, but the power law was not. The constants A, n, Q_{HW} for the equation: $A(\sinh \alpha\sigma)^n=\varepsilon \exp (Q_{HW}/8.31\ T)$ were derived for α ranging from 0.01 to 0.08 MPa^{-1}. In the ε dependence on σ, n varies almost inversely with α, whereas in the Arrhenius plot, the slope varies almost linearly with α; Q_{HW} is almost constant for α in the range 0.03 to 0.08 MPa^{-1}. The constants differed slightly for longitudinal and transverse specimens. The use of α=0.04 or 0.06 MPa^{-1} makes comparison with published data simpler; the agreements were reasonable given the variations in composition. In general, the behavior of 3004 is intermediate between 5005 and 5182. Optical microscopic examination revealed elongated grains in which the subgrains became more clearly defined as T rose and ε diminished. Clearly dynamic recovery provided good hot workability through a reduced strain-energy, particle-stabilized substructure which also inhibited recrystallization unless the product was heated to about 50° above the deformation temperature as previously published.

11:10 AM

Yield Criterion for Orthotropic Sheet Metals: *Dorel Banabic*[1]; [1]University of Stuttgart, Insti. for Met. Form. Techn., Holzgartenstrasse 17, 70174 Stuttgart, Germany

The paper presents a new yield criterion for orthotropic sheet metals under plane-stress conditions. The criterion is derived from the one proposed by Barlat and Lian in 1989. Two additional coefficients have been introduced in order to allow a better representation of the plastic behaviour of the orthotropic sheet metals. The predictions of the new yield criterion are compared with the experimental data for two materials.

Structural Biomaterials for the 21st Century: Surface Modifications and Environmental Effects

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 Engineering Department, Austin, TX 78712-1063 USA

Tuesday AM Room: 229
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Eric Taleff, University of Texas, Mech. Eng. Dept., Austin, TX 78712-1063 USA

8:30 AM Keynote

Micro-Chemical and Mechanical Properties/Structure Relationships at the Dentitional Tissues-Adhesive-Composite Interfaces: *J. Lawrence Katz*[1]; Tsutomu Nomura[2]; Ajay Wagh[1]; Paulette Spencer[3]; Yong Wang[3]; [1]Case Western Reserve University, Biomed. Eng., Cleveland, OH 44106-7207 USA; [2]Case Western Reserve University, Sch. of Dent., Cleveland, OH 44106 USA; [3]University of Missouri-Kansas City, Sch. of Dent., Kansas City, MO USA

Micro-Raman and Scanning Acoustic Microscopy (SAM) have been used to study the dentitional tissues interfaces with adhesives and composites. SAM was used in the burst mode at 400 MHz (nominal lateral resolution 2.5 μm) to study the micromechanical properties of the dentitional tissues-adhesive-composite interfaces in vitro in the same portion of excised teeth studied with μ Raman, optical and scanning electron microscopy. A calibration curve, to obtain elastic modulus values from the acoustic impedance values for the dentin-interface-adhesive-composite components obtained with the SAM, was developed using a number of known materials providing a range of acoustic impendances. Elastic modulus for dentin ranged from 13 GPa (partially demineralized) to 28 GPa; for the adhesive 5.0 GPa; and for the interface, less than 2.0 GPa. The combination of microscopy, μ Raman and SAM is especially synergistic in that it provides the means for relating structure, chemical condition, e.g. degree of polmerization, and mechanical properties at interfaces.

9:00 AM

Titanium Surface Modification with Dry Process for Dental Implants: *Masao Yoshinari*[1]; Toshio Igarashi[2]; Yutaka Oda[2]; [1]Tokyo Dental College, Dept. of Dent. Matls. Sci. and Oral Hlth. Sci. Ctr., 1-2-2 Masago Mihama-ku, Chiba City, Chiba 261-8502 Japan; [2]Tokyo Dental College, Dept. of Dental Mats. Sci., 1-2-2 Masago Mihama-ku, Chiba, 261-8502 Japan

Since dental implants are used in contact with various tissues, it is necessary to have optimum surface compatibility with the host bone tissues, subepithelial connective tissues, and epithelial tissues. Furthermore, dental implants are required to remain plaque-free at the surface exposed to the oral cavity. Such materials can be created under well-controlled conditions by modifying the surfaces of metals that are in contact with those tissues. Tissue-compatible implants, which are compatible with all host tissues, have to integrate with bone tissues, easily form hemidesmosomes, and prevent the bacterial adhesion. This paper summarizes the research work aimed at developing the tissue-compatible implants by modifying titanium surfaces using a dry process for closely adhering to titanium substrate and ensuring a good wear resistance, including the ion beam dynamic mixing (thin calcium phosphates), ion implantation (Ca, N, F), titania spraying, ion-plating (TiN, alumina), and ion beam mixing (Ag, Sn, Zn, Pt) with Ar.

9:20 AM

Characterization of Low Temperature Formed Hydroxyapatite Coating on Chemically Modified Titanium Surfaces: *Russell S. Wang*[1]; U. Sampathkkulumaran[1]; D. Lennon[1]; A. Caplan[1]; [1]Case Western University, Dept.of Restor.. Dent., Sch. of Dent., 10900 Euclid Ave., Cleveland, OH 44106-4905 USA

Titanium (Ti) coated with hydroxyapatite (HA) via plasma-spray has been controversial for clinicians. Despite the extensive use of the plasma sprayed HA coatings on orthopedic and dental implants, limitations of the plasma spray process are widely recognized. A type of synthetic organic surfaces, called a self-assembled monolayer (SAM), to mimic natural biomineralizing organic surface, was used to chemically modify the Ti substrate. HA was nucleated from a simulated body fluid solution (SBF) at 37 degrees, by placing the substrate surface opposed to granular bioglass. In situ HA growth was sustained by immersion of the substrates in more concentrated SBF. Low temperature formed HA coatings on Ti substrate were analyzed by x-ray diffraction, scanning electron microscopy. Also, characterization of the interactions between SAM coated Ti and human bone marrow stromal cells in vitro was studied. Three samples groups were prepared: Ti coated with HA by SAM; Ti coated HA by plasma spray; and Polished Ti without HA coating. Cell attachment assay, cell growth kinetic assay and bone nodules formation assay were conducted. We conclude that HA coatings on Ti by SAM method may have clinical validity to process Ti for future use in medical and dental implants.

9:40 AM

Biocompatability Studies Using Cell Culture Testing of Ti40Ta and Ti50Ta Alloys for Implant Consideration: *Rudy A. Villa*[1]; Celina Ortiz[1]; Elizabeth A. Trillo[1]; Stephen W. Stafford[1]; Larry E. Murr[1]; [1]The University of Texas at El Paso, Metall. Dept., 500 University, El Paso, TX 79968 USA

The TiTa series alloys have recently been of great interest for the use as orthopedic (surgical) implants. In this research cell culture tests are being conducted to examine cell adhesion and cell morphology of epithelial cells in vitro on the polished and rough surface Ti, Ta, Ti6Al4V, Ti40Ta, and Ti50Ta specimens. Fixation and critical point drying was performed to prepare the cell culture samples for microscopy. Optical microscopy was employed to determine surface structure of metal surfaces. Pure Ti, Ta, and Ti6Al4V displayed a small fine grained homogeneous microstructure. The Ti40Ta and Ti50Ta samples contained an Ñ phase in an Ñ+Ò matrix. Fine bands of martensite were also observed in both alloys. SEM microscopy was utilized to visually examine the morphology and cell behavior on the alloys. This project was funded by General Services Administration (GSA) Grant Project #PF90-018.

10:00 AM Break

10:10 AM Keynote

Metallic Biomaterials in Body Fluid and Their Surface Modification: *Takao Hanawa*[1]; Sachiko Hiromoto[1]; Akiko Yamamoto[1]; Norio Maruyama[1]; Kozo Nakazawa[1]; [1]National Research Institute for Metals, Biomatls. Rsch. Team, 1-2-1 Sengen, Tsukuba 305-0047 Japan

The surface properties of metallic materials used for biomedical devices such as artificial joints, bone plates, and dental implants are discussed based on empirical data of stainless steel, cobalt-chromium alloy, and titanium, focusing on the reconstruction of the surface oxide film by the interaction between the surface and chemical species in the body. In particular, calcium phosphate precipitation on the surface and its rate characterized using X-ray photoelectron spectroscopy and quartz crystal microbalance. This is one of the causes of biocompatibility of the materials. Preferential dissolution of component elements from alloys during wear and fretting fatigues tests, influencing the toxicity of the alloys, is also discussed. In addition, surface modification of titanium to improve bone conduction is reviewed. Apatite coating and non-apatite coat-

ing techniques are studied. Effect of calcium ion mixing into titanium is given as an example.

10:30 AM

Corrosion Resistance and Strength of BioDur®108 Alloy, a Nickel-Free Austenitic Stainless Steel: *Ronald C. Gebeau*[1]; Robert S. Brown[1]; [1]Carpenter Technology Corporation, Carp. Steel Div., P.O. Box 14662, Reading, PA 19612-4662 USA

BioDur®108 Alloy is an essentially nickel-free austenitic stainless alloy developed by Carpenter Technology Corporation in response to nickel-allergy problems that have been associated with nickel-containing stainless alloys used in medical applications. The austenitic structure of this alloy is maintained by a high nitrogen content, approximately 1%. Besides austenitic stability, the high nitrogen content also contributes to high levels of corrosion resistance and strength. The strength and corrosion resistance of BioDur 108 Alloy will be compared to nickel-containing austenitic stainless alloys that have been used as biomaterials. In such alloys, strength tends to be dominated by nitrogen content, while corrosion resistance is strongly related to the chromium, molybdenum and nitrogen contents. However, the quantitative effect of nitrogen on corrosion resistance in high nitrogen alloys is significantly different than in alloys with lower nitrogen levels. The manganese content also has an effect, particularly on the passive current density measured in potentiodynamic corrosion testing at rapid scan rates.

10:50 AM

Improved Wear and Corrosion Performance of Nitrogen Ion Implanted Titanium Alloys for Medical Implants: *Shinji Fukumoto*[1]; Kaoru Nakamura[2]; Harushige Tsubakino[1]; Yoshimitsu Okazaki[3]; Mititaka Terasawa[1]; Toru Mitamura[1]; [1]Himeji Institute of Technology, Fac. of Eng., 2167 Shosha, Himeji, Hyogo 671-2201 Japan; [2]Graduate School of Himeji, Insti. of Techn., 2167 Shosha, Himeji, Hyogo 671-2201 Japan; [3]Mechanical Engineering Laboratory, 1-2 Namiki, Tsukuba, Ibaraki 305-8564 Japan

Nitrogen ion implantation was carried out on the titanium alloys which were developed for medical implants to improve their wear and corrosion resistance. The titanium nitride was identified by a grazing incidence X-ray analysis. The coefficient of dynamic friction of ion implanted titanium alloy was kept lower than that of substrate until the nitride layer was broken. The wear volume was decreased by ion implantation. The open circuit potential of implanted titanium alloy was more noble in comparison with unimplanted one. The passive current density of implanted titanium alloy was lower than unimplanted one in physiological saline solution of PBS(-). Moreover, the wear resistance in PBS(-) of ion implanted titanium alloy was improved. Multi-implantation which titanium alloys were implanted successively at 3 energies with the dose in the various ratio was carried out. The wear and corrosion resistance were more improved in comparison with normal ion implantation.

11:10 AM

Hydrogen Embrittlement of Ni-Ti Alloy in a Biological Environment: *Kenzo Asaoka*[1]; Ken'ichi Yokoyama[1]; [1]The University of Tokushima, Sch. of Dent., 3-18-15 Kuramoto-cho, Tokushima 770-8504 Japan

It is known that Ni-Ti super-elastic alloy is susceptible to environmental embrittlement in a corrosive atmosphere. Diffusion rate of hydrogen is thought to be one of the deciding factors of service life. Accelerated testing of hydrogen embrittlement was carried out; that is, alloy samples with a diameter of 0.56 mm were charged with hydrogen using an electro-chemical system. The alloys with charging of 1 A/m^2 and 10 A/m^2 for 24 h and 120 h, respectively, were tested. Hardness numbers in cross-sectional area and amount of absorbed hydrogen in the charged alloys were measured. Distribution of hydrogen concentration was computed for the infinite cylinder model by theoretical differential equation. If the hardness is linear with the concentration of hydride and/or hydrogen, the diffusion constant of hydrogen into the Ni-Ti alloy could be estimated as 1.4-1.8 x 10^{-14} m^2/s. Experimental results of the hardness and occlusion of hydrogen support the estimated diffusion constant.

11:40 AM

Friction and Wear Behavior of Diamond Films Compared with Bulk Diamond: *Ali Soleman Al-Watban*[1]; [1]Riyadh Technical College, P.O. Box 53699, Riyadh 11593 Saudi Arabia

Growing interest in polycrystalline diamond (PCD) films and Diamondlike carbon (DLC) films has been stimulated by the expectation that these materials could possess the advantageous properties of bulk diamond. Coated diamond and diamondlike films have great application in biomedical instrumentation sector. They can be used as optical, friction-reducing, corrosion-preventing and wear-resistant coatings. Since the properties of synthesized materials are in large measure determined not only by their compositions, but also by the method of processing and their resultant microstructure, it is of interest to compare the tribological properties of PCD and DLC films with those of natural diamond. Most of the tribological studies have indicated the strong effect of several scales of surface roughness on friction. For example, the sliding friction coefficients of certain PCD surfaces against steel and sapphire can be ten times higher than those of smooth diamond surfaces against the same materials. The residual stresses on the surface of the coating layer and the integrity between the substrate and the coating have great impact on their wear properties. The purpose of this study is to compare the tribological properties of diamond films with those of bulk diamond.

Alumina & Bauxite: Alumina Industry Trends, Products, Environment

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

Tuesday PM Room: 217
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Ivan Anich, Alocal World Alumina, Techn. Deliv. Grp., Booragoon, Western Australia

2:00 PM

A Global Environmental Impact Assessment for Bauxite Mining-Land Use and Soil Erosion: *Peter Sliwka*[1]; Christian Bauer[1]; Karsten Eden[2]; Juri Grassmann[2]; Per Nicolai Martens[3]; Michael Röhrlich[3]; Martin Ruhrberg[3]; Henrike Sievers[2]; [1]Collaborative Research Center 525, Dept. of Eng. Geo. and Hydrogeo., RWTH Aachen, Lochnerstrasse 4-20, Aachen, North-Rhine-Westfalia 52074 Germany; [2]RWTH Aachen, Dept. of Min. and Eco. Geo., Wüllnerstrasse 2, Aachen, North-Rhine-Westfalia 52062 Germany; [3]RWTH Aachen, Dept. of Min. Eng. I, Wüllnerstrasse 2, Aachen, North-Rhine-Westfalia 52062 Germany

Bauxite is the main raw material for the aluminum production. Most bauxite is mined in surface operations in tropical and subtropical climates. Due to the land use of mining processes serious environmental impacts in the close vicinity of the pits may be caused. To gain information on the extent of environmental impacts from bauxite mining on global scale three sub-programs of the Collaborative Research Center 525 Aachen (CRC) have assessed major environmental impacts due to land use. For 52 open pit mines worldwide the land use and soil erosion potential have been calculated. For this case study information from three data bases of the CRC has been used. The first database, "Bauxite deposits", contains information on deposit type, reserves and statistical life time of each mine. The second database, called "WOBEX", contains information on mining processes, annual production and operational management. In addition to the databases mentioned above the environmental information system "EIS" presents data on climate, topography, morphology, soil types and geology.

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Evaluation of Bauxite Availability: J. Hausberg[1]; J. Grassmann[1]; H. Sievers[1]; *F. M. Meyer*[1]; [1]Collaborative Research Center 525, Instit. of Min. and Eco. Geo., Wüllnerstr. 2, Aachen 52056 Germany

Sustainable yields of renewable resources have long been recognized as valuable concepts. Applying the concept of sustainability to non-renewable mineral resources such as bauxite, however, leads to concern about both depletion of resources and degradation of the environment, and also raises questions about the adequacy of supply to meet future demands. Current bauxite supply estimates, derived from ratios between present reserves and annual production, indicate adequate supply for at least 200 years. If, in addition to economic factors, environmental considerations are included in bauxite reserve calculations, then the presently estimated adequacy of supply may be too high. Evaluation of typical ore characteristics of 174 bauxite deposits world-wide let to the identification of ten critical parameters that effect mineral availability in the context of sustainability. Results indicate that future increase in quality requirements in the environmental category by 50% will result in a reduction of presently available resources by 20% whereby the number of producing bauxite deposits world-wide will decrease to 84. A quality increase by 70% will reduce resources to 21% of the present figure.

3:00 PM Break

3:15 PM

Boehmite Process: An Alternative Technology in Alumina Production: *Dimitrios Panias*[1]; *Ioannis Paspaliaris*[1]; Achilleas Amanatidis[1]; Achim Hollnagel[2]; Hans-Werner Schmidt[2]; [1]National Technical University of Athens, Dept. of Min. and Metall. Eng. and Lab. of Metall., Iroon Polytechniou 9, Zografos, Athens 157 80 Greece; [2]LURGI Metallurgie GmbH, R+D-Dept., Lurgiallee 5, Frankfurt/MD 60295 Germany

Boehmite process is an innovative variation of the current Bayer process for the production of smelter grade alumina. It comprises three main stages. The first one is the bauxite digestion with caustic soda to produce aluminate liquor. The second is the boehmite precipitation from the supersaturated sodium aluminate solutions under atmospheric conditions. The last one is the boehmite calcination in order to produce anhydrous smelter grade alumina. The precipitation stage is highly innovative because for first time crystalline boehmite is precipitated from the Bayer liquors at temperatures lower than 100°C with an efficiency comparable to the one achieved in gibbsite precipitation stage of the current Bayer process. This paper aims at a detailed description of the boehmite process. The main parameters affecting the efficiency of boehmite precipitation process and the particle size distribution of produced solids will be presented and discussed. Moreover, the boehmite calcination process will be presented giving emphasis to the quality of produced alumina. On the whole, the two alternatives, the current Bayer process and the boehmite process, will be compared and the advantages arising by the application of the new process will be discussed.

3:45 PM

Co-Processing of Different Type of Bauxites with High Efficiency: *Károly Solymár*[1]; Vasile Cismaru[2]; Vicol Alistarh[2]; Dan M. Gheorghe[2]; Tibor Ferenczi[3]; [1]ALUTERV Aluminium Designing and Contracting, Ltd., H-1116 Budapest, Fehérvári út 144, Hungary; [2]BBG Alum S.A. Tulcea, Isaccei nr. 83, Tulcea, Romania; [3]ALULAB Research and Development, Ltd., H-1116, Budapest, Fehérvári út 144, Hungary

The key precondition to reach high liquor productivity is the high supersaturation of the spent liquor. This goal should be achieved at the maximum possible alumina extraction yield. Such pregnant liquor can be produced processing only one type of bauxite: by low temperature digestion of gibbsitic bauxite, the elevation of the digestion temperature and/or by dosage of lime or other catalytic additives, tube digestion and by the counter-current double digestion process developed by ALCAN. The following variants to achieve the maximum alumina extraction yield and high cycle efficiency by the co-processing of different types of bauxite are discussed: Simultaneous processing of diasporic and/or boehmitic and/or gibbsitic bauxites and joining the pregnant liquors. Sweetening of the blow-off slurry of the diasporic and/or boehmitic bauxites in a flash tank by dissolving gibbsitic bauxite. Double stage digestion of combinations of diasporic, boehmitic and gibbsitic bauxites (essentially a special variant of the sweetening.

4:15 PM

Raw Material for Catalysts: Prospect of Alumina Plants: *Vadim A. Lipin*[1]; [1]Russian National Aluminium-Magnesium Institute (VAMI), 86 Sredny pr., St. Petersburg 199106 Russia

Possibility of small technologies application to alumina plant processed of aluminium content feedstock is of great interest to chemistry. Advantage processed in Russia alkali alumosilicate raw materials is the opportunity of passing reception of chemical products for oil gas processing and other industry. Alumina-chemical complex can include the production of silica white, zeolites, active alumina oxide or hydroxide, calcium aluminates, rubidium-cesium concentrates. Contrary to the traditional methods the reception of these products by sintering method passing with alumina allows

essentially to reduce and in a number of cases to avoid ecological problems. Besides cost of alumina produced by sintering method is reduced. The technological features of catalysts production and initial components for their from alkali alumosilicate of raw materials was considered.

Aluminum Joining-Emphasizing Laser and Friction Stir Welding:
Session 1 - Joining Aluminum for Automotive Applications

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

Tuesday PM Room: 214
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: John A.S. Green,The Aluminum Association, Washington, DC 20006-2168 USA; J. Gilbert Kaufman, Kaufman Associates, Columbus, OH 43220 USA

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Aluminum Tailor-Welded Blanks for Automotive Applications: *Jo Ann M. Clarke*[1]; William H. Christy[2]; [1]Alcan Global Automotive Products, 37676 Enterprise Court, Farmington Hills, MI 48331 USA; [2]Alcan International, Ltd., 945 Princess St., P.O. Box 8400, Kingston, Ontario K7L 5L9 Canada

Faced with increasing demands for fuel economy, the automotive industry has seriously begun to incorporate aluminum alloys into vehicles. Integration of aluminum alloys in closures, body structure, and frames provides desired light-weighting without compromising performance or safety. Currently, steel tailor-welded blanks are widely used in the automotive industry for body and closure panels. Tailored blanks are characterised by their optimised usage of alloy and gauge. Given the relative cost of aluminum alloys compared to steel, this concept lends itself well to the implementation of tailored blanks in aluminum. The laser is an effective joining method to produce aluminum tailor-welded blanks. This paper will describe metallographic analysis and formability results for laser welded tailored blanks produced using CO2, Nd:YAG, and direct diode lasers, and will discuss several potential automotive applications.

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Laser Welding of Aluminum Alloys Using a Dual Laser Beam Technique: *Jian Xie*[1]; [1]Edison Welding Institute, Laser Process., 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

Aluminum alloys have been widely used in industries because of the attractive features of low density and good corrosion resistance. For example, there is a trend to use more aluminum alloys in the automotive industry to reduce vehicle weight for improved fuel efficiency and reduced atmospheric pollution. Laser welding is being evaluated as one of the major joining techniques for aluminum alloys due to several advantage such as high speed, little distortion, non-contact processing, good mechanical properties, and consistent weld quality. However, laser welding of aluminum alloys is not as easy as steel because of high reflection for laser beams, high thermal conductivity, and low viscosity. As a result, many weld defects are observed in laser aluminum welds such as blowholes, spatter, undercut, irregular beads, cracking and porosity. Presence of the weld defects will significantly degrade the mechanical properties such as tensile strength, fatigue performance, and corrosion resistance. Therefore, it is important to develop innovative laser processes for improving weld quality of aluminum alloys. In order to improve quality of laser aluminum welds, EWI investigated the impact of an innovative laser welding process called Dual Beam Laser Welding. A high power laser beam (either CO2 or Nd:YAG lasers) is split into two equal power beams by a beam splitting system, and then the dual beams in tandem are used to weld aluminum sheets. The aluminum welds produced by the dual beam Nd:YAG laser had an appearance as smooth as steel welds. Blowholes, underfill, irregularities, spatter were seldom observed in the dual beam aluminum welds. By investigating the dynamic behaviors of plasma plumes, it was found that the improvement in weld quality was due to the enhanced keyhole stability during the dual beam laser welding. The dual beam laser welding technique has been used to weld aluminum sheets in butt and lap joint configurations including tailor welded blanks, similar thickness butt welds, partial and full penetration lap welds. It was found that the aluminum welds made by the dual beam laser welding technique have good mechanical properties.

3:00 PM

Microstructural Evaluations of Electrode Life Behavior When Resistance Spot Welding 5754 Sheet: *Jerry Ellison Gould*[1]; Wayne Chuko[1]; [1]Edison Welding Institute, Resist. and Solid State Weld., 1250 Arthur E. Adams Dr., Columbus, OH 43221 USA

Aluminum sheet products are currently of broad interest for automotive body manufacture. The primary joining method for such sheet components is resistance spot welding. Resistance spot welding of aluminum sheet, however, as been hampered by electrode life concerns. During repeated resistance spot welding, aluminum sheet can suffer periodic interfacial failure of the joints, typically in numbers upward of 5%. In this work, the metallurgical origins of these periodic failures was investigated on 5754 sheet. Electrode life tests were conducted, using periodic sectioning to reveal underlying weld microstructures, and 100% destructive testing to characterize frequency of interfacial failure. Results show that during electrode life testing, both porosity at the weld periphery, as well as frequency of interfacial failures, increase with electrode wear. Correlations between this edge porosity and frequency of failure was also established. Results were explained in terms of variations in contact resistance and specific pressure as the electrodes wear.

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Advancements in Aluminum Resistance Spot Welding to Improve Performance and Reduce Energy: *Donald J. Spinella*[1]; Edward P. Patrick[2]; [1]Alcoa Technical Center, Join. and Assemb. Div., 100 Technical Dr., Alcoa Center, PA 15069 USA; [2]E.P. Patrick and Associates, 4530 William Penn Hwy. #7300, Murrysville, PA 15668 USA

Aluminum resistance welding is dominated by the spatial uniformity of current along the electrode contact and interfacial surfaces rather than bulk resistances traditionally associated with steel. This observation is complicated by aluminum's thermal and electrical conductivity requiring two to three times coated steel's current level but only one-third the duration to generate a resistance weld. Earlier work addressing the effects of equipment, coatings, and lubricants established that electrode life and weld quality were enhanced by conditions providing uniform current distribution at electrode contact and interfacial surfaces. Embracing this fundamental knowledge a patent-pending advancement dramatically improving both the electrode life and energy input requirements for aluminum products has been developed. Experiments incorporating the new enhancement on both 5xxx and 6xxx alloys have demonstrated electrode life increases up to several times the Aluminum Association's standard practices. Additionally, the process enhancement has significantly reduced undersized welds that occur intermittently while improving overall weld symmetry and shape. In terms of energy inputs required, the new technique facilitates 10% reductions in both current and force, driving the overall aluminum spot weld energy to levels comparable to coated steels.

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Single-Sided Projection Welding of Aluminum Sheet Using the Hy-Pak® Welding Process: *Donald J. Spinella*[1]; Robert VanOtteren[2]; Edward P. Patrick[3]; [1]Alcoa Technical Center, Join. and Assemb., 100 Technical Dr., Alcoa Center, PA 15069 USA; [2]Newcor Bay City, Weld. Rsch. and Dev., 1846 Trumbull Dr., P.O. Box 918, Bay City, MI 48707 USA; [3]E.P. Patrick and Associates, 4530 William Penn Hwy #7300, Murrysville, PA 15668 USA

Several significant process and product developments have enabled single-sided projection welding of 6xxx-T4 automotive aluminum sheet (patents pending). The welding was performed on a hem geometry typically encountered on door and hood applications using an enhanced version of the Hy-Pak® technology. This process is unique because both electrodes approach from the same side of the component, enabling no visible weld mark on the opposing surface. An experimental design was developed to understand the influence of the major process variables (weld current, weld force, projection height) on the tensile shear and button peel performance. The DOE was performed on single-sided projection welds consisting of 0.8mm 6111-T4 aluminum sheet. The results indicate a range of acceptable process parameters that produced tensile shear strengths on the order of the Aluminum Associations T-10 minimum values for resistance welds of that gauge.

Aluminum Reduction Technology: Anode Effects and Environmental

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

Tuesday PM
February 13, 2001
Room: 206-207
Location: Ernest N. Morial Convention Center

Session Chair: Jim Metson, University of Auckland, Dept. of Chem., Private Bag, Auckland 92019 New Zealand

2:00 PM
Aiming for Zero Anode Effects: *Warren Haupin*[1]; Edward Seger[2]; [1]Consultant, 2820 Seventh St. Rd., Lower Burrell, PA 15068 USA; [2]Consultant, 746 Kiski Park Dr., Apollo, PA USA

The conventional predictor of an incipient anode effect is a high rate of rise in cell voltage. This triggers a fast feed of alumina. Sometimes, however, the fast feed fails to prevent the anode effect. Other indicators can be combined to more accurately trigger the fast feed. These include hysteresis in cell volts vs. current and/or a rapid change in anode current distribution. The authors point out the need for more accurate alumina feeding and conditions where use of preemptive anode effect quenchs is desirable.

2:25 PM
Inhibition of the Anode Effect in Aluminium Electrolysis: Understanding the Mechanism in the Case of Doped Carbon Anodes: *Philippe Meunier*[1]; [1]University of New South Wales (Pechiney), Sch. of Chem. Eng. and Indust. Chem., Sydney, NSW 2052 Australia

The mechanism of the inhibition of the anode effect occurring during aluminium electrolysis has been studied with different techniques: cyclic voltammetry, chronopotentiometry and impedance spectroscopy coupled with gas analysis. Different doping agents (metal oxides and salts of lead, ruthenium, lithium and calcium) in graphite anodes have been tested in different bath compositions from pure cryolite up to 3% Al2O3. The doping agents have been classified according to their inhibiting activity with reference to two important parameters: the critical current density of the anode during cyclic voltammetry and the alumina concentration in the bath when the anode effect occurs. Compounds formed on the anode surface have been analysed by XPS and electron microscopy and a possible mechanism for anode effect has been proposed from the results of this study.

2:50 PM
Experimental Studies on Anode Effects by the Visualization of the Molten Aluminum Surface Oscillations: *Aureliu Panaitescu*[1]; Augustin Moraru[1]; Nicu Panait[1]; Gheorghe Dobra[2], Nicolae Munteanu[2], Marian Cilianu[2]; [1]University "Politehnica" Bucharest, Elect. Eng. Dept., 313 Splaiul Independentei, 77206 Bucharest, Romania; [2]S.C. ALRO S.A., 116 Pitestilor St., 0500 Slatina, Romania

The results of experimental researches on anode effects are presented. The effects were produced by interrupting the cells' feeding with alumina. The gas layers which appear below anodes during the anode effect insulate relatively large surfaces of the anodes. Therefore, the electrolysis total current is shared non uniformly in the electrolyte and molten metal mass, the Lorenz forces are not balanced and the movements of the electrolyte and of the molten metal become very intense. The "equivalent" shape of the molten aluminum surface and its vertical oscillations are visualized in real time on a computer screen by an original installation of data acquisition and processing. The paper presents the influence of magnetic field symmetrization produced by the external busbars on the molten metal movements during the anode effects. In this paper is developed the study of anode effects by analyzing the dynamics of the molten media during these phenomena.

3:15 PM
Resource Conservation by Improvements of Primary Aluminium Production: Bernd Friedrich[1]; Joachim Krüger[1]; Georg Rombach[1]; *Jürgen Schlimbach*[1]; [1]RWTH Aachen, IME Dept. of Nonferr. Process Metall., Intzestr. 3, Aachen 52056 Germany

This paper presents the balance of mass flows due to the primary aluminium production from bauxite to molten metal and the identification of optimisation potentials. To balance the mass flows and energy requirements of the world wide aluminium production the developed process chain is divided into technique specific modules. There are nearly 70 alumina refineries with a total capacity of 56 Mio. t/a of alumina and nearly 200 smelters with a total capacity of 26 Mio. t/a of aluminium in operation. Different smelter technologies are classified in view to the specific energy demand, anode consumption and emissions like fluor or SO2. The specific electrical energy requirements for electrolysis ranges from 17.5 to 13.0 kWh/t of molten aluminium with an capacity weighted average of 14.9 kWh/t. Installation of best smelting practice will lower the electrical energy demand to nearly 87% of today's energy consumption.

3:40 PM Break

3:50 PM
Effect of LiF on the Vapour Pressure over Cryolite Containing Melts: *Øyvind Tveter Gustavsen*[1]; *Terje Østvold*[1]; [1]NTNU, Dept. of Chem., Sem Sælands vei 12, Trondheim 7491 Norway

To understand the behaviour of LiF in NaF-AlF3 melts, vapour pressures and vapour composition data were measured over the three MF-M'F-AlF3 (M, M'=Li, Na, K) mixtures at cryolite ratios, CR=2 and 3. A maximum in the pressure of the major vapour species NaAlF4 was observed at about n(LiF)=n(NaF). Similar results were obtained in all the systems studied. Based on the pressures, activity coefficients were obtained. A pronounced increase were observed in the AlF3 activity when LiF was added. Similar results were obtained for all the systems studied. The concentrations of the species AlF63-, AlF52-, AlF4- and F-, as measured by Raman spectroscopy, were available in the literature. These data showed a maximum in AlF4- concentration at about n(LiF) = n(NaF). This partly explains the observed maximum in the NaAlF4 pressure. A first approximation model based on nearest neighbour anion-cation interactions showed fair agreement with measured vapour pressures. Vapour pressures over industrial NaF-AlF3 melts containing Al2O3, CaF2, LiF, MgF2 and KF could be calculated using a model based on the present laboratory vapour pressure data. This model can be used to estimate the fluoride loss caused by evaporation from Al-electrolysis cells during operation.

4:15 PM
Understanding the Effects of the Hydrogen Content of Anodes on Hydrogen Fluoride Emissions from Aluminium Cells: *Edwin C. Patterson*[1]; M. M. Hyland[1]; V. Kielland[2]; B. J. Welch[1]; [1]The University of Auckland, Dept. of Chem. and Matls. Eng., High Temp. Matls. and Process. Grp., Auckland 92019 New Zealand;

[2]Hydro Aluminium Technology Center Ardal, R&D, Ovre, Ardal N-6884 Norway

Recent reviews have pointed to the need to clarify the contribution of the identified sources of fluoride emissions from aluminium cells. Of all the sources investigated, the influence of the residual hydrogen content of prebaked carbon anodes is the least understood. This paper outlines both experimental and industrial results of such an investigation. In laboratory studies, HF generated from an experimental electrolysis cell was analysed using a FTIR spectrometer. Contrary to past research, the reaction of this hydrogen content (0.03 to 0.08 wt% of the anode) with the electrolyte was found to be virtually complete, resulting in significant HF generation from the anode surface. This was also seen in industrial measurements. In the in-plant studies it was found that if the emission was broken down into the variable alumina feeding contribution and a constant background contribution, the anode hydrogen reaction contributes to almost half this background emission. Hence in an overall emission reduction scheme the effectiveness of the anode baking process contributes to an important portion of the overall HF emissions.

4:40 PM

The Practice of Dry Scrubbing: *G. A. Wellwood*[1]; [1]Torftech Limited, Ferndale Court, West End Rd. Mortimer, Reading, Berkshire RG7 3TS UK

Dry scrubbing is based on the chemisorption reaction between smelter grade alumina and gaseous fluoride. One of the key performance issues of this technique is the specific fluoride loading of the alumina as a function of the gaseous fluoride concentration in the scrubbed exhaust. Improving the fluoride loading of the alumina while maintaining an acceptable concentration of fluoride in the exhaust is a key economic and environmental leverage point. The system isotherm, which represents the equilibrium fluoride loading/exhaust gas concentration data, defines the best possible performance. The first stage of this investigation involved the construction of such an isotherm based on actual smelter exhaust and plant alumina. To generate the data for the practical isotherm, an ideal gas-solid contacting stage was required. The relatively simple transport reactors utilised in commercial dry scrubbing systems typically involve solids recycle and are therefore not conducive to equilibrium studies. A pilot plant featuring a high efficiency single stage gas-solid contactor was therefore developed. Despite being based on an actual exhaust gas/alumina system, the practical isotherm was found to significantly over predict the performance of dry scrubbing plants based on transport reactors. This finding led to the second stage of the investigation that revealed the adsorption process is actually controlled by the diffusion of gaseous fluoride through the boundary layer of gas surrounding each alumina particle. Boundary layer diffusion is in turn a function of differential gas-solids (slip) velocity and reactors capable of increasing slip velocity yield superior performance, providing near maximum fluoride loadings with only a single solids pass.

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High Performance and Cost Efficient Dry Scrubber Retrofit at HAW: *E. Sturm*[1]; M. Worbis[1]; G. Wedde[2]; E. Holmefjord[2]; [1]Hamburger Aluminium Werke GmbH Germany; [2]Alstom Power Norway

Pot upgrades and compliance with new emission regulations often requires major performance improvements of existing dry scrubber installations. The successful counter-current dry scrubbing principle (Abart) has been applied to an existing dry scrubber at the HAW smelter in Germany. A combined reactor-separator is retrofitted to the existing inlet gas duct for pre-adsorption of fluorides using enriched alumina from the dry scrubber. To minimise investment and operating costs the pre-adsorption reactor was designed for high efficiency at low pressure-drop. The convenient integration of this concept into the existing plant shows the flexibility of the reactor as a potential dry scrubbing retrofit concept. The combined performances of the reactor and the existing dry scrubber as experienced at HAW demonstrate a cost efficient system that satisfies strict emission regulations.

Carbon Technology: Cathode 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

Tuesday PM — Room: 215-216
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Richard O. Love, Century Aluminum of West Virginia, Ravenswood, WV 26164 USA

2:00 PM

Improvement in the Calcination Process of Anthracite for Cathode Carbon Blocks: *Ryosuke Kawamura*[1]; Tsutomu Wakasa[1]; [1]Nippon Denkyoku K.K., Res. & Dev. Dept., Kambara 5600, Kambara-cho, Ihara-gun, Shizuoka Prefecture 421-3201 Japan

The calcination of anthracite in an electric oven was studied to improve uniformity of the product. As is well known, electrically calcined anthracite is widely used as the main raw material in cathode carbon blocks for aluminum reduction cells, and its quality is one of the most important factors affecting their properties. However, electric ovens in which anthracite is calcined usually have large temperature distributions even though the anthracite's quality strongly depends on the heat treatment temperature. In this work, the design of the electrodes in a prototype calcining oven was modified to reduce the temperature distribution inside the oven. The improved oven performance was confirmed by improved uniformity of the calcined anthracite.

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Crack Quantifying in Anthracite Grains by the Use of Image Analysis: *Jorund Gimmestad Hop*[1]; Harald Arnljot Øye[1]; [1]Norwegian University of Science and Technology, Dept. of Chem., Sem Selandsvei 12, Trondheim 7491 Norway

Image analysis used to calculate porosity has been further developed to quantify cracks in anthracite grains in the magnitude 30-150 microns. The output from the analysis is crack-width versus crack area. During the first hours of electrolysis, the anthracite grains in anthracitic cathode materials crack to some degree because of sodium penetration. The degree of cracking seems to be related to the calcining temperature. Anthracite grains calcined to 1500, 2000 and 2500°C were exposed to sodium vapour. The surfaces of the grains were analysed with image analysis before and after sodium exposure. It appears that higher calcining temperatures lead to a lower degree of cracking, which is quantified by the image analysis method.

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Porosity Modifications in the Carbon Cathode of Aluminum Reduction Cell: *S. M. El-Raghy*[1]; J. Wiliamson[2]; T. M. Samy[3]; M. O. Ibrahiem[4]; [1]Cairo University, Faculty of Eng., Dept.of Min., Petrol. and Metall., Giza, Cairo, Egypt; [2]London University, Imperial College, London, UK; [3]Tabin Institute of Metallurgical Studies, Tabin, Helwan, Egypt; [4]Alkuminum Company of Egypt, Nag Hamadi, Egypt

High porosity in carbon cathodes in an aluminium reduction cell allows the penetration of different electrolyte components, particularly sodium fluoride. Swelling of the cathode over time due to this penetration generates high stresses on the cathode shell influencing cell life. A study was conducted to modify the open porosity of the carbon blocks. Reduction of the porosity was achieved by impregnation with a high carbon yield resin (furfuryl alcohol) for different periods. Factors influencing the efficiency of the modification process included pressure (vacuum) of impregnation, catalyst concentrations and curing time. Open porosity was reduced from 15% to 1.5%. This reduction in porosity resulted in stronger blocks, compressive strength increased from 410 to about 600 Kg/cm^2. Electrical resistivity dropped by about 10%. Another important property namely, sodium penetration, was reduced by about 65% compared with the reference untreated carbon block.

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Differentiating Cathode Block Binder Pitch Behaviour: Parin Rafiei[1]; *Frank Hiltmann*[2]; Margaret M. Hyland[1]; Barry J. Welch[1]; [1]University of Auckland, High Temp. Mats. and Proc. Grp., Dept. of Chem. & Mats. Eng., Private Bag 92109, Auckland, New Zealand; [2]SGL Carbon GmbH, Griesheim Plant, Stroofstasse 27, D-65933 Frankfurt, Germany

Cathode blocks made with similar filler aggregate materials and similar physical properties have been known to exhibit quite different performances in smelting cells. Accordingly an investigation has been undertaken to ascertain whether the coal tar pitch used can play a role by exhibiting different behaviours during the baking process. The first part of the study has been to develop a test to differentiate coal tar pitches with similar physical properties but from different sources. The pitches have been subjected to slow heat treatment and differentiated by analysis of volatile decomposition products released. In the initial heat-up, as a consequence of both volatilization and decomposition, polyaromatic hydrocarbons are released. These products were collected by Solid Phase Micro-Extraction (SPME) and analyzed by Gas Chromatography/Mass Spectrometry (GC/MS). It has been found that the pitches fall into distinctly different groups. The difference in heat treatment behaviour identified above was then correlated to physical properties of pilot plant cathode specimens made using the two extremes of pitches.

Cast Shop Technology: Grain Refining

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

Tuesday PM Room: 208-210
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chair: David StJohn, CAST Queensland University, Brisbane, Qld, Australia

2:00 PM

The Impact of TiCAl and TiBAl Grain Refiners on Casthouse Processing: Adriano M. Detomi[2]; Alexandre J. Messias[2]; Stephen Majer[1]; *Paul Stephen Cooper*[1]; [1]London and Scandinavian Metallurgical Company, Ltd., Al Div., Fullerton Rd., Rotherham, South Yorkshire S60 1DL UK; [2]Companhia Industrial Fluminense, BR 383-Km 94, 36.300-000, Sao Joao Del Rei-MG, Brasil

Al-3% Ti-0.15% C grain refiner has been in commercial use for over five years. It has seen use in a wide range of alloy systems and solidification process methods. A number of differences have been observed when comparing to the traditional Al-Ti-B grain refiners. These include melt cleanliness, interactions with degassing and filtration systems, grain structure and surface appearance. Using state of the art equipment at the Companhia Industrial Fluminense in Brazil, these issues have been assessed on a production scale level. A wide variety of different alloys have been cast on a wheel and belt caster and processed to wire. The addition point of the grain refiner was varied between before and after the degasser, all additions being made before a ceramic foam filter. The effect of the grain nucleating particles and their interactions with melt cleanliness equipment was studied.

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The Effect of Alloy Content on Grain Refinement Practice in Aluminium Alloys: *Mark A Easton*[1]; David H StJohn[2]; [1]Monash University, Dept. of Mats. Eng., Wellington Rd., P.O. Box 69M, Clayton, Victoria 3800 Australia; [2]University of Queensland, Dept. of Min., Min. and Mats. Eng., Brisbane, Queensland 4072 Australia

The grain refining ability of five wrought aluminium alloys is investigated to determine optimum grain refining practice. Using predictions of a recently developed model incorporating the effect of solute on grain size, titanium additions are made independently of TiB2 additions to determine the optimum ratio and level of TiB2 and solute titanium additions for each of the alloys. For alloys that do not contain elements that poison the nucleating ability of TiB2, it is found that one level of TiB2 may be added to a range of alloys. To facilitate the achievement of a fine grain size, additions of titanium as solute can be made to alloys that are more difficult to grain refine because of their low solute levels. The amount of solute titanium required is predicted by the model. The implications for grain refinement practice in the casthouse are subsequently discussed.

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Anomalous Grain Coarsening Behaviour Observed in Aluminium Alloys Cast with Low Superheat and Low Grain Refiner Additions: *John Andrew Taylor*[1]; Hao Wang[1]; David H StJohn[1]; Ian Bainbridge[2]; [1]CRC for Cast Metals Manufacturing (CAST), Dept. Min., Mins. and Mats. Eng., The University of Queensland, Brisbane, QLD 4072 Australia; [2]Direct Chill Casting Pty. Ltd., P.O. Box 82, Boyne Island, QLD 4680 Australia

Chemical grain refinement of aluminium alloys has been commercially practiced for many years now. Low superheat casting has also been recognised as a means of inducing grain refinement in aluminium alloys. However, recent grain refining studies have indicated that when both techniques are used in combination, the result is not always an improvement in overall refinement. In fact, grain coarsening has been observed under certain circumstances. Grain size has been measured in test castings made from 356, 6063 and 2024 alloys poured at low superheats using various grain refiners at a range of addition levels. The results of these trials are reported and discussed in this paper. Some grain coarsening effects were observed at lower addition levels, particularly with Al-B master alloy. This suggests implications for impurity control. High addition levels of refiners may yield only marginal improvements in grain size compared with non grain-refined low superheat samples.

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Effectiveness of In-Situ Aluminum Grain Refining Process: *Joseph A. Megy*[1]; Douglas A. Granger[3]; Geoffrey K. Sigworth[2]; Christopher R. Durst[1]; [1]JDC, Inc., South Chester St., New Cumberland, WV 26047 USA; [2]GKS Engineering, 116 Derby St., Johnstown, PA 15905 USA; [3]GRAS, Inc., Murraysville, PA 15668 USA

The fy-Gem process involves the in-situ formation of heterogeneous boride nuclei in molten aluminum for the purpose of grain refinement. This three-year study has demonstrated grain refinement in various aluminum alloy types. While the wrought alloys will require further improvements to the process before it grain refines as well as existing grain refiners, the fy-Gem process has shown that it can produce grain refinement comparable to current commercial practices in foundry alloys. The major benefit of the fy-Gem process includes the elimination of KAlF salt and boride agglomerates, which are usually present in commercial grain refiners, and in the projected cost savings through the elimination of several intermediate steps involved in the conventional production of commercial grain refiners. This paper will discuss the findings of research and development on this project, including bench scale tests, pilot studies, and theoretical work.

3:55 PM

Method for Optimized Aluminum Grain Refinement: *Rein R. Vainik*[1]; *Lennart Backerud*[1]; [1]Opticast AB, Osterlangatan 39, SE-111 31 Stockholm, Sweden

A new method has been developed for measurement of grain size and determination of necessary grain refining additions. Due to rapid sample preparation and grain size analysis, the method can be used on-line in all cast houses, where holding furnaces are used. The grain sizes measured are compared against calibration curves and the optimum amount of addition is calculated. The method can also be used for production of ingots in any specified grain size range. In practical application cost savings of more than 50% have been accomplished, at the same time as a considerable quality improvement has been achieved.

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

Tuesday PM Room: 222
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Jerome Kruger, Rockville, MD 20850-1921 USA; Digby D. Macdonald, Pennsylvania State University, Center For Electrochem. Sci. and Techn., University Park, PA 16802 USA

2:00 PM

The Role of Electronic Properties of Passive Films in Pitting Corrosion: *Z. Szklarska-Smialowska*[1]; [1]The Ohio State University, Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

During the last decades many studies were done evaluating the electronic properties of the passive films. However, not many papers exist dealing with the electronic properties of the film in conjunction with a passive film's breakdown. Most of the authors have examined the inhibition of pitting during illumination but fewer have studied the influence of the band-gap energy and the flat-band potential on the resistance of a passive metal to pitting corrosion. The existing hypothesis taking into consideration the semiconductive properties of the passive film in the film breakdown will be discussed and a new mechanism of a passive film's breakdown will be presented.

2:30 PM

Corrosion of Metals By Contact With Metal Oxides: *Norio Sato*[1]; [1]Hokkaido University, Electrochem. Lab., Graduate School of Eng., Kita-Ku, Sapporo 060-0813 Japan

The electrode potential of oxide-covered metals and the mixed electrode potential of corrodible metals in contact with metal-oxides were discussed in view of ionic and electronic transfer processes across the electrode interfaces. The oxide-covered metal electrode stands either at the electrode potential of metal oxide formation or the flat band potential of metal oxide. The mixed electrode of a corrodible metal and a metal-oxide shifts its metal potential toward the flat band potential of the metal oxide in the cathodic direction if the oxide is n-type, or in the anodic direction if the oxide is p-type. Photoexcitation of contacting oxides enables the anodic oxygen evolution to occur on n-type oxides and the cathodic hydrogen evolution to occur on p-type oxides: hence, n-type oxides tend to reduce the metallic corrosion and p-type oxides tend to increase the corrosion.

3:00 PM

Pitting in Alumina Films: Void Initiation and Growth by STM-Induced Electric Fields: *Jeff A. Kelber*[1]; N. P. Magtoto[1]; Chengyu Niu[1]; M. Anzaldua[1]; [1]University of North Texas, Dept. of Chem., P.O. Box 305070, Denton, TX 76203 USA

STM-induced localized electric fields > 5 MV/cm induce void formation and growth at the interface between 7.5 Å thick Al2O3 films and the Ni3Al(111) substrate. STM constant current imaging at varied bias voltages shows that the voids form at the oxide/metal interface and grow into the metal with time of exposure to the field. At a critical size (~ 3 nm deep, 50 nm wide), the thin oxide overlayer collapses. Void formation at the interface, followed by growth into the metal and oxide collapse, closely parallels behavior observed by PAS for pit formation and growth in electrochemically etched alumina films on aluminum substrates. Electric fields required to induce void initiation and growth are similar in magnitude for those induced by specific adsorption of anions (e.g. Cl-) the surface of an oxide thin film, and are also similar to those required to induce time-dependant breakdown in magnetoresistance alumina tunneling barriers. The data demonstrate that pitting can occur without chloride penetration of the oxide matrix, and indicate that similar field-induced mass transport underlies both localized corrosion of passive films and the breakdown of tunneling-based nanoelectronic devices.

3:30 PM

On The Effect of Surface Layers On the Crack Initiation on Nickel Base Alloys in High Temperature Water: *P. Combrade*[1]; [1]Centre Technique Framatome, BP 181, 71 205 Le Creusot Cedex, France

It is now well established that the surface layers may have a strong effect on the crack initiation behavior of different nickel-base alloys in high temperature water. The surface layers involved in these effects include the oxide films as well as substrate layers which can be modified either by the fabrication processes or by the exposure to high temperature water. On Alloy 600, a correlation has been observed between crack initiation time and different characteristics of the oxide film (thickness, morphology, chromium depletion of the substrate) but not with the film repair kinetics as observed on many other SCC systems, including Alloy 600 in caustic environments. On Alloys X 750 and 718, crack initiation was shown to depend on the surface conditions resulting from fabrication processes i.e. oxide films and damages to the substrate. This paper intends to summarize these observations and to discuss possible consequences for the understanding of the crack initiation mechanisms.

4:00 PM

The Effect of Mechanical Stress on Localized Corrosion: *Hans Boehni*[1]; Thomas A. Suter[2]; Eric G. Webb[1]; Richard C. Alkire[2]; [1]ETH, IBWK, ETH Hoenggerberg, HIF E11, Zurich, ZH 8093 Switzerland; [2]University of Illinois, Dept. of Chem. Eng., 114 Roger Adams Laboratory, 600 South Mathews Ave., Urbana, IL 61801 USA

Corrosion pits on passive metals such as stainless steels are often initiation sites for stress corrosion cracking (SCC). In this study the effect of mechanical stress on localized corrosion processes was investigated, using microcapillaries as electrochemical cells. The use of microcapillaries allowed to test small areas with no and with one single MnS-inclusion present on metal surface. Due to mechanical stress microcrevices may be formed at or within the inclusions, where the concentrations of aggressive species such as chloride or hydrogen ions reach a critical value for stable pit propagation. Therefore mechanical stress accelerates the onset of pitting and in this way the formation of potential starting sites for stress corrosion cracking.

4:30 PM

Effect of Chloride Ion on the Crevice Corrosion of Alloy T-2205: *Jamal N. Al-Khamis*[1]; Howard W. Pickering[2]; Bader G. Ateya[2]; [1]Saudi Aramco, The Consulting Services Dept., Eng. Ofc. Bldg., E-7600, Dhahran 31311 Saudi Arabia; [2]The Pennsylvania State University, Mats. Sci. & Eng., 326 Steidle Bldg., University Park, PA 16802 USA

Chloride ion is known to promote localized corrosion and stress corrosion cracking(SCC). The role of the electrode potential, E(x), on the crevice wall (caused by IR voltage) is also known to be important in crevice corrosion, but its role in SCC is less clear. This importance of E(x) in crevice corrosion depends on the size of the active peak in the system's polarization curve. In its complete absence, crevice corrosion can not, in principle, occur by the IR voltage drop mechanism. This fact could explain the need for an induction period prior to the onset of crevice corrosion, during which time the crevice solution becomes more corrosive and an active peak forms in the system's polarization curve. Experimental results will be presented on the role of chloride ion in crevice corrosion of alloy T-2205 duplex stainless steel.

5:00 PM
Initiation of Cl-SCC Cracks from Crevices Rather Than From Pits: *Shigeo Tsujikawa*[1]; [1]The University of Tokyo, Dept. of Mats. Sci., Tokyo 113 Japan

SCC in aqueous solutions containing chloride (Cl-SCC) is frequently encountered in austenitic Fe-Cr-Ni stainless steels. Two necessary conditions for Cl-SCC initiation were determined in previous studies: 1) cracks occur at a dissolving surface, 2) a microcrack can grow to a macrocrack (growing crack) only when the crack growth rate, C, is faster than the dissolution rate, V, at the surface. In neutral solutions, the dissolving surface specified in the first condition consists of large pits or a corroding crevice depending on the Cl-concentration. With decreasing bulk Cl-concentration, the dissolution rates at pits increases too rapidly, while those in crevices increase more slowly to continue to satisfy the V<C condition, where crevices must be a predominant initiation site for the SCC cracks. SCC tests were conducted at various electrode potentials and temperatures with a spot-welded specimen which has both a crevice and residual stress. Dissolution rates V of crevice corrosion of stainless steels under glass were determined via the Moire technique. The competition concept V<C could explain potential range for SCC and critical temperature for SCC at and below which a steel will not exhibit SCC.

Computational Thermodynamics and Materials Design: Phase Equilibria and Phase Transformations 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, 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

Tuesday PM — Room: 201
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Greg B. Olson, Northwestern University, Dept. of Mats. Sci. and Eng., Evanston, IL 60208

2:00 PM
Modeling the Ferrite-to-Austenite Transformation in Stainless Steel Welds as a Paraequilibrium Transformation: *John M. Vitek*[1]; Ernst Kozeschnik[2]; Stan A. David[1]; [1]Oak Ridge National Laboratory, P.O. Box 2008, MS 6096, Bldg 4508, Oak Ridge, TN 37831-6096 USA; [2]Graz University of Technology, Insti. for Mats. Sci., Weld. and Form., Kopernikusgasse 24, Graz A-8010 Austria

The kinetics of the austenite-to-ferrite transformation in low alloy steels were recently modeled and compared for conditions of orthoequilibrium or paraequilibrium at the interface[1]. It was found that the transformation under paraequilibrium conditions was seven orders of magnitude faster than the transformation with orthoequilibrium at the interface. A similar analysis setting paraequilibrium at the interface was carried out for the ferrite-to-austenite transformation that takes place in stainless steel welds upon cooling. Alloy compositions were comparable to those used in earlier experimental studies in which a massive ferrite-to-austenite transformation was proposed to explain the weld microstructures. The results of the calculations are compared to the experimental results and the alternative explanation of the observations in terms of a paraequilibrium transformation mechanism are discussed. 1. Calphad XXIX, Cambridge, Massachusetts, June, 1999. This research was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

2:30 PM
Coherent Phase Equilibria under Different Boundary Constraints: *Long-Qing Chen*[1]; Zi-Kui Liu[1]; [1]The Pennsylvania State University, Dept. of Mats. Sci. and Eng., Steidle Bldg., State College, PA 16802 USA

It is well known that coherency elastic stress in a solid can significantly modify the equilibrium compositions of a two-phase solid. The main objective of this work is to study the effect of different boundary constraints on the phase transition temperatures in a single-component system as well as two-phase coherent phase equilibria in a binary solid. The boundary conditions considered include stress-free boundary conditions, constraint boundary conditions, mixed stress-free and constraint boundary conditions (the thin film boundary condition). In addition, the effect of a constant applied strain or applied stress on transition temperatures and two-phase equilibria will be examined.

3:00 PM
Computational Investigations on the Microstructure Formation in Multi-Components Alloy Systems Based on the Discrete Type Phase Field Method: *Toru Miyazaki*[1]; Toshiyuki Koyama[1]; [1]Nagoya Institute of Technology, Nagoya 466-8555 Japan

The kinetic simulations based on the non-linear diffusion equation become very powerful method in fundamental understanding the dynamics of phase transformation with the recent remarkable development of computer. In the present study, we calculate the dynamics of microstructure changes due to the phase decomposition and microstructure coarsening in Fe-Al-Co and GaAs-InP alloy systems based on the phase field method. The composition dependencies of atomic interchange energy are taken into account so as to be applicable for the phase diagram of the real alloy systems. The elasticity and mobility of atoms are assumed to depend on the local order parameters such as composition, degree of order and so on. Time dependent morphological changes of the microstructure such as formation of modulated structure by spinodal decomposition, strain induced morphological changes of precipitates, the order-disorder phase transition with phase decomposition, discontinuous precipitation will be demonstrated. The results simulated are quantitatively in good agreement with the experimental results in the real alloy systems.

3:20 PM
Modeling Calculation of Sulfide and Phosphate Capacities: *Xiaozheng Lin*[1]; Ramana G. Reddy[1]; [1]University of Alabama, Tuscaloosa, Metall. and Mats. Eng., P.O.Box 870202, Tuscaloosa, AL 35487 USA

The Reddy-Blander thermodynamic model for calculating sulfide and phosphate capacities has been presented, so the capacities can be predicted a priori based upon the knowledge of the thermodynamic properties of the basic oxide components. The model is used to predict the capacities of silicate, aluminate and other binary slags, the results are in good agreement with measured data. The Reddy-Blander model is also applied to ternary and multicomponent slag systems, the model can also give reasonable predictions to the sulfide and phosphate capacities of these slag systems. The oxide activities and other properties used in the calculations are got from F*A*C*T. Software for the calculation of sulfide capacities has been developed, which permits the easy access to the calculated and experiment data.

3:40 PM Break

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Simulation of DTA Measurements from Calculated Enthalpy-Temperature Data: *William J. Boettinger*[1]; Ursula R. Kattner[1];

[1]NIST, Metall. Div., 100 Bureau Dr. Stop 8555, Gaithersburg, MD 20899-8555 USA

DTA (Differential Thermal Analysis) measurements are a standard method of determining equilibrium phase boundary temperatures. The measurement results are not only affected by phase transformation kinetics but also heating/cooling rates and heat transfer between furnace, sample and reference material. As a result, accurate measurements require proper calibration procedures as well as careful experimentation and interpretation of the data. Simulation of DTA measurements using enthalpy-temperature data provides understanding into how thermal events from phase transformations are reflected by the DTA signals, providing a basis for more accurate interpretation of DTA results. A simple model for heat flow between sample and furnace which predicts sample temperature as a function of time for given heating/cooling rates will be presented. Thermodynamic calculations of multicomponent alloys are used to obtain the phase transformation temperatures and enthalpy-temperatures data as input for the heat flow model to predict DTA measurements.

4:15 PM

Modeling Nucleation Kinetics in the Ni-Al Binary System Utilizing Computational Thermodynamics and Diffusion Kinetics: *Brett A. Boutwell*[1]; Raymond G. Thompson[2]; [1]AEA Technology Engineering Services, Inc., Mats. & Chem. Proc. Assess., 241 Curry Hollow Rd., Pittsburgh, PA 15236 USA; [2]University of Alabama at Birmingham, Dept. of Mats. and Mech. Eng., 1150 10th Ave. South, Birmingham, AL 35294-4461 USA

Nucleation is an important phenomena in understanding the phase transformation behavior of a material or alloy. The nucleation process estiblishes important microstructural details such as the distribution and critical size of newly formed phases which will influence the properties and performance of the material. Therefore, an understanding of the nucleation process can provide a better understanding of the microstructural evolution of a material and lead to better control over its properties. A model for predicting the nucleation kinetics of coherent, homogeneous precipitates using computational thermodynamic and diffusion kinetic data was developed. The nucleation model incorporates classical nucleation theory with derivations of incubation time from the theory of time reversal and requires the input of thermodynamic driving forces for phase formation and diffusion coefficients, which were obtained from Thermo-Calc and DICTRA. The model was used to predict the nucleation kinetics of gamma prime precipitates in a Ni-Al binary system. The results of the nucleation model were then compared to published experimental results.

4:40 PM

Multi-Component Solidification Simulation with a New Scheil-Gulliver Model with Back-Diffusion: *Ernst Kozeschnik*[1]; W. Rindler[1]; B. Buchmayr[1]; [1]Graz University of Technology, Insti. for Mats. Sci., Weld. and Form., Kopernikusgasse 24, A-8010 Graz Austria

The micro-segregation behavior during solidification of alloys is frequently described with the classical Scheil-Gulliver model.(1, 2) By assuming negligible diffusion in the solid phases, infinitely fast diffusion in the liquid and local equilibrium at the solid/liquid interface, the solute enrichment or depletion of the residual liquid as a function of temperature can easily be computed by an iterative algorithm. A corresponding numerical procedure is implemented, e.g., in the software packages ThermoCalc (3) or MatCalc (4). However, it is well known that the Scheil-Gulliver model usually only applies to the micro-segregation of substitutional elements and it is not generally applicable to interstitials, such as carbon or nitrogen, due to their fast diffusion. During solidification simulations in typical interstitial/substitutional alloys, such as steels, the rapid diffusion of interstitials has to be accounted for by including the back-diffusion mechanism into the numerical algorithm.(5) In the MatCalc software, the recently developed partial equilibrium module has been used to develop a multi-component Scheil-Gulliver model that can account for the back-diffusion of rapid diffusers.(6) The model is briefly described and its applicability is demonstrated exemplarily in the ternary model system Fe-Cr-C as well as the complex commercial casting steel GS 17 CrMoV 5 1 1. Due to the simplicity of the model and the high numerical stability, it is particularly suitable for solidification simulations in complex alloy systems. Excellent agreement between experiments, thermo-kinetic calculations carried out in Fe-Cr-C with the DICTRA program (7) and the new Scheil-Gulliver algorithm with back-diffusion is confirmed.

5:05 PM

An Analytical Model for Solute Redistribution during Dendritic Solidification: *Xiangdong Yao*[1]; *Arne Dahle*[1]; *David StJohn*[1]; *Cameron Davidson*[2]; [1]University of Queensland, Min., Min. and Mats., St Lucia Campus, Brisbane, QLD 4072 Australia; [2]CSIRO Manufacturing Science and Technology, P.O. Box 833, Kenmore, Brisbane, QLD 4069 Australia

A new analytical mathematical model for solute redistribution was developed, by solving Laplace equations with free boundary condition. Limited diffusion in liquid and in solid was considered. The solutions of diffusion were then coupled with the assumptions of mass conservation and local equilibrium at the liquid/solid interface. The model can calculate micro-segregation at the level of primary or secondary arm spacing for columnar dendrites or equiaxed dendrites. The results were compared with calculations based on existing models (Scheil, Brody-Flemings, Clyne-Kurz), as well as with some available experimental data in as cast Al-Cu, Mg-Al and Al-Si alloys.

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

Tuesday PM — Room: 225
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Jim Whitlock, Whitlock and Associates, Spearfish, SD 57783 USA; Jack Adams, Weber State University, Center for Bioremediation, Ogden, UT USA

2:00 PM Invited

Remediation Technologies for the Separation and Destruction of Aqueous Cyanide Species: *Courtney A. Young*[1]; [1]Montana Tech, Metall. and Mats. Eng., 1300 W. Park St., Butte, MT 59701 USA

Cyanide is a toxic species that is found predominantly in industrial effluents generated by metallurgical operations. Cyanide's strong affinity for metals makes it favorable as an agent for metal finishing and treatment and as a lixivant for metal leaching, particularly gold. These technologies are environmentally sound but require safeguards to prevent accidental spills. Various methods of cyanide remediation by separation and destruction are therefore reviewed. Reaction mechanisms are given throughout. The methods are compared in regards to their effectiveness in treating various cyanide species: free cyanide, thiocyanate, weak-acid dissociables, and strong-acid dissociables.

2:25 PM Invited

Recent Advances in Technologies for Biological Treatment of Thiocyanate, Cyanide, Heavy Metals and Nitrates: *James L. Whitlock*[1]; [1]Whitlock and Associates, 315 Canyon View Ln., Spearfish, SD 57783 USA

Biological treatment of mining and industrial effluents reached full scale commercialization in the early 1980's. These proven low

cost technologies have been limited in application due to a number of factors including, extended periods of time required for pilot studies as compared to chemical treatment technologies, lack of biological expertise in metallurgical personnel, long treatment periods, and performance failures based on improper application and process management issues. Renewed interest in biological treatment technologies is being driven by permits with lower effluent concentration criteria, the need to remove thiocyanate, nitrates and specific toxic heavy metals. Chemical treatment technologies, in general, have not been able to meet these present requirements in terms of effluent quality and treatment costs. This paper addresses the present state of biological treatment of mining and industrial wastes and the recent advances in these technologies. The future looks very bright for application of these low capital and operational cost technologies.

2:50 PM Invited

Biological Cyanide Degradation: J. *Van Komen*[1]; D. J. Adams[1,2]; [1]Applied Biosciences Corporation, Salt Lake City, UT USA; [2]Weber State University, Ctr. for Bioremediation, Ogden, UT USA

Cyanide heap leaching is the predominant technology used in processing low-grade gold ores. During closure of a heap leach operation, residual cyanide must be removed from the process and waste solutions, as well as the heap. Biological cyanide oxidation is a proven, economical technology for destroying cyanide in process and waste waters and spent heaps. The use of biological cyanide degradation eliminates the need for toxic or corrosive chemical oxidizers and has been implemented at full scale at treatment costs of ~$0.50/1,000 gal. Cyanide concentrations up to 250 mg/L, in process solutions and wastewaters, have been treated successfully. Some process solutions and wastewaters treated also contained arsenic, copper, iron, silver, selenium, mercury, nitrate and zinc, much of which was removed in the cyanide degradation process. Under optimal conditions, microorganisms can rapidly oxidize free cyanide in some process solutions from over 250 mg/L down to 0.1 mg/L WAD cyanide, in 4 to 5 hr. Cyanide is degraded to ammonia and carbon dioxide, with 50% of the cyanide carbon liberated as CO2. In general, carbon tank based bioreactors degrade cyanide at significantly higher rates than process or wastewater pond configured treatments, which usually degrade cyanide more rapidly than in heap treatments. Current investigations on use of cell-free enzyme preparations for cyanide degradation will also be presented. The use of enzymes as an alternative to live microbial cells shows promise. Observed advantages of enzymes over live microbial cells for cyanide degradation include: (1) the ability to tolerate and degrade higher cyanide concentrations, (2) nutrients to support live microbial cells are not required and (3) the effects of other toxic inorganics, such as arsenic and copper, found in process waters are eliminated.

3:15 PM Break

3:30 PM Invited

Demonstration of an Integrated Bioreactor System for the Treatment of Cyanide, Metals, and Nitrates in Gold Mine Process Water: *Marietta Canty*[1]; Randy Hiebert[1]; [1]MSE Technology Applications, Inc., P.O. Box 4078, Butte, MT 59701 USA

A paper is presented in which an innovative, proprietary, biological process is described for the treatment of cyanide-, metals-, and nitrate-contaminated mine process water. The technology is capable of detoxifying cyanide and nitrate and immobilizing metals in wastewater from agitation cyanide leaching, as well as heap leaching. A pilot-scale demonstration is described in which a pilot-plant was constructed to detoxify cyanide and immobilize several metals from a mine process waste stream. The demonstration was performed jointly under the Mine Waste Technology Program (MWTP), which is funded by the Environmental Protection Agency (EPA) and is jointly administered by the U.S. Department of Energy (DOE), and the Superfund Innovative Technology Evaluation (SITE) Demonstration Program. MSE Technology Applications located in Butte, Montana was the contractor responsible for conducting the project. The pilot-scale unit was field-tested at the Echo Bay McCoy/Cove Mine southwest of Battle Mountain, Nevada. The biot reatment process was considered to be very successful in detoxifying cyanide from the mine process water. The process was also determined to be very effective in removing metals. The pH was consistently neutralized and nitrates were successfully removed in the mine process water. As a result of conducting these pilot-scale tests, the biotreatment process was concluded to offer an innovative, cost-effective alternative for the treatment of mining waste waters.

3:55 PM Invited

Degradation of Cyanide and Metal-Cyanide Complexes by Bacterial Consortia: *Garth James*[1]; [1]MSE Technology Applications, 920 Technology Blvd. Ste. B, Bozeman, MT 59718 USA

Bacterial consortia capable of degrading cyanide and metal-cyanide complexes were obtained from gold mine process waters using enrichment cultures. The cultures contained either cyanide, tetracyanonickelate, or ferrocyanide as a sole nitrogen source. Several bacteria were isolated from the consortia, but none of these organisms were capable of degrading cyanide or cyanide-metal complexes at rates equivalent to those of the intact consortia. The consortium from the cyanide enrichment was capable of completely degrading cyanide from an initial concentration of over 100 ppm in less than one day, when cultured using a fed-batch method. Tetracyanonickelate and ferrocyanide were degraded more slowly. Overall, these results demonstrate that bacteria capable of degrading cyanide and cyanide-metal complexes can be isolated from mine process solutions and are capable of significantly reducing cyanide concentrations under appropriate conditions.

4:20 PM Invited

Cyanide Heap Biological Detoxification: Patrick Clark[2]; *Diane M. Jordan*[1]; Tom M. Malloy[1]; [1]MSE Technology Applications, Inc., 200 Technology Way, Butte, MT 59702 USA; [2]U.S. Environmental Protection Agency, Cincinnati, OH USA

As part of the Mine Waste Technology Program (MWTP), a large-scale column test utilizing bacteria and a sulfide ore was conducted at McClelland Laboratories, Inc., in Sparks, Nevada. Four technology providers were participated in this demonstration. The sulfide ore, process effluent and make-up water was provided by a mining company in Nevada. The column for each technology provider along with a process effluent and hydrogen peroxide column were set-up side by side on a pad designed for column testing outside the laboratory building. The latter two columns were used for experimental controls. These six large-scale columns measured 22 feet in height with 4-foot outside diameters. The goal of this project was to evaluate the viability and feasibility of these biodegradation technologies to reduce the cyanide to the regulatory level of 0.2 parts per million (ppm) within an acceptable timeframe and with low operational costs. The column testing began on December 3, 1998 and operated until May 17, 1999. One technology provider reached the regulatory level within the 158 days. The remaining three biotechnology columns were approaching the regulatory limit at completion of the demonstration. The MWTP is funded by the U.S. Environmental Protection Agency (EPA) and is jointly administered through an interagency agreement with the U.S. Department of Energy (DOE) under DOE contract number DE-ACC22-96EW96405.

Defect Properties and Mechanical Behavior of HCP Metals and Alloys: Creep, Fatigue, and Fracture

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

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

Tuesday PM Room: 211
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Geoffrey W. Greenwood, University of Sheffield, Eng. Mats., Sheffield S1 3JD UK; Norman S. Stoloff, Rensselaer Polytechnic Institute, Mats. Sci. and Eng., Troy, NY 12180 USA

2:00 PM Invited

Creep Strength of Mg Based Alloys: *Kouichi Maruyama*[1]; Mayumi Suzuki[1]; Hiroyuki Sato[2]; [1]Tohoku University, Dept. of Mats. Sci., 02 Aoba-yama Aoba-ku, Sendai 980-8579 Japan; [2]Hirosaki University, Dept. Intell. Mach. and Sys. Eng., Hirosaki 036-8561 Japan

Mg based alloys are an attractive material because of their low density and excellent recyclability. One of their disadvantages is the inferior creep strength as compared with Al alloys. In this talk it will be discussed why Mg alloys have inferior creep resistance and how to improve their creep strength. Creep of Mg-Al and Al-Mg solid solution alloys were studied at 600K over a wide range of stress. Normalized creep rates (creep rate/diffusion constant) of the Mg alloys were lower than those of the Al alloys under the same normalized stress (stress/shear modulus). This fact suggests that the high diffusivity and low shear modulus of Mg are responsible for the inferior creep resistance of Mg alloys. Yttrium is one of the most effective elements in improving creep strength of Mg alloys. Strengthening mechanism of Mg-Y alloys will be discussed in conjunction with TEM observation of dislocation substructure.

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Room Temperature Creep and Deformation Mechanisms in a Ti-6wt% Al Alloy: *T. Neeraj*[1]; J. L. Robertson[2]; G. S. Daehn[1]; M. J. Mills[1]; [1]The Ohio State University, Dept. of Mats. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Oak Ridge National Laboratories, Neutron Scatter. Grp., Bldg. 7962 MS 6393, 1 Bethel Valley Rd., Oak Ridge, TN 37831-6393 USA

It is now firmly established that titanium alloys exhibit significant creep at room temperature. In commercial titanium alloys, deformation has been observed to be planar in the alpha phase at room temperature. Short-range order (SRO) of titanium and aluminum atoms was postulated as the reason for the observed planar slip. In this study, creep and deformation behavior of a Ti-6wt% Al alloy has been characterized. The presence of SRO in this alloy has been characterized, for the first time, using neutron diffraction. The diffuse APB energy created on the slip plane due to the destruction of SRO has been evaluated from dislocation structures. Further, a residual displacement fault was observed in the slip bands. This has been characterized in detail and the magnitude of which has been evaluated using quantitative image matching simulations. Finally, an attempt has been made to correlate the microstructure with the macroscopic creep response after heat treatments to modify the SRO state.

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Creep Processes in Magnesium Alloys and Their Composites: *Vaclav Sklenicka*[1]; Marie Pahutova[1]; Kveta Kucharova[1]; Milan Svoboda[1]; Terence G. Langdon[2]; [1]Academy of Sciences, Instit. of Phys. of Mats., Zizkova 22, Brno CZ-616 62 Czech Republic; [2]University of Southern California, Depts. Mats. Sci. and Mech. Eng., Los Angeles, CA 980089-1453 USA

Although extensive information is available on the creep behavior of pure polycrystalline magnesium and Mg solid solution alloys, the creep properties of more complex magnesium alloys and Mg-based composites have received only limited attention. The present study was therefore initiated to perform experiments which were conducted on representative magnesium alloys (AZ 91 and QE 22) and their composites in order to evaluate the creep properties and the mechanisms associated with flow at elevated temperatures. A comparison between the creep characteristics of squeeze-cast AZ 91 and QE 22 magnesium alloys reinforced with 20 vol.%Al2O3 short fibres and unreinforced AZ 91 and QE 22 matrix alloys shows that the creep resistance of the reinforced materials is considerably improved compared to the matrix alloys. By contrast, the investigations of the creep behaviour of a particulate 15 vol.%SiC -QE 22 composite prepared by powder metallurgy have revealed no substantial increase in the creep strength of the composite compared to the matrix alloy.

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Room Temperature Creep and Tensile Behavior of Ti-6242Si in Single Colony and Aligned Microstructures: *Joseph Tatalovich*[1]; Michael F. Savage[1]; Marc Zupan[2]; Michael J. Mills[1]; Kevin J. Hemker[2]; [1]The Ohio State University, Dept. of Mats. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]The Johns Hopkins University, Dept. of Mech. Eng., 200 Latrobe Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

Industrially relevant, alpha(hcp)/beta(bcc) titanium alloys with a large alpha volume fraction have been long known to exhibit significant primary creep strains at ambient temperatures and at stresses below their yield strength. These alloys are also observed to have over an order of magnitude deficit in low cycle fatigue life during application of a dwell at maximum stress at room temperature. The colony microstructures exhibit ambient temperature creep and tensile (constant strain rate) behavior that depends sensitively on the orientation of the colonies. Microsample testing was employed to determine the extent of these asymmetries for both prism and basal slip. Additional experiments have also been performed on aligned alpha equiaxed microstructures; the results will be compared to polycrystalline equiaxed microstructures. Scanning electron microscopy has been used to study the slip line morphology as a means to determine the deformation mechanisms.

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High-Cycle Fatigue in Titanium Alloys: *Robert O. Ritchie*[1]; Jan O. Peters[1]; Brad L. Boyce[1]; Josh P. Campbell[1]; [1]University of California, Berkeley, Mats. Sci. and Eng. Dept., 463 Evans Hall, #1760, Berkeley, CA 94720-1760 USA

High-cycle fatigue (HCF) of titanium alloys is a prime cause of military aircraft turbine engine failures. It results from fatigue-crack growth in blades and disks, initiated at small defects often associated with fretting or foreign object damage (FOD). In this presentation, the nature of fatigue in a Ti-6Al-4V blade alloy, with bimodal and lamellar microstructures, is examined under representative high frequency and high load-ratio conditions, with emphasis on behavior following FOD and under mixed-mode loading conditions. It is shown that for all crack sizes with dimensions large compared to microstructural size-scales, a worst-case fatigue threshold can be defined which represents a lower-bound stress intensity for fatigue-crack growth. This holds for mixed-mode loading (for mode-mixities between pure mode I and II), at load ratios from 0.1 to 0.8, provided the threshold is characterized under worst-case mode I conditions in terms of the strain energy release rate (incorporating both tensile and shear components). However, for crack sizes comparable with microstructural dimensions, as can be found in damaged regions due to FOD impacts, fatigue thresholds can be far lower, e.g., by a factor of two. In such instances, the critical condition for HCF in Ti-6Al-4V can be evaluated using a modified Kitagawa-Takahashi diagram, where the limiting conditions are defined in terms of the stress-

concentration corrected fatigue limit and the "worst-case" threshold.

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Effect of Yield Stress on Fracture of Hydrides in Zr-2.5Nb Alloys during Delayed Hydride Cracking: *In Sup Kim*[1]; Je Yong Oh[1]; Young Suk Kim[2]; [1]Korea Advanced Institute of Science and Technology, Nucl. Eng., 373-1, Kusong-dong, Yusong-gu, Taejon 305-701 South Korea; [2]Korea Atomic Energy Research Institute, Yusong-gu, Taejon 305-701 South Korea

In modeling of the delayed hydride cracking (DHC) velocity above the range of the threshold stress intensity, the fracture condition of hydrides has been neglected, while the diffusion of hydrogen has been emphasized. Although the diffusion of hydrogen is a dominant factor in this case, the fracture condition of hydrides must be considered to evaluate the exact DHC behavior. The yield stress can be a main factor that affects the fracture condition of hydrides. The DHC velocities in Zr-2.5Nb alloys were investigated at various yield stress ranges. The yield stress influenced the critical hydride length and the fraction of hydrides, which led to fracture. The effect of yield stress was taken into account to modify the current DHC model, and the calculated values were compared with the experimental results.

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Anisotropic Threshold Stress Intensity Factor, KIH, and Crack Growth Rate in Delayed Hydride Cracking of Zr-2.5%Nb Alloy: *Young Suk Kim*[1]; Sung Soo Kim[1]; Sang Chul Kwon[1]; [1]Korea Atomic Energy Reserach Institute, Zirconium Group, 150, Dukjin-dong, P.O. Box 105, Yusong, Taejon, ChungNam 305-353 Korea

Since delayed hydride cracking (DHC) is known to occur by the fracturing of reoriented hydrides, the initiation of a DHC crack would depend strongly on the orientation of a hydride habit plane where the hydrides can preferentially precipitate. When the cracking plane is in accordance with the hydride habit plane, the initiation of DHC will be easy, leading to a lower threshold stress intensity factor, KIH, necessary for the initiation of DHC. To confirm this view, we investigated the crack growth pattern and KIH in Zr-2.5Nb alloy with a circumferential texture by changing the initial notch direction from the longitudinal direction to the circumferential one. The initiation and growth of the DHC crack occurred only on hydride habit planes, {10.7}. The threshold stress intensity factor, KIH was discussed phenomenologically based on the crack growth pattern and analytically as a function of the tilting angle of the hydride habit plane to the cracking plane. In addition, a supplementary experiment was conduc ted to demonstrate the linear dependence of KIH on the basal pole component in the cracking plane. Though there is the same extent of the basal pole component in the cracking plane, DHC tests showed that the crack growth rate of Zr-2.5Nb tubes varies with the crack growth direction, being two times faster in the longitudinal direction than that in the radial direction. To gain an understanding of this anisotropic DHC behavior, tensile tests were additionally conducted on small specimens with a gauge length of about 2 mm, taken from three directions of the tube. The work hardening rate was found to be higher in the axial direction rather than in the radial direction. A change in texture before and after DHC tests was also confirmed suggesting that part of applied stress is relaxed in inducing the twinning. Thus, the anisotropic growth rate of the DHC crack in Zr-2.5Nb tubes with the direction was discussed based on different work hardening rate and textural change with the direction, and the distribution of hydride habit planes {10.7}.

Defect Properties and Mechanical Behavior of HCP Metals and Alloys: High-Rate Deformation and Hot Working

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 Div., 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

Tuesday PM — Room: 212
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: G. (Rusty) T. Gray, Los Alamos National Laboratory, MST-8, Los Alamos, NM 87545-9021 USA; Sean R. Agnew, Oak Ridge National Laboratory, Met. and Ceram. Div., Oak Ridge, TN 37831-6115 USA

2:00 PM Invited

Effects of High Rates of Loading on the Deformation Behavior and Failure Mechanisms of HCP Metals and Alloys: *K. T. Ramesh*[1]; [1]The Johns Hopkins University, Mech. Eng., 3400 N. Charles St., Baltimore, MD 21218 USA

A substantial amount of work has been performed on the effect of high rates of loading on the deformation and failure of fcc and bcc metals (although less so on pure bcc metals). It is known, for example, that the strengths of these materials are typically significantly higher at high strain rates (>1e3/s), and that alloying content typically reduces the rate sensitivity. In contrast, the influence of strain rate and temperature on the flow stress of hcp metals has received relatively little attention. The low symmetry of these materials and the development of twinning leads to a particularly rich set of potential mechanisms at high rates. Understanding these mechanisms becomes important as hcp metals (particularly titanium) are increasingly used in applications such as ballistic impact. Further, high-speed machining of hcp metals is an upcoming manufacturing problem. The results of high-strain-rate deformation and dynamic failure studies on titanium, Ti-6Al-4V and hafnium are presented. Strain rates as high as 1e5/s are considered. Adiabatic shear localization and void nucleation, growth and coalescence are found to be ubiquitous dynamic failure mechanisms in these metals. Both microscopic characterization and macroscopic constitutive behavior are investigated. The constitutive modeling of twinned structures is of particular interest.

2:30 PM

The Role of Twinning on the High Strain Rate Compression Response of Beryllium: *William R. Blumenthal*[1]; Carl R. Necker[1]; Robert D. Field[1]; Stephen P. Abeln[1]; George T. Gray[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Tech. Div., Mail Stop G-755, Los Alamos, NM 87544 USA

The compressive stress-strain response of polycrystalline beryllium was studied as a function of crystallographic texture and test temperature at high strain rates. The mechanical response was correlated to microstructural and texture measurements. Commercial grade vacuum hot-pressed beryllium with near-random texture and highly textured rolled-sheet beryllium were tested in several principal orientations using a split-Hopkinson pressure bar apparatus. X-ray diffraction polefigures were measured before and after high rate compressive deformation. Optical metallography revealed deformation twinning to be a key deformation mechanism. The strain-hardening behavior was strongly dependent on the temperature (77K to 800K), degree of initial texture, and on the activation of a single twinning deformation system. Twinning deformation could be completely suppressed in the rolled beryllium by orienting the compression axis with the through-thickness direction. These

results are necessary to validate advanced constitutive models that incorporate texture evolution and twinning deformation mechanisms.

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Microstructural Characteristics and Spatial Distribution of Shear Bands in Titanium and Ti-6Al-4V Alloy: *Qing Xue*[1]; Vitali F. Nesterenko[1]; Marc A. Meyers[1]; [1]University of California, San Diego, Dept. Mech. and Aero. Eng., 9500 Gilman Dr., La Jolla, CA 92093-0418 USA

The Evolution of multiple adiabatic shear bands was investigated in commercial pure titanium(CP Ti) and Ti-6Al-4V alloy through the radial collapse of a thick-walled cylinder under high-strain-rate deformation (~10e4/s). The shear-band initiation, propagation, as well as spatial distribution were examined under different effective strains. The evolution of the shear band pattern during the deformation process reveals self-organization characteristics. Three principal mechanisms are considered in initiation: (a) momentum diffusion by stress unloading, (b) perturbation in the stress/strain/temperature fields, and (c) microstructural inhomogeneities. The shear bands nucleate at the internal boundary of the specimens and construct a periodical distribution at early stage. The interaction between shear bands due to the unloading control the growth of shear bands. Some shear bands slow down and even stop during the interaction. The propagating shear bands compete to create a new spatial distribution. A discontinuous growth mode for shear localization under periodic perturbation is proposed. Self-organized initiation and propagation modes are discussed in relation to the interaction among the shear bands. The evolution of the spacing of shear bands is associated with different growth stages, which are dictated by competitive mechanisms. The damages and microstructural characteristics were also discussed.

3:10 PM

Damage Accumulation and the Onset of Adiabatic Shear in Incipiently Failed Ti-6Al-4V Under High-Rate Loading: *Duncan A. Macdougall*[1]; William R. Thissell[1]; [1]Los Alamos National Laboratory, MST-8, MS-G755, Los Alamos, NM 87545 USA

Damage accumulation entails the progressive nucleation, growth, and coalescence of voids and micro-cracks. These features can often culminate in failure surface formation. However, certain materials such as Ti-6Al-4V, have a propensity to fail prematurely in a diabatic shear. A mechanistic understanding of both the kinetics of damage accumulation and the conditions under which shear localization may occur are important for developing a damage model to simulate this behavior. Aerospace grade Ti-6Al-4V was investigated in this study. Incipient and full failure tests were performed using a momentum-trapped tensile Hopkinson bar at strain rates from 500-4000 1/s. The specimens used included cylindrical uniaxial stress and notched geometries to explore stress states from 0.17 <-P/2t <1.5 (P is hydrostatic stress and t is shear stress). The recovered samples were sectioned, and metallographically analyzed. The resulting damage was quantified using image analysis and optical profilometry. The initiation of shear localization was noted in some specimens.

3:30 PM Break

3:50 PM Invited

Microtructure Evolution During Hot Working of Titanium Alloys: *S. L. Semiatin*[1]; T. R. Bieler[2]; [1]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St. Ste. 1, Wright-Patterson Air Force Base, OH 45433-7817 USA; [2]Michigan State University, East Lansing, MI 48824-1226 USA

The evolution of microstructure during hot working of alpha-beta titanium alloys will be discussed. Attention will focus on the breakdown of the colony alpha microstructure to obtain an equiaxed one. Experimental and modeling work to deduce the slip systems in the alpha phase at hot-working temperatures and the micromechanisms of globularization will be reviewed. The influence of the formation of shear bands within alpha-phase platelets and slip transfer across alpha-beta interfaces on the kinetics of structure evolution and observed flow phenomenology (e.g. flow-softening behavior and the variation of rate sensitivity with strain)will be described.

4:20 PM

Evolution of Microstructure during Hot Deformation of Zirconium Alloys: *J. K. Chakravartty*[1]; G. K. Dey[1]; S. Banerjee[1]; [1]Bhabha Atomic Research Centre, Mats. Sci. Div., Mumbai 400 085 India

The hot deformation characteristics of zirconium base alloys have been studied by constructing deformation processing maps which describe the variation of strain rate sensitivity ($m=\delta \ln\sigma/\delta \ln\varepsilon$) with temperature and strain rate. The domains within these maps have been correlated with specific microstructural processes that occur during hot deformation by metallographic investigations and kinetic analysis. Various deformation mechanisms like dynamic recrystallization (DRX), dynamic recovery and super plasticity have been identified in the alloys studied depending on the strain rate, temperature and alloy composition. The substructures formed during deformation have been examined by transmission electron microscopy. The dislocation configurations associated with the substructures during dynamic recovery and DRX have been characterized. Kinetic analysis have been performed to identify the rate controlling process for dynamic recrystallization. TEM investigations of super plastically deformed material did not reveal any substructure within equiaxed grains. However some dislocation interactions have been observed in regions near to grain boundaries.

4:40 PM

Micromechanisms of Deformation and Fracture of Titanium Alloys Under Shock Loading: *Svetlana Atroshenko*[1]; [1]Institute for Problems of Mechanical Engineering RAS, Lab. Phys. of Fract., V.O., Bolshoy 61, St. Petersburg 199178 Russia

Shock loading was conducted with a 37 mm bore-diameter light gas gun. Backside spallation was realized under uniaxial strain conditions within the velocity range 200-650 m/s by impactor of thickness 1-3 mm. Specimens-targets were in the form of discs 52 mm in diameter and with a thickness of 10 and 5 mm. The experiments were carried out on hexagonal closed packed (HCP) titanium alloy and two-phase HCP and body cubic centered (BCC) one. BCC carbon and armour steels were studied for comparison. In order to investigate the localization deformation processes on the mesoscopical scale level, the method of registration grids was used. These grids have been drawn before shock loading on the preliminary polished halves of the target which after that have been joined in special mandrel and have been subjected to deformation. The specimen surface with drawn grid was placed along the shock wave propagation direction. The grid was drawn by diamond pyramid with the help of microhardness device PMT-3.

Hume Rothery Award Symposium - Electronic Structure and Alloy Properties: Applications

Sponsored by: Electronic, Magnetic & Photonic Materials Division; Structural Materials Division

Program Organizers: Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA

Tuesday PM — Room: 202
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Josef Kudrnovsky, Inst. of Phys., Prague, Czech Republic; Julie B. Staunton, University of Warwick, Coventry, UK

2:00 PM Invited

Electronic Structure and Thermodynamics of Point Defects in Binary Intermetallics: *Pavel A. Korzhavyi*[1]; B. Johansson[1]; [1]Royal Institute of Technology, Dept. of Mats. Sci. and Eng., Brinellvagen 23, Stockholm 100 44 Sweden

The electronic structure and formation energies of native defects (antisite atoms and vacancies) in NiAl and Ni_3Al intermetallic compounds are calculated by means of the locally self-consistent Green's function method. The equilibrium defect concentrations are studied

within the Wagner-Schottky model of a lattice gas of non-interacting defects. A complete analysis of possible solutions to the Wagner-Schottky model is performed. We show that the type of constitutional and thermal disorder in a binary compound is completely determined by two parameters which are certain dimensionless combinations of the point defect energies. These parameters are used to construct two-dimensional maps showing the domains of possible types of disorder for different compositional regions of the compound. The theory is applied toNiAl and Ni_3Al.

2:20 PM Invited

Quasiparticles in d-Wave Superconductors Within Density Functional Theory: *Zdzislawa Szotek*[1]; Balazs Gyorffy[2]; Walter Temmerman[1]; Ole K. Andersen[3]; Ove Jepsen[3]; [1]Daresbury Laboratory, Compu. Sci. and Eng. Dept., Daresbury, Warrington WA4 4AD UK; [2]University of Bristol, H.H. Wills Phys. Lab., Tyndall Ave., Bristol BS8 1TL UK; [3]Max-Planck-Institut fuer Festkoerperforschung, Postfach 800665, Stuttgart D-70506 Germany

We present a semiphenomenological approach to calculating the quasiparticle spectra of high temperature superconductors. It is based on a particularly efficient parametrization of the effective electron-electron interaction afforded by the density functional theory for superconductors and a tight-binding-linearized-muffin-tin-orbital scheme for solving the corresponding Kohn-Sham-Bogoliubovde Gennes equations. We apply this methodology to YBa2Cu3O7 (YBCO) and illustrate its potential by investigating a number of site and orbital specific, but otherwise phenomenological models of pairing in quantitative detail. We compare our results for the anisotropy of the gap function on the Fermi surface with those deduced from photoemission experiments on single crystals of YBCO. Also, the low temperature specific heat and penetration depth are calculated and compared with measurements. We investigate the doping dependence of the superconducting gap, transition temperature, T_c, and penetration depth. We present new evidence that the Van Hove-like scenario is an essential feature of superconductivity in the cuprate superconductors. Since our description of pairing is semiphenomenological, we shed new light on the physical mechanism of pairing only indirectly and conclude, provisionally, that the dominant pairing interaction operates between electrons of opposite spins, on nearest neighbour Cu sites in d_x2-y2 orbitals.

2:40 PM Invited

Computer Simulations of Self Assembled Monolayers: *Friederike Schmid*[1]; Dominik Duechs[1]; Christoph Stadler[2]; [1]Universitaet Bielefeld, Fakultaet fuer Physik, Universitaetsstrasse 25, E5-114, Bielefeld D33615 Germany; [2]Universitaet Mainz, Institut fuer Physik, Staudingerweg 7, Mainz D 55099 Germany

Self assembled monolayers of organic molecules on solid substrates can be used to design well-defined surfaces, which can be structured efficiently on a nanometer scale. The molecular order of the molecules, in particular their tilt with respect to the surface, is believed to play an important role for the stability of those patterns. We discuss Monte Carlo simulations of a simple, idealized model for self assembled monolayers, with particular emphasis on the different types of tilt order and tilting transitions.

3:00 PM Break

3:20 PM Invited

The Hume-Rothery Size Rule and Double-Well Microstructures in Gold-Nickel: *Jack S. Kirkaldy*[1]; Kamal Janghorban[1]; George C. Weatherly[1]; [1]McMaster University, Brockhouse Instit. for Matls. Rsch., 1280 Main St. W., ABB 430, Hamilton, ON L8S 4M1 Canada

The miscibility gap and high critical Tc (~860°C) in Au-Ni arises primarily due to the Goldschmidt radius of Au being 1.15 times that of Ni. While early microstructure observed on down-quenching to ~350°C favoured coarse lamellar grain boundary and bulk nucleated near-spherical faceted lamellar colonies, modulated patterns of 1-5 nm were also observed below that in competition to at least 100°C. Our current observations on pre-quenched samples of 25-75 at% Ni have revealed 3-dimensional modulations (~10 nm) on the TEM hot stage up to 500°C which develop L1o ordering at the nodes, and this complex microstructure offers nucleation sites for short-lived single crystal lamellar colonies ~100 nm in the bulk. A dendritic-like instability then ensues which ultimately accommodates to the aforementioned near-spherical faceted colonies, evidently retaining some form of single crystal propensity, as for example a twinning relationship. The 75% alloy generates a metastable lamellar product of L1o order at 50:50 and nearly pure nickel. Ginzburg-Landau-Hillert-Maugis chemical reaction theory encompasses all aspects of the microstructure including the absence of a coherent critical point and spinodal. The Cahn diffusion theory expresses a number of inconsistencies. The role of vacancy and electronic correlation effects are briefly discussed.

3:40 PM Invited

Development of Electronic Phase Diagram for Plutonium-Gallium Alloys: *David E. Dooley*[1]; David L. Olson[2]; Frank E. Gibbs[3]; Glen R. Edwards[2]; [1]Los Alamos National Laboratory, NMT-16, P.O. Box 1663, MS E574, Los Alamos, NM 87545 USA; [2]Colorado School of Mines, Metall. and Mats. Eng., 1500 Illinois St., Golden, CO 80401 USA; [3]Kaiser-Hill LLC, Remediation Industrial Site Services, 10808 Highway 93, Golden, CO 80403-8200 USA

With the continuation of effort to understand and predict plutonium alloy stability, it is important to revisit the empirical electronic concepts and analytical methodologies of the 1960's. Hume, Rothery, Gschneider, Waber, Brewer, Miedema and others introduced approaches to make electronic correlations to crystal phase structure and phase transitions. Even though these models are qualitative by today's analytical and measuring practices, they bridged the essential gap between electronic interactions, solution thermodynamics, and phase diagram construction. The chemical potential of an element is constant within a two-phase region and is related to Gibbs free energy. The electron concentration is introduced into the analysis as a variable within the enthalpy and entropy terms of the free energy equation. Knowing the electron concentrations for each phase and incrementing on temperature, the composition of the phase boundary is determined. This paper describes and displays how an equilibrium phase diagram for plutonium-gallium is constructed from thermodynamic and electronic properties.

4:00 PM Invited

Large Thermal Softening of the Phonon Density of States of Uranium: *Michael E. Manley*[1]; Brent Fultz[2]; Robert J. McQueeney[1]; [1]Los Alamos National Laboratory, LANSCE-12, MS H805, Los Alamos, NM 87545 USA; [2]Caltech, Mats. Sci., MS 138-78, Pasadena, CA 91125

Time-of-flight inelastic neutron scattering spectra were measured on uranium at several temperatures from 50K to 1213K that include its three crystalline phases. Phonon density of states curves were extracted. A large and continuous decrease in phonon energies with increasing temperature was observed over the entire temperature range of the alpha-phase. Power spectrum analysis suggests that the phonon softening in the high temperature classical limit comes from continuous harmonic softening. Thus, rather than the usual anharmonic type phonon softening, the phonon softening comes mainly from a change in the electronic contribution to the phonon potentials. Phonon densities of states measured near the alpha-beta and the beta-gamma transition indicated vibrational entropy changes of (Sbeta-Salpha)vib= (0.15±0.01) kB/atom and (Sgamma-Sbeta)vib= (0.36±0.01) kB/atom. The former makes up about 35% and the latter 65% of the total entropy change determined from the latent heats. The remaining entropy must be electronic.

4:20 PM Invited

Simple Rules for Determining the Valencies of f-Electron Systems: Leon Petit[2]; Axel Svane[2]; Zdzislawa Szotek[1]; Paul Strange[3]; Hermann Winter[4]; *Walter M. Temmerman*[1]; [1]Daresbury Laboratory, Compu. Sci. and Eng. Dept., Daresbury, Warrington WA4 4AD UK; [2]University of Aarhus, Inst. of Phys. and Astron., Aarhus DK-4000 Denmark; [3]Keele University, Dept. of Phys., Keele, Staffordshire ST5 5DY UK; [4]Forschungszentrum Karlsruhe, INFP, Postfach 3640, Karlsruhe D-76021 Germany

The electronic structure of f-electron systems is calculated with the self-interaction corrected local-spin-density (LSD) approximation. This scheme allows for a splitting of the f electron manifold into an integral number of localised and band electrons. Therefore,

in comparison with the LSD where all f states are pinned at the Fermi energy, only maximum one f band is left at the Fermi energy. This band will be partially occupied with occupancy n_f, and the f-electron fluctuations will be reduced compared with the LSD. When n_f exceeds a critical value of approximately 0.6 it becomes energetically more favourable to localise this state and the valency is reduced by one. These results can also be rationalised on the basis of the occurence of a d to f electron promotion.

4:40 PM Invited
What is the Ground State of Al_2Cu?: *Chris Wolverton*[1]; Vidvuds Ozolins[2]; [1]Ford Motor Company, Phys. Dept., MD 3028/SRL, P.O. Box 2053, Dearborn, MI 48176 USA; [2]Sandia National Laboratories, Livermore, CA USA

Theories of structural stability in ordered intermetallics are historically formulated in terms of energetic effects (e.g., electron-per-atom ratio, strain accomodation, size-effects, and electronegativity differences), whereas entropic effects are typically ignored in these theories. First-principles total energy calculations at T=0K incorporate all these energetic contributions, yet surprisingly predict the energy of the observed Al_2Cu-Θ phase to be above that of its metastable counterpart, Θ'. We show that vibrational entropy reverses this energetic preference at finite temperature, and hence is responsible for stabilizing the equilibrium phase. Thus, in contrast to conventional metallurgical wisdom, we assert that the ground state of Al_2Cu is not Θ, but rather Θ'. We discuss the implications of this rather surprising prediction.

5:00 PM Invited
Multiple Scattering Theory in Clean Superconducting Layered Structures: *Adri Lodder*[1]; Roland E.S. Otadoy[1]; Rutger T.W. Koperdraad[1]; [1]Free University, Phys. and Astron., De Boelelaan 1081, 1081 HV Amsterdam 1081 HV, The Netherlands

An exact expression is derived for the matrix Green's function of a clean superconducting layered structure with an arbitrary number of interfaces. A multiple scattering approach is employed, in which the interfaces act as scattering centers. The theory is applied to systems with transverse dimensions which vary from very narrow to wide. In determining the gap parameters of the superconducting parts selfconsistently, it comes out that for transverse dimensions smaller than about twenty percent of the superconducting coherence length superconductivity is suppressed. The local density of states is calculated for SNS and SNSNS junctions. For critical values of the transverse dimensions the exact results exhibit a clear lift of the degeneracy in the Andreev bound states energies, typical for results obtained in the frequently used Andreev approximation.

Emerging Technologies for Metals Production II

Sponsored by: Extraction & Processing Division, Light Metals Division, Aluminum Committee, Process Fundamentals Committee, TMS Young Leaders Committee
Program Organizers: Samuel A. Davis, TIMET, Henderson, NV 89009 USA; Toni Marechaux, US Dept. of Energy, Office of Industrial Technology, Washington, DC 20585-0121 USA; Thomas P. Robinson, US Department of Energy, Office of Industrial Technology, Washington, DC 20585-0121 USA

Tuesday PM Room: 221
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Toni Grobstein Marechaux, US Department of Energy, Ofc. of Indust. Techn., Washington, DC 20585 USA

2:00 PM Opening Remarks

2:05 PM
Magnesium Technologies, Present and Future: *Robert E. Brown*[1]; [1]Magnesium Assistance Group, Inc, 226 Deer Trace, Prattville, AL 36067-3806 USA

Magnesium was discovered by Davy in 1808. The production processes as they exist are not competitive economically with aluminum. The electrolyic magnesium process is divided into two steps, one to make the $MgCl_2$ feed and the other to apply large amounts of electric current to dissociate the feed into magnesium (450 g) and chlorine (1200 g). The production of fully anhydrous, pure cell feed is a major problem. Recently there has been a growth of thermal processes, mainly the Pidgeon silicothermic production process using FeSi to reduce dolomite in horizontal retorts. Pechiney has a large vertical electric furnace process that also uses FeSi to reduce calcined dolomite in a molten bath of alumina. Work is proceeding to develop new or modified processes that will greatly improve magnesium production. The present methods and the research and R&D will be reviewed in this paper.

2:35 PM
Radical Innovation in Magnesium Smelting--An Assessment of Unexplored Chemistries: *Donald R. Sadoway*[1]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. & Eng., 77 Massachusetts Ave., Room 8-109, Cambridge, MA 02139-4307 USA

Today magnesium is produced either by molten chloride electrolysis or by metallothermic reduction. While both existing technologies could benefit from improvements, it is unlikely that these will lead to dramatic reduction in the price of the metal. As an exercise in assessing the prospects for radical innovation an analysis of unexplored approaches has led to a "short list" of candidate chemistries. These will be presented along with a discussion of the technical obstacles that must be overcome if a new technology is to emerge.

3:05 PM Break

3:25 PM
Bismuth and Tellurium Used for Thermoelectric Materials: *Funsho Ojebuoboh*[1]; [1]ASARCO, Inc., 495 E. 51st Ave., Denver, CO 80216 USA

Bismuth and tellurium are key constituent metals in the fabrication of thermoelectric modules. Tellurium and bismuth each account for more than 50% of thermoelectric materials on a weight as well as molecular fraction basis. Therefore, impurities in either of these raw materials can be a significant factor in the yield of the compound materials and the performance of modules. The Globe unit of Asarco, Inc. is a key supplier of many high-purity metals. In addition to bismuth and tellurium, the other constituent metals, antimony and selenium are supplied by Globe to thermoelectric businesses. The purpose of this presentation is to review the processes involved in the purification and production of these metals to meet preferred specification.

3:45 PM
Production of Nanocrystalline Metal Powders in Electric Arcs: *Brent A. Detering*[1]; [1]Idaho National Engineering Laboratory, Indust. & Mat. Techn., MS 2210, P.O. Box 1625, Idaho Falls, ID 83415-2210 USA

High temperature electric arcs were used to produce nanocrystalline powders of refractory metals, including titanium and vanadium. Electric arcs have the process advantages of high temperature and energy density, control of reaction conditions (oxidizing, reducing, or inert), small size, and rapid product cooling. These properties were used to develop unique processes for the production of metal and metal alloy powders. Electric arc temperatures were determined and the results used to build computational fluid flow models of the production process. Additionally, these processes were evaluated and refined using equilibrium chemistry models. The advantages, disadvantages, and economic evaluation of these processes will be discussed.

High Temperature Coatings - IV: Interdiffusion of Coatings

Sponsored by: Materials Processing and Manufacturing Division, ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee, Surface Engineering Committee
Program Organizers: Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Janet Hampikian, GA Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Tuesday PM Room: 219
February 13, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: John E. Morral, University of Connecticut, Inst. of Mats. Sci., Storrs, CT 06268-3136 U.S.A.; Yong-ho Sohn, University of Central Florida, Dept. of Mech., Orlando, FL USA

2:00 PM Invited
Interdiffusion Behavior in Aluminide Coated Nickel-Based Alloys at 1150°C: *Brian Gleeson*[1]; Eddie Basuki[2]; Alan Crosky[3]; [1]Iowa State University, Dept. Mats. Sci. & Eng., 3157 Gilman Hall, Ames, IA 50014 USA; [2]Institute Teknologi Bandung, Corrosion Centre, Inst. for Res., Bandung 40132 Indonesia; [3]University of New South Wales, Sch. of Mats. Sci. & Eng., Sydney, NSW 2052 Australia

The interdiffusion behavior between aluminum-rich β-NiAl coatings and nickel-based alloy substrates will be discussed. Tests were conducted at 1150°C for up to 6500 min. The resulting microstructural changes basically involved two major sequential stages: β homogenization causing coating enlargement followed by β recession. In the early stages of homogenization, the inward diffusion of chromium from the interdiffusion zone into the substrate and the outward diffusion of nickel from the substrate into the coating induced the formation of a β-NiAl+γ-Ni region at the coating/substrate interface. Moreover, chromium exhibited uphill diffusion in the Ni-rich β region of the coating during the early stages of homogenization. With time, the outer β-layer completely homogenized, and the influence of chromium diffusion decreased whilst the outward diffusion of nickel and inward diffusion of aluminum became predominant. Further depletion of chromium and aluminum, coupled with nick el enrichment, caused the β to transform to γ'-Ni3Al. The compositions within the interdiffusion zone changed with time but were in general accordance with the equilibrium Ni-Cr-Al phase diagram. Interpretation of the complex interdiffusion behavior was aided by the application of diffusion paths.

2:25 PM
Diffusional Reactions in High Temperature Coatings: *John Ågren*[1]; [1]Royal Institute of Technology, Mats. Sci. and Eng., KTH, SE-100 44 Stockholm, Sweden

The combination of a multicomponent coating on a muticomponent substrate alloy is a very complex system involving not only traditional multicomponent diffusion effects, like up-hill diffusion, but also difficult questions on the shape and stability of the diffusion path in a multi-component multi phase system. A satisfactory understanding serving as a basis for coating engineering can be achieved by a combined thermodynamic and diffusion kinetic approach. The long-term research within the field in our and other groups will be briefly reviewed and some scientific challenges will be discussed in more detail.

2:45 PM
Analyzing High Temperature Coating Interdiffusion: *J. E. Morral*[1]; [1]University of Connecticut, Metall. and Mats. Eng., 97 N. Eagleville Rd., Box U-136, Storrs, CT 06269-3136 USA

During the processing or service of high temperature coatings, elements from the coating and substrate can mix or react to form new phases by a diffusional mechanism. The resulting interdiffusion microstructure can be characterized by microprobe and image analysis. Combining concentration profiles for each phase with volume fraction profiles yields a diffusion path that illustrates the composition changes caused by interdiffusion. The diffusion path, in combination with a phase diagram, can then be used to interpret why a particular microstructure has formed and possibly how to alter the microstructure by modifying the coating or substrate chemistry.

3:05 PM
Predicting Interdiffusion Microstructures using the Phase Field Approach: Kaisheng Wu[1]; *Yunzhi Wang*[1]; John E. Morral[2]; [1]The Ohio State University, Mats. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA; [2]University of Connecticut, Metall. and Mats. Eng., Box U-136, 97 North Eagleville Rd., Storrs, CT 06269-3136 USA

At high temperatures the interdiffusion between two materials that are in intimate contact can lead to very complicated interdiffusion microstructures, as observed, for example in bond coatings on Ni-based superalloy turbine blades. In previous work, microstructural details including diffusion taking place in phases other than the continuous matrix have been ignored. This could be a serious drawback considering the very high volume fraction of second phase particles encountered in superalloys. In this presentation, a phase field approach to the interdiffusion problem in high temperature coatings will be presented where the dynamic evolution of interdiffusion microstructure and its effect on interdiffusion will be explicitly considered. Diffusion is allowed in all coexisting phases and different atomic mobilities are assumed for different components. The model has been validated against analytical solutions for simple cases and has been applied to various diffusion couples of a prototype ternary system.

3:25 PM Break

3:40 PM
Formation of MoSi2 Coatings From Co-Deposited SiC and Mo: Frank M. Kustas[1]; *Brajendra Mishra*[2]; Jinhui Zhou[2]; [1]Engineered Coatings, Inc., Parker, CO USA; [2]Colorado School of Mines, Metall. & Mats. Eng., 1500 Illinois St., Golden, CO 80401 USA

Molybdenum disilicide (MoSi2) is an oxidation-resistant coating used for thermal barrier applications. Unfortunately it suffers from low damage tolerance and toughness, which has restricted its use. In this paper, an alternate method to fabricate thin films of MoSi2 is presented. Using a co-deposition method, silicon carbide (SiC) and molybdenum (Mo) were co-deposited using bias-assisted unbalanced magnetron sputtering and then subsequently reacted at elevated temperatures to induce the formation of MoSi2. Films were fabricated using different working gas (i.e., Ar) pressures, percentages of Mo, and bias levels to investigate the effects of these parameters on the resultant film microstructure. Adhesion, structure (phases, composition), and nanoproperties (i.e., hardness and modulus) were measured for both as-deposited and reacted films. It was observed that working gas pressure, during co-deposition of SiC and Mo, is a critical parameter that influences coating microstructure and properties.

4:00 PM Invited
Synthesis of Hf-Doped CVD-NiAl Coatings by Sequential and Continuous Doping Procedures: *Woo Young Lee*[1]; Gi Youl Kim[1]; Limin He[1]; Justin Meyer[1]; [1]Stevens Institute of Technology, Dept. of Chem., Biochem., and Mats. Eng., Burchard Bldg. 308B, Hoboken, NJ 07030 USA

A laboratory-scale CVD reactor was used to study "sequential" and "continuous" Hf doping procedures. The sequential procedure, which consisted of "hafnizing" the surface of a single crystal Ni superalloy with HfCl4 and H2 followed by aluminizing with AlCl3 and H2, resulted in significant Hf incorporation through the formation of Hf-rich precipitates. However, these precipitates acted as diffusion barriers to retard the subsequent growth of the NiAl coating matrix. In contrast, the continuous procedure, in which HfCl4 and AlCl3 were simultaneously introduced with H2, required rather high HfCl4 concentrations to dope the coating near the apparent solubility limit of Hf in the NiAl matrix. The segregation of Hf and

the formation of a thin Ni3Al layer (about 0.5 microns) at the coating surface were consistently observed for the continuous doping experiments. These observations suggested that: (1) the coating growth occurred at the interface between the Ni3Al layer and the NiAl coating matrix and (2) the inc orporation of Hf into the growing NiAl matrix was most likely dictated by the difference in Hf solubility between the Ni3Al and NiAl phases.

4:25 PM

A New Analysis for the Determination of Ternary Interdiffusion Coefficients for Ni-Cr-Al and Fe-Ni-Al Alloys: *Yong-ho Sohn*[1]; Mysore A. Dayananda[2]; [1]University of Connecticut, Dept. of Metall. and Mats. Eng., 97 North Eagleville Rd., U-136, Storrs, CT 06269 USA; [2]Purdue University, Sch. of Eng., 1289 MSEE Bldg., West Lafayette, IN 47907-1289 USA

An analysis based on the direct calculation and integration of interdiffusion fluxes from the concentration profiles of a single diffusion couple is presented for the determination of ternary interdiffusion coefficients over selected composition ranges. On the basis of the new analysis ternary interdiffusion coefficients are determined from the concentration profiles of selected Ni-Cr-Al and Fe-Ni-Al diffusion couples. These coefficients are compared with those determined by other techniques, such as the Boltzmann-Matano analysis, the Krishtal method and the square root diffusivity method, which require two diffusion couple experiments characterized by intersecting diffusion paths. The advantages and limitations of the new analysis are discussed.

4:45 PM

Comparison of the Cyclic Oxidation Resistance of Platinum Aluminum and NiCoCrAlY Coatings and the Effect of Pretreatments on the Oxidation of these Coatings: *N. M. Yanar*[1]; G. H. Meier[1]; F. S. Pettit[1]; [1]University of Pittsburgh, Mats. Sci. Dept., 848 Benedum Hall, Pittsburgh, PA 15261 USA

Two important coatings used to protect nickel base superalloys and as a part of thermal barrier systems are platinum modified aluminide coatings and NiCoCrAlY coatings. The cyclic oxidation resistance of these coatings on the superalloy Rene N5 will be compared at 1100°C. The effects of elements in superalloy substrates on the oxidation of these coatings will be discussed, and the use of pretreatments, such as polishing and preoxidation at low oxygen pressures, to improve oxidation behavior will be described. It will be shown that substrate elements, especially tungsten and tantalum, diffuse out into the coatings along grain boundaries and affect the oxidation of the coatings.

5:05 PM

In-Situ Processing of Nickel Aluminide Coatings on Steel Substrates: Rajesh Ranganathan[1]; Olga Vayena[2]; *Teiichi Ando*[1]; Haris Doumanidis[2]; Craig Blue[3]; [1]Northeastern University, Mech., Indust. and Manuf. Dept., 334, Snell Engineering Bldg., 360, Huntington Ave., Boston, MA 02115 USA; [2]Tufts University, Mech. Eng., 204, Anderson Hall, 200 College Ave., Boston, MA 02155 USA; [3]Oak Ridge National Laboratory, 1 Bethel Valley Rd., Bldg. 4508, Mail Stop 6083, Oak Ridge, TN 37831 USA

The feasibility of producing nickel aluminide coatings on steel substrates by controlled reactive thermal processing of pre-plated precursors was studied. The basic procedure comprises plating of a steel substrate surface with nickel and aluminum and controlled partial melting of the precursor to cause in-situ formation of nickel aluminides in the resultant coating. Different heat sources including NdYAG laser, plasma arc, and infrared radiation were tested. The heating conditions were controlled for uniform heating and optimum coating microstructure by a real-time, distributed-parameter thermal control system using feedback from aninfrared pyrometer.

International Symposium on Deformation and Microstructure in Intermetallics: Microstructure

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

Tuesday PM — Room: 220
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Ronald Gibala, University of Michigan, Ann Arbor, MI 48109-2136 USA; Bimal Kad, University of California, San Diego, CA USA

2:00 PM Invited

Stacking Fault Energy and Yield Stress Asymmetry in Molybdenum Disilicide: *Terence E. Mitchell*[1]; Michael I. Baskes[1]; Shao-Ping Chen[1]; John P. Hirth[1]; Richard G. Hoagland[1]; [1]Los Alamos National Laboratory, MST-8, MS-K765, Los Alamos, NM 87545 USA

Stacking fault energies in MoSi2 due to shear along <331> have been calculated by ab initio and modified embedded atom method (MEAM) calculations. The results are used to investigate the configuration and mobility of 1/2<331> dislocations. Shear of 1/6<331> in the {103} plane of MoSi2 produces an anti-phase boundary (APB) whose geometry, called APB(1), is different from that produced by 1/6<331> in the opposite direction, APB(2). MEAM calculations show that APB(1) is stable while both types of calculations show that APB(2) is unstable. Both ab initio and MEAM calculations show that there is a stable fault close to APB(2) with a displacement of ~1/8<331> in the same direction. The {103} planes have an unusual five layer stacking sequence with successive planes offset by 1/5<301>. Shear of 1/10<351> in the correct direction gives a low energy fault. This vector is close to the 1/8<331> shear that produces a stable fault. Various dissociated configurations of 1/2<331> dislocations are considered based on these p artials. All can have asymmetrical arrangements which will respond differently to the direction of the applied stress, explaining the yield stress asymmetry in MoSi2.

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Dislocations in the Mo5SiB2 T2 Phase: *R. D. Field*[1]; D. J. Thoma[1]; J. C. Cooley[1]; F. Chu[1]; [1]Los Alamos National Laboratory, MST-6, Mail Stop G770, Los Alamos, NM 87545 USA

The T2 phase, which has a broad range of solubility around the Mo5SiB2 stoichiometry, has received considerable attention recently as a potential constituent in Mo-Si based alloys for high temperature structural applications. This ternary intermetallic compound has a body-centered tetragonal Cr5B3 structure (I4/mcm), with the c lattice parameters approximately 2x the a parameter (a=0.600nm, c=1.103nm). In this study, dislocations in T2 phase material were investigated in the transmission electron microscope (TEM). Examples of the two shortest possible Burgers for the structure, <100> (0.600nm) and 1/2<111> (0.696nm) were observed. Details of the analysis will be discussed as well as implications for general plasticity in the material. Support of DOE-OBES, Division of Materials Sciences is gratefully acknowledged.

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A Deformable C14 Laves Phase Produced at a 316L Stainless Steel/Niobium Inertia Friction Weld: *David F. Teter*[1]; Mark J. Cola[1]; Robert M. Dickerson[1]; Dan J. Thoma[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Techn., MS: G770, Los Alamos, NM 87545 USA

Inertia friction welding (IFRW) is well suited for joining dissimilar metals. The inherent solid-state nature and rapid thermal cycle afforded by IFRW can reduce base metal interdiffusion and the reaction between base metals. The objective of the current work was

to characterize the microstructural features of the 316L stainless steel-niobium interface. TEM of the interface revealed a continuous interaction layer, 200-nm wide at the SS-Nb interface with a structure based on the NbFe2 Cl4 Laves phase. STEM X-ray line scans revealed a uniform distribution of Fe, Cr and Ni in the Laves phase with these elements diffusing from the stainless steel to the Nb. Uniaxial tensile tests and hydrostatic burst tests show that the intermetallic layer deforms 6-10% and fails in the base metal. Geometric and electronic structure models suggest that this Laves phase will be deformable as observed in the experiments, and the details of these models will be discussed.

3:10 PM

Alloying of MoSi2 for Improved Mechanical Properties: *Adel A. Sharif*[1]; Amit Misra[2]; John J. Petrovic[2]; Terence E. Mitchell[2]; [1]University of Michigan-Flint, Eng. Sci., 213 Murchie Sci. Bldg., 303 East Kearsley St., Flint, MI 48502-1950 USA; [2]Los Alamos National Laboratory, Mats. Sci. and Techn., MST-8, MS K765, Los Alamos, NM 87545 USA

Effects of alloying on mechanical properties of solidification processed polycrystalline MoSi2, ternary (Mo, 1 at% Re)Si2, Mo(2 at% Al, Si)2, (Mo, 1 at% Nb)Si2, and quaternary (Mo, 1 at% Re)(2 at% Al, Si)2 alloys were evaluated by microhardness testing at room temperature and compression testing at elevated temperatures. Re is found to be a potent solid solution hardening addition to C11b MoSi2, whereas, Al and Nb resulted in room temperature solid solution softening. The quaternary (Mo, 1 at% Re)(2 at% Al, Si)2 alloy exhibited enhanced ambient temperature ductility and higher elevated temperature strength compared to pure MoSi2. The anomalous solid solution softening by alloying MoSi2 with 2 at% Al or 1 at% Nb and the rapid solid solution hardening observed in MoSi2 due to 1 at% Re are discussed.

3:30 PM Break

3:50 PM Invited

Investigation of Microstructural Parameters in the Intermetallics Ni_3Al and TiAl Through Transient Mechanical Tests: *Joël Bonneville*[1]; Jean Luc Martin[2]; [1]Université de Poitiers, Physique Dept., LMP-CNRS-UMR 6630, Bat.SP2MI, Futuroscope, Vienne 86960 France; [2]EPFL, Physique, IGA, LPM, Lausanne, Vaud 1015 Switzerland

The kinematics of plastic gliding in crystalline materials is usually described in terms of the Orowan's equation, which relates the plastic strain-rate with two important physical quantities: the velocity (v) and the density of mobile dislocations(ρ_m). Developing a fundamental understanding of the deformation mechanisms will therefore depend on our ability to characterise these two relevant quantities. Conventional mechanical tests usually measure or impose the strain-rate and do not allow for a separate determination of the dislocation mobility (v) and of the mobile dislocation density (ρ_m). Two experimental techniques have been designed to investigate the respective contributions of v and ρ_m to the plastic strain-rate. These tests, which consist of repeated load relaxation and repeated creep experiments, are undertaken during deformation tests performed at constant strain-rate. The measured parameters are the activation volume V, the change of the internal stress with plastic strain ($\delta\sigma_i/\delta\varepsilon_p$) and the variation of the mobile dislocation density ($\Delta\rho_m/\rho_m$), which results from a balance between dislocation multiplication and exhaustion. These techniques have been applied on two intermetallic compounds, i. e., Ni_3Al of the $L1_2$ structure and TiAl of the $L1_0$ structure. The values of V will be discussed in terms of dislocation mobility and those of mobile dislocation exhaustion parameters in terms of work-hardening. These results provide a key answer for the understanding of the origin of the flow stress anomaly for both intermetallic compounds.

4:20 PM Invited

Deformation Mechanisms in Lamellar TiAl Alloys: *Alain Couret*[1]; [1]CEMES-CNRS, 29 Rue J. Marvig, BP 4347, Toulouse Cedex 4 31 055 France

The deformation mechanisms in the lamellae and at the interfaces in TiAl alloys have been studied by in situ straining experiments performed inside the transmission electron microscope. In this paper, the glide mechanisms of ordinary dislocations and the role of the gamma/gamma interfaces on the plasticity will be studied and analysed. The ordinary dislocations are elongated along their screw orientation and anchored at many points. They move by jumps between these locking positions. A glide mechanism will be proposed from the quantitative analysis of the dynamics of this movement. Several examples of interactions between dislocations (ordinary and Shockley) and interfaces will be described, interpreted and compared. It will be shown that part of the incident shearing is often transmitted in the neighbouring lamella. The role of the internal stress due to dislocation pile-ups at the interfaces will be demonstrated.

4:50 PM

Evolution of Microstructure and Defect Structure in Massively Transformed L1o-Ordered Manganese Aluminide Alloys: *Jörg M.K. Wiezorek*[1]; Cagatay Yanar[1]; Velemir R. Radmilovic[2]; William A. Soffa[1]; [1]University of Pittsburgh, Dept. Mats. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA; [2]University of Belgrade, Dept. of Phys. Metall., Belgrade 11001 Serbia

The attractive technical magnetic properties of alloys based on the L1o-ordered intermetallic phase tau-MnAl are extremely sensitive to the microstructure and defect structure produced during the formation of the L1o-phase within the parent high-temperature epsilon-phase (hcp). In this study modern metallographic techniques including high-resolution electron microscopy (HREM) and in-situ TEM heating experiments have been applied to elucidate the nature of the phase transformation and evolution of the unique microstructure and defect structure governing the resultant structure-property relationships of these materials. The atomic processes occuring at the migrating interphase interfaces during transformation are shown to play a critical role in the generation of the so-called polytwinned microstructures and the profusion of planar defects characteristic of the MnAl-base alloys. Micromechanistic models are proposed that describe the interphase-interface related defect genesis.

International Symposium on Shape Casting of Aluminum: Science and Technology: Microporosity Formation: Advances in Modeling and Experimentation

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

Tuesday PM — Room: 224
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Jon Dantzig, The University of Illinois, Dept. of Mech. Eng., Urbana, IL 61801-2906 USA; Ravi Vijayaraghavan, Ford Motor Company, Ford Res. Lab., Dearborn, MI 48124 USA

2:00 PM Keynote

Control of Defects to Attain Enhanced Performance in Aluminum Cast Components: *Diran Apelian*[1]; [1]WPI, Metal Proc. Insti., 100 Institute Rd., Worcester, MA 01609 USA

Aluminum castings are widely used today for a variety of societal applications, and the demand for Al castings is expected to continue. Castings are being used to manufacture a variety of critical and high integrity parts such as control arms, cross members, and other automotive components. Performance requirements, particularly fatigue and dynamic properties, in high integrity cast components are attained through the control of microstructural defects

such as microporostiy, oxides, etc. In this presentation, the performance requirements will first be reviewed to establish a context. This will be followed by a review of defects found in Al castings, and the means by which we can control or alleviate them. Lastly, recent results on fatigue properties of cast aluminum alloys will be presented and reviewed.

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Modeling of Porosity Formation Using a Mushy Zone Refinement Method: *Michel Rappaz*[1]; Christel Pequet[1]; [1]EPFL, Dept. of Mat., Phys. Metall. Lab., MXG, Ecublens, Lausanne CH-1015 Switzerland

Porosity is a major defect in cast metallic alloys, resulting from a lack of feeding and segregation of gaz. Quantitative prediction of such defect requires to solve the combined Darcy and mass conservation equations in the evolving mushy zone, together with the segregation of gaz and nucleation/growth of pores. A major difficulty of such a task is the poor resolution in the mushy zone of a fixed enmeshment used to solve the heat-and-mass transport equations in the whole domain. In order to remove this limitation, a dynamic mushy zone refinement method has been developed: at each time step, the position of the mushy zone in a fixed FEM mesh is identified and then refined with small volume elements. Open, semi-open and closed liquid regions are automatically detected and appropriate boundary conditions are applied for each of them. A few examples, mainly related to shape aluminum castings, will be shown.

3:30 PM

A Model of Grain Growth and Pore Formation in Al-Si-Cu Alloys: *Ali Chirazi*[1]; Peter D. Lee[1]; Ravi Vijayaraghavan[2]; Jacob W. Zindel[2]; [1]Imperial College, Dept. of Mats., Prince Consort Rd., London SW7 2BP England; [2]Ford Motor Company, Mats. Sci., MD 3182, Rm. 2122, 2101 Village Rd., Dearborn, MI 48124 USA

319 is an important aluminum alloy extensively used in the automotive industry. One of the problems associated with aluminum casting is the formation of micro porosity. This is detrimental to the final cast mechanical properties. There are two main reasons for the formation of micro porosity: the metal shrinkage and formation of gas bubbles due to the change in hydrogen solubility in aluminum. A model, which takes into account the formation and growth of both grains and pores, is presented. A cellular automata (CA) method is used to simulate the nucleation and growth process and also the impingement of pores on the grain boundaries. 319 is approximated by the Al-Si-Cu ternary system. The nucleation processes of grains and pores are based on statistical distributions empirically fitted to experimental data. The grain growth is controlled by a ternary diffusion involving Al, Si and Cu. Pore growth is controlled by the H diffusion. The ternary diffusion calculation is based on a ternary phase diagram obtained using CALPHAD method. The phase calculated phase diagram provides us with the phase transformation temperatures, partition coefficients and the evolution of Si and Cu concentrations as solidification proceeds. The influence of the cooling rate and initial hydrogen concentration on the formation and growth of the micro-porosity have been considered and simulated results are compared to experimental measurements.

4:00 PM

Mathematical Modeling of Microporosity in A356 Aluminum Die-Castings: *Phuong Vo*[1]; Daan Maijer[1]; Steve Cockcroft[1]; Chris Hermesmann[2]; [1]University of British Columbia, Mets. and Mats. Eng., 309 6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada; [2]Canadian Autoparts Toyota, Inc., 7233 Progress Way, Delta, British Columbia V4G 1E7 Canada

A 2-D axisymmetric mathematical model to characterize the evolution of microporosity in aluminum is currently being developed. Fundamental principles of heat and mass transfer will be applied using a commercial FEM package to formulate a treatment consisting of a microscopic model, which incorporates nucleation and grain growth, coupled with a macroscopic heat transfer model. Theoretical mechanisms of microporosity formation will be included with hydrogen evolution and interdendritic shrinkage effects considered as criteria for pore formation. Preliminary work has focused on assessing a number of criteria functions developed by several authors against data from literature and experiment. Early qualitative results indicate that different criteria functions can predict microporosity with varying degrees of success. Model validation results in the form of temperature and criteria function predictions are compared with experimentally measured data from a directionally chilled A356 aluminum alloy casting with conditions (melt composition, mold material, etc.) set to resemble industrial practice.

4:30 PM

Numerical Determination of the Permeability of an Al-Si-Cu Alloy: *John Anthony Spittle*[1]; Stephen Graham Brown[1]; [1]University of Wales Swansea, Mats. Eng., Singleton Park, Swansea SA2 8PP UK

The macro-modelling of various solidification phenomena including interdendritic flow and microporosity formation is dependent on a knowledge of the permeabilities in two phase solid-liquid regions. Experimental determination is notoriously difficult,time consuming and subject to microstructural instability during the course of a test. A 3D numerical model has therefore been examined for determining liquid permeability, as a function of fraction solid and solid geometry, in a solidifying Al3Si3Cu alloy. The model involves the microstructural evolution of an equiaxed dendritic grain using a novel cellular automaton-finite difference procedure. The permeability of this grain as solidification proceeds is then evaluated using a finite difference solver for the interdendritic fluid flow. The permeability data are compared with experimentally reported values for equiaxed Al alloys and with values calculated by applying the Kozeny-Carman equation to the computer generated dendrite.

5:00 PM

Effect of Key Elements on the Feeding Characteristics of Aluminum-Silicon Casting Alloys: *Manas Ranjan Dash*[1]; Makhlouf Makhlouf[1]; [1]Metal Processing Institue, Worcester Polytechnic Institute, 100 Institute Rd., Worchester, MA USA

The most common and serious defect in aluminum castings is porosity, which is a result of two phenomena, insufficient feeding and/or hydrogen precipitation during solidification. In this paper, the feeding mechanisms of aluminum-silicon alloys are studied. A method to quantify the effect of alloying elements Si, Fe, Mg, Mn, Cu, Sr, and Ti as well as the cooling rate on the amount of porosity retained in aluminum castings has been developed. The casting is a plate with a feeder in one end and an iron chill at the other. The density of samples taken from locations along the length of the plate is measured using Archimedes principle following ASTM procedures. Hot isostatically pressed samples will be used to determine the theoretical density of each of the alloys. The percentage porosity is calculated from the difference between theoretical and measured density. The relative contribution of each of the alloying elements to porosity formation will be calculated using analysis of variance.

Lead-Free Solder Materials and Soldering Technologies IV: Interfacial Reactions, Intermetallics

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

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

Tuesday PM
February 13, 2001

Room: 227
Location: Ernest N. Morial Convention Center

Session Chairs: C. Robert Kao, National Central Univesity, Dept. Chem. Eng., Chungli City, Taiwan; King-Ning Tu, UCLA, Mats. Sci. & Eng., Los Angeles, CA USA

2:00 PM Invited

Solder Reaction in Flip Chip Technology: *King-Ning Tu*[1]; [1]UCLA, Mats. Sci. and Eng., 405 Hilgard Ave., Los Angeles, CA 90095-1595 USA

In microelectronic packaging, the use of solder is ubiquitous and the increasing use of solder in flip chip technology is a trend. In this talk, we shall discuss why we use solder in chip-to-organic packaging and why after hundreds years of solder use, we still have to study solder reaction. Using eutectic SnPb as an example, we shall compare the wetting reaction and solid state aging reaction between the solder and Cu. In the wetting reaction, the intermetallic compound formed has a scallop-type morphology which enables the Cu to reach the molten solder quickly, resulting in a high rate of free energy gain. In solid state aging, such a high rate of reaction is impossible, the morphology of the intermetallic compound becomes planar. The rate-limiting step in these interfacial reactions, whether it is diffusion-control or ripening-control, will be discussed. Example of Pb-free solders such as eutectic SnAg will be given as well.

2:25 PM

The Growth of Eta-Phase Intermetallic Whiskers in Liquid Tin-Solid Copper Reaction Couples: *Robert A. Gagliano*[1]; Morris E. Fine[1]; [1]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

The growth behavior of rod-like (whiskers) η-phase (Cu_6Sn_5) was investigated in liquid tin-solid copper reaction couples annealed at 275°C for times ranging from 30 seconds to 18 hours. The rod-like η-phase was examined two-dimensionally, using cross-sectional metallography, and three-dimensionally by stripping away the "tin bump" with a suitable etchant. Whiskers were observed to form atop the interfacial η-phase scallops, but not directly on the copper substrate. Detached whiskers were also noted in the bulk of the tin. For all annealing times, faceted, hollow whiskers were found dispersed within the tin matrix. At the shortest times, very few whiskers were observed connected to the η surface. With longer aging times, the density of whiskers emanating from the η-Sn interface increased and the morphology of the "attached" whiskers evolved from a round-ended, solid rod to an open-ended hexagonal faceted tube. Quantitative measurement of the attached and detached whisker length distribution vs. annealing time will be presented. Possible theoretical origins of the observed effects will be discussed.

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Kinetics of Intermetallic Formation at Sn-Ag-Cu/Cu Interface: *Jong-Hwan Park*[1]; *Jong-Hyun Lee*[1]; Yong-Ho Lee[1]; *Yong-Seog Kim*[1]; [1]Hong Ik University, Mats. Sci. and Eng., Mapo-Gu Sangsu-Dong 72-1, Seoul 121-791 Korea

Sn-3.5(wt.%)Ag-0.7Cu solder paste was printed on Cu substrates prepared by either electroplating or cold rolled process and reflowed to form solder bumps. Scallop size formed at the interface was measured as a function of reflow time and its growth direction was characterized using EBSP method. Most of the scallops grew in <0001> direction and the boundaries between the scallops were high angle in nature. Reflow solder bumping on a Cu6Sn5 pellet indicated that the solder penetrate into the high angle boundaries and form the channels between the scallops. Based on these observations, a model of intermetallic formation at the Sn-Ag-Cu/Cu interface was developed and was found to be in good agreement with experimental results.

3:05 PM Invited

Evolution of Intermetallic Compounds During Reaction of Pb-free and Pb-Sn Solders with Copper and Electroless Nickel: S. L. Liew[1]; A. S. Zuruzi[2]; K. S. Siow[3]; Y. Li[1]; *S. K. Lahiri*[2]; [1]National University of Singapore, Dept. of Mats. Sci., 10 Kent Ridge Crescent, Singapore 119260 Singapore; [2]Institute of Materials Research and Engineering, 3 Research Link 117602 Singapore; [3]Infineon Technologies Asia Pacific, 168 Kallang Way, Singapore 349253

With the current trend towards reducing or completely eliminating Pb in solders, various potentially important Pb-free compositions are currently being investigated. A common feature among most of these Pb-free solders is their high Sn content. Sn reacts with common metallisations such as Cu and Ni to form intermetallics. Changes in the solder composition can affect the reaction kinetics and the intermetallic roughness at the interface, which in turn may affect package reliability and wettability of the components during rework. Understanding of the kinetics and roughness evolution is thus important for identifying the appropriate solder composition and the underlying material system in order to obtain a reliable and robust solder joint. In this paper, we present the results of our investigation on the effect of Sn concentration on the formation of Cu-Sn and Ni-Sn intermetallic compounds using SnPb and Pb-free solders, and copper and nickel as the underlying materials.

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Interfacial Reaction Between a Pb-free Solder and Die Attach Metallizations: *Gautam Ghosh*[1]; Michael J. Pfeifer[2]; [1]Northwestern University, Dept. Mat. Sci. Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA; [2]Motorola, MD18, 4000 Commercial Dr., Northbrook, IL 60062 USA

The interfacial reaction between the Sn-3.5Ag-0.8Cu solder and the backside metallization schemes in field effect transistor (FET) and diode are studied. The FET metallization scheme is Si/Al(0.05μm)/Ti(0.2μm)/Ni(1μm)/Ti(0.003μm)/Ag(0.3μm) and the diode metallization scheme is Si/Ti(0.2μm)/Ni(0.3μm)/Ag(2μm) that are used in making devices for automotive applications. The interfacial microstructures after relow are studied by SEM. Due to rather high temperatures that these devices would likely to experience while in service, the interfacial reaction of the solder joints in the solid state might play a crucial role in determining the reliability of the devices. Therefore, the evolution of interfacial microstructure during solid state aging at temperatures up to 150°C are also studied. The role of the metallization schemes on the interfacial microstructures and the consequent effect on the reliability of the devices will be discussed.

3:50 PM Break

4:05 PM Invited

Reaction of Eutectic BiSn Solder with Au/Ni Surface Finish: G. L. Luo[1]; C. E. Ho[1]; A. H. Lin[1]; *C. Robert Kao*[1]; [1]National Central University, Dept. of Chem. Eng., Chungli City, Taiwan

The Au/Ni two-layer structure is a very common surface finish for the solder-ball pads in Ball-Grid-Array and other types of electronic packages, while eutectic BiSn eutectic solder is a promising lead-free solder, especially for consumer electronics applications. The objective of this study is to investigate the reaction between eutectic BiSn solder and Au/Ni surface finish during reflow and subsequent aging. Comparison with the reaction between eutectic PbSn solder with Au/Ni is to be made. Common to both types of reaction, an Au-Ni-Sn ternary compound formed from the reaction of the Au layer with Sn in the solder. However, distinctive and interesting differences also exist for these two types of reaction.

Rationale for these similarities and differences will be presented in this talk.

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Interfacial Reactions in Solder/Metallization Diffusion Couples: *Eric J. Cotts*[1]; Anis Zribi[1]; [1]SUNY Binghamton, Phys. Dept., P.O. Box 6016, Science 2, Binghamton, NY 13902 USA

As length scales decrease in electronics packages, kinetic considerations have come to dominate solder alloy formation processes. Phase selection at solder/metal interfaces is determined by which phase grows the fastest, i.e. by kinetics, rather than by energetics. Diffusion rates of metal atoms such as Cu, Au or Ni in Sn are orders of magnitude higher than rates of Sn in standard metallization constituents. In fact, the most Sn-rich phase (such as PdSn4, or Cu6Sn5) in a Sn-metal system is generally found to form first in conventional PbSn solder/metal diffusion couples. With the advent of Pb-free solders, solder/metal systems have become more complex. While Pb is removed from these solders, relatively small amounts (a few atomic percent) of metal atoms (for example Ag or Cu) are added to Sn to lower melting points of the solders and improve their mechanical properties. Furthermore, some metallizations are coated with a layer of Au which dissolves rapidly into solder upon melting. Thus a small atomic percentage of metal atoms (for example Cu, Ag, Ni or Au) are often found in a Sn matrix in Pb-free solders in solder joints. Because these species diffuse at high rates in Sn, investigation of interfacial growth processes in Pb-free solder/metallization couples must consider growth of alloys with relatively high concentrations of these elements. In fact, we observe a number of interface growth processes determined by the ternary addition of one of these constituents, e.g. the formation of (AuNi)Sn4 at Ni/solder interfaces upon thermal aging, or the formation of (CuNi)6Sn5 at Ni/solder interfaces. We present results of our observations of these growth processes. We attempt to characterize the kinetics of these processes with simple models.

4:50 PM

Effect of Ni Layer as a Diffusion Barrier on the Interface Reaction Between Solder and Cu Substrate: *Jae Yong Park*[1]; Chul Woong Yang[1]; Eun Jeong Kwon[1]; Choon Sik Kang[2]; Choongun Kim[3]; [1]Sungkyunkwan University, Metall. & Mats. Eng., Chunchun-dong, Jangan-gu, Suwon, Kyunggi-do 440-746 Korea; [2]Seoul National University, Mats. Sci. & Eng., Shillim-dong, Kwanak-gu, Seoul 151-742 Korea; [3]University of Texas, Arlington, Mats. Sci. & Eng., Arlington, TX USA

Soldering for electronic assembly is a process that forms intermetallic compounds in the interface by reaction between Sn in a solder and Cu. Interface reaction is occurred during the usage period of electronic products as well as the soldering process because melting point of a solder is relatively low. Therefore, after a long period of time, intermetallic compounds grow continously, and result in spalling phenomena. Especially for the flipchip package that contains limited amount of Cu coated layer, the excess consumption of Cu results in dewetting. Therefore, Thin Ni layer is coated on the Cu substrate to prevent this dewetting phenomena and to decrease the reaction rate in the interface. However, compounds of Sn-Cu-Ni by soldering with Ni diffusion barrier have not been characterized yet. In this paper, reactions between solder and Cu with Ni barrier are studied. Ni/Cu/Cr is evaporated on Si wafer, and soldered with various Sn-base solders. Interface reaction is studied using TEM analysis, and intermetallic compounds are characterized.

5:10 PM

Solid-Liquid Interdiffusion Bonding Between In-Coated Silver Thick Films: *Jing-Chie Lin*[1]; Long-Wei Huang[1]; Sheng-Long Lee[1]; [1]National Central University, Dept. of Mech. Eng., Chungli City, Taiwan

The solid-liquid interdiffusion bonding between two pieces of In-coated silver thick films has been investigated in this work. Silver thick films were screen-printed on an aluminum oxide substrate. The silver thick film was coated with a layer of indium in thickness ranging from 3 to 8 μm. Two pieces of In-coated thick films were held together in a compressive stress (0.03-0.04 M Pa) and heated (at 180-250 °C) for various durations (600-3600 s) to be bonded. The bond strength was estimated via a special designed tension tester. The bonding phases were using scanning electron microscopy (SEM), electron probe microanalysis (EPMA) and X-ray diffractometer (XRD). The tension tests demonstrated that the bond strength is greater for the specimens coated with thinner (3μm) indium than that coated with thicker (8μm) indium. The analysis of EPMA and XRD indicated that the bonding phase is quite different between the two cases. In the case coated with 3μm In, the bonding region consists of an Ag2In phase in central zone and an Ag phase in the periphery. In contrast, in the case coated with 8im, a phase of AgIn2 is in the central zone and an Ag2In in the periphery. Consequently, the bonding strength is correlated to the phases in the bonding region. A model for the solid-liquid interdiffusion of In-coated silver thick films is proposed.

5:30 PM

Intermetallic Growth of Wire-bond at 175°C High Temperature Aging: *Ker-Chang Hsieh*[1]; J. X. Pon[1]; C. C. Chen[2]; [1]National Sun Yat-sen University, Instit. of Mats. Sci. and Eng., Kaohsiung, Taiwan; [2]Philips Electronic Building Elements Industries, Ltd., Techn. Devel. Div., 10, Chin 5th Rd., N.E.P.Z., P.O. Box 35-48, Kaohsiung, Taiwan

This is the bondability evaluation work for an Al-pad diffusion process. Two types of samples are applied in this study, one is finished product for the cross-section examination and the other is wire-bond only used for the plane view examination after etching with KOH solution. These samples were aging at 175°C under air. The aging periods are from 0 hour to 1008 hours. According to the cross-section samples, the intermetallic phases formation sequences at 175°C are, Stage I: AlAu2 phase formed within 4 hours with ~1 um thickness. Stage II: AlAu2 phase gradually transformed to the Al2Au5 phase between 4 hours to 72 hours period. The thickness of Al2Au5 phase is ~2 um. Stage III. AlAu4 phase formed with Al2Au5 phase in the 72 to 240 hours period and the porosity found within the reacted phases. Al2Au5 phase amount decreased combined with AlAu4 phase amount increased as the aging hours increasing. The total phase thickness increased to ~ 4 um. Stage IV AlAu4 phase is the major phase after 336 aging hours combining with the porosities or cracks. The reacted phase layer thickness increase to ~ 5 um and reach the steady state. Also, there is titanium rich thin layer formed within the reacted phase layer. The layer thickness is too thin to identify the phase composition. Ternary phase AlAu2Ti exist in the samples with aging period 72, 144, 240, 336 and 672 hours. This phase has been reported at 500°C phase diagram.

Lightweight Alloys for Aerospace Applications: Processing and Properties - II

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

Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Nack J. Kim, Center for 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

Tuesday PM Room: 213
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Session Chair: William Frazier, Naval Air Systems Command, Pax River, MD USA

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Microstructure and Elastic Moduli of Several Particle Reinforced Metal Matrix Composites: *Mark C. Koopman*[1]; N. Chawla[2]; C. Coffin[1]; G. Green[3]; K. K. Chawla[1]; [1]University of Alabama at Birmingham, Dept. of Mats. and Mech. Eng., BEC, Room 254, 1530 3rd Ave. South, Birmingham, AL 35294-4461 USA; [2]Arizona State University, Dept. of Chem. and Mats. Eng.,

P.O. Box 876006, Tempe, AZ 85287-6006 USA; [3]Tougaloo College, Dept. of Chem., 500 West Countyline Rd., Tougaloo, MS 39174 USA

Several systems consisting of a metallic matrix with varying amounts and morphologies of carbide particle reinforced composites were characterized by microscopy and image analysis. Resonance Ultrasound Spectroscopy and impulse excitation techniques were used to determine elastic moduli. Correlations between fabrication methods, resultant microstructures and moduli of different composites will be presented.

2:30 PM

The Hardness of Al-Ge-Si Ternary Alloys: *David Mitlin*[1]; V. Radmilovic[2]; U. Dahmen[1]; John William Morris[1]; [1]University of California, Mats. Sci. and Eng. Dept., Berkeley, CA 94720 USA; [2]Lawrence Berkeley Laboratory, Nat. Cent. Elec. Micros., 1 Cyclotron Rd., Berkeley, CA 94720 USA

The focus of this work was the hardness of Al-1at.%Si-1at%Ge alloys. Despite the relative hardness of the Si-Ge precipitate, these alloys do not develop exceptional strength on aging. The reason is the sluggish precipitation of the diamond-cubic Si-Ge phase, which is, apparently, due to the high interfacial tension of the precipitates. The aging reactions, kinetics and resulting mechanical properties are characterized with high-resolution transmission electron microscopy and analyzed in terms of the distributed point-obstacle model of Glazer and Morris. We discuss how this problem can be overcome.

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The Effect of Aging Delay and Artificial Aging Time on the Quench Sensitivity of a Cast Al-7wt.%Si-0.6wt.% Mg (A357) Alloy: *Murat Tiryakioglu*[1]; James T. Staley[2]; John Campbell[3]; [1]Western Kentucky University, Dept. of Manuf. Sci., Adv. Manufact. Instit., Bowling Green, KY 42101-3576 USA; [2]Retired, Durham, NC USA; [3]University of Birmingham, Sch. of Metall. and Matls., Edgbaston, UK

The heat treatment of A357 alloy components usually involve an aging delay between the quench and the subsequent artificial aging. A minimum aging delay is required in some specifications ans standards for the heat treatment of this alloy. There is evidence in the literature showing both that the mechanical properties of cast Al-7%Si-Mg alloys may be adversely affected by the duration of the aging delay, and the highest properties are obtained when parts are artificially aged immediately after the quench. These studies, however, have been conducted using tensile specimens quenched in cold water at a high quench rate. Since aerospace castings can have quite complex geometries with varying section thicknesses, a wide range of average cooling rates are obtained during the quenching of these components. Hence, the combined effect of the aging delay and the artificial aging time was intestigated in our study to determine the aging delay and artificial aging time at 200°C that gives the least quench sensitivity. Specimens were quenched in six different media, and five different aging delays ranging from 0 to 24 hours were given the specimens. Components were then artificially aged at 200°C for 0.5, 1, 2 and 4 hours. The combinations of aging delay and artificial aging time that yield the least quench sensitivity are discussed in the paper.

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The Effect of Mg Content and Artificial Aging on the Work Hardening Characteristics of Cast Al-7%Si-Mg Alloys: *Murat Tiryakioglu*[1]; John Campbell[2]; James T. Staley[3]; [1]Western Kentucky University, Dept. of Manuf. Sci., Bowling Green, KY 42101-3576 USA; [2]University of Birmingham, Sch. of Metall. and Matls., Edgbaston B15 2TT UK; [3]Retired, Durham, NC USA

Cast Al-7%Si-Mg alloys are widely used in aerospace applications due to their excellent castability. Despite their wide use, the effect of different factors on tensile properties is not completely understood. This is probably due to the fact that there exists a defect hierarchy in these alloys. Recent research has shown that work hardening characteristics of these alloys can be used to assess the effect of microstructural defects on the tensile properties in these alloys. Hence the work hardening charateristics of cast Al-7%Si-Mg alloys were investigated. Work hardening behavior was characterized in terms of tangent modulus-true stress relationship. Kocks-Mecking model was found to express the work hardening behavior of these alloys. The effects of three levels of Mg (0.20, 0.40 and 0.60 wt.%) and different artificial aging treatments on Kocks-Mecking parameters were investigated. The relationships between Kocks-Mecking parameters and microstructure are discussed.

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Investment Casting of Titanium Alloys with CaO Crucible and CaZrO3 Mold: *Shaekwang Kim*[1]; [1]Sung Kyun Kwan University, Sch. of Metall. and Matl. Eng., 300 Chunchun-dong, Jangan-gu, Suwon, Gyunggi-do 440-746 Korea

Although titanium alloys are excellent candidates for aerospace applications, titanium usage is stronly limited by its higher cost relative to competing materials. There are two approaches to achieve this, development of lower cost alloys and/or processing improvement to reduce the fabrication casts, with the after probably offering the greatest potential. A significant cost reduction can be realized, if ceramic materials capable of containing the molten titanium without undue contamination can be developed, thereby allowing induction melting as well as conventional molding practices to be used for titanium investment castings. The work has been conducted for the purpose of developing a cost-savings process for producing investment castings of titanium alloys. Effects of mold materials and binders on metal/mold reactions of titanium investment castings have been evaluated, and an investment casting process for noncritical applications of titanium investment castings have been developed, using CaO crucible and CaZrO3 mold.

4:10 PM

Modeling of the Warpage Behavior of 7075 Aluminum Alloy Extrusions: *C. Lei*[1]; A. Yue[2]; D. Manriquez[2]; J. Foyos[2]; J. Quilla[2]; S. Hannan[2]; S. Harris[2]; S. Vasquez[2]; T. Ruperto[2]; E. W. Lee[1]; Omar Es-Said[2]; [1]Naval Air Systems Command, Code 4.3.4.2, Aircraft Div., Patuxent River, MD 20670 USA; [2]Loyola Marymount University, Nat. Sci. Found. Res. Exp. for Undergrad. Prog., Los Angeles, CA 90045 USA

Extruded I sections were machined into four different section shapes, L, short depth L, T and short depth T. The furnace was preheated to 780°F and the samples were placed inside. The temperature was raised to 880°F and then quenched in either a 30% polyalkylene glycol solution or water, both at 59°F. Points on the distorted samples were recorded before and after the solution treatment; the difference between the measurements indicated the extent of warpage. A finite element software, COSMOS, was used to simulate the data to predict the thermal gradients in the quenched samples.

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Effect of Grain Size and Stability on Ambient Temperature Creep of Beta Titanium Alloys: *Durgalakshim Doraiswamy*[1]; Sreeramamoorthy Ankem[1]; [1]University of Maryland, College Park, Mats. Sci. Eng., Bldg. 090 Stadium Dr., College Park, MD 20742

It has been recently shown that Beta Titanium alloys can creep at ambient temperatures at 95% Yield Stress. In this investigation the effect of grain size on the creep deformation behavior of Beta Ti-9.4%Mn alloy has been studied. Further, the effect of stability of beta phase at a particular grain size and stress level has been studied. These results show that, in general, the extent of creep deformation decreases with decrease in grain size and also decreases with increase in stability of beta phase. The details of the investigation will be presented. This work is being supported by Office of Naval Research under Grant No. N0001496101819.

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High Temperature Load Relaxation Behavior of TiAl Alloy: *Yong Nam Kwon*[1]; Sun Keun Hwang[2]; Young Won Chang[1]; [1]Center for Advanced Aerospace Materials, POSTECH, Pohang 790-784 Korea; [2]Inha University, Dept. of Metall. Eng., Inchon 402-751 Korea

The present study aims at the interpretation of deformation behavior of fully lamellar TiAl alloy at the elevated temperatures within the framework of the recently proposed internal deformation variable theory. First of all, flow curves were obtained from the load

relaxation tests performed at the temperature range of 600-900ºC. Then the flow curves were analyzed based on the above-mentioned internal variable theory. It has been found that the overall stress of each flow curve consists of internal stress and frictional stress. With the increment of temperature the contribution of internal stress seems to be larger. In addition, it has been observed that linear array of dislocations were formed at the lamellar interface during the deformation at 800°C. In this respect, the high temperature deformation of fully lamellar TiAl alloy is likely to be dominated by the dislocation glide within the γ lath. The parameters defined in the constitutive equations were determined using a nonlinear curve fitting. In this way, much significant information on the deformation behavior of TiAl alloy could be obtained.

Magnesium Technology 2001: Alloy Development

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

Tuesday PM — Room: 203-205
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Eric A. Nyberg, Pacific Northwest National Laboratotry, Mats. Proc. Grp., Richland, WA 99352 USA

2:00 PM

Magnesium Alloy Development Guided by Thermodynamic Calculations: Joachim Groebner[1]; Dmytro Kevorkov[1]; *Rainer Schmid-Fetzer*[1]; [1]Technical University of Clausthal, Inst. of Metall., Robert-Koch-Str. 42, Clausthal-Zellerfeld D-38678 Germany

In traditional alloy development experimental investigations with many different alloy compositions are performed. The selection criteria for multicomponent alloying elements and their compositions become diffuse in a traditional approach. Computational thermochemistry can provide a clear guideline for such selections and helps to avoid long-term experiments with less promising alloys. It enables the calculation of multicomponent phase diagrams and the tracking of individual alloys during heat treatment or solidification by calculation of phase distributions and phase compositions. These are the basic data to understand and control the behavior of any novel or modified Mg-alloy. Thus it is a powerful tool to cut down on cost and time during development of Mg-alloys. We report on recent applications and our progress in construction of the necessary thermodynamic database for several promising alloying elements like Al, Li, Si, Mn, Ca, Sc, Y, Zr and Rare Earths, using the Calphad method combined with own key experiments.

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Computational Thermodynamics and Experimental Investigation of Mg-Al-Ca Alloys: *Koray Ozturk*[1]; Alan Luo[2]; Zi-Kui Liu[1]; [1]The Pennsylvania State University, Dept. of Mats. Sci. and Eng., Steidle Bldg., University Park, PA 16802 USA; [2]General Motors Res. and Dev. Cen., Mats. and Proc. Lab., 30500 Mound Rd., Warren, MI 48090-9055 USA

The thermodynamic properties of the ternary Mg-Al-Ca system are investigated, based on the three binary systems, i.e. the Al-Mg system, the Ca-Mg system and the Al-Ca system. The ternary system contains five different intermetallic compounds that are treated as stoichiometric compounds. The calculated ternary system and Scheil simulations are used to direct new experiments in the project. Experimental investigations include diffusion multiples and individual alloys for establishing the ternary phase equilibria and fine tuning the phase relationships. Metallographic samples are prepared. The phase compositions and the crystal structures of the phases are determined using EPMA, SEM and TEM.

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Creep-Resistant Mg-Al-Sr Alloys: *Mihriban O. Pekguleryuz*[1]; Eric Baril[1]; [1]Noranda Inc Technology Centre, Mats. Eng., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada

This paper presents the development a new family of creep-resistant magnesium alloys based on the Mg-Al-Sr system. Creep resistance, and the tensile yield strength of these alloys at 150°C and 175°C show significant improvement over Mg-Al-RE and Mg-Al-Si system. The microstructure of the alloys is characterized by Al-Sr containing intermetallic second phases. STEM analysis indicates the presence of Al-Sr-Mg containing phases in addition to Al-Sr phases. The creep mechanism of these alloys is under investigation via transmission electron microscopy.

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Die Casting Magnesium Alloys for Elevated Temperature Applications: *Boris Bronfin*[1]; Eli Aghion[1]; Frank von Buch[2]; Soehnke Schumann[3]; Mark Katsir[1]; [1]Dead Sea Magnesium, Res. Div., P.O. Box 1195, Beer-Sheva 84111 Israel; [2]Volkswagen AG, Veh. Res., Volkswagen AG Letter Box 1777, Wolfsburg D-38436 Germany; [3]Volkswagen AG, Veh. Res., Volkswagen AG Letter Box 1777, Wolfsburg D-38436 Germany

The growing use of magnesium alloys for producing automotive drive train components requires development of new alloys with improved creep properties. This paper discloses the results of a comprehensive study aimed at the development of creep resistant cost-effective die casting alloys with capability of long term operations at temperatures up to 150°C under high loads. The newly developed alloys designated as MRI 15X series exhibit die-castability corrosion resistance, room temperature strength and short-term elevated temperature strength similar or better than those of AZ91D alloy. However, the most important fact is that the new alloys have creep resistance at temperatures of 130°C-150°C under stress of 50-85MPa significantly better than that of the commercial alloys AZ91D, AE42 and AS21. The principles of alloying and the metallurgical aspects of new alloys are presented and discussed.

3:40 PM Break

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Diecasting and Diecast Properties of Alloys Based on the Mg-Al-Sr System: Mihriban Ozden Pekguleryuz[1]; *Donald Argo*[1]; Pierre Labelle[1]; [1]Noranda Inc Technology Centre, Mats. Eng./Light Met., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 4R8 Canada

The need to develop magnesium diecasting alloys for transmission and engine components is leading to active R&D in magnesium alloy development. Mg-Al-Sr based alloys are a new addition to the creep-resistant magnesium alloys developed in the recent years. This paper presents the investigation of the diecastability of Mg-Al-Sr and Mg-Al-Sr-X alloys. Mechanical properties (tensile, creep, impact fatigue) of both separately-cast test specimens and samples removed from diecast automotive parts (cam-cover) are high and creep resistance shows improvement over existing magnesium diecasting alloys AS41 and AE42. Corrosion resistance of the alloys compares well with high purity magnesium alloys AZ91D and AM60B. Diecastability of the alloys was determined via diecasting trials involving 300 automotive cam-covers per alloy. Both the Mg-Al-Sr and Mg-Al-Sr-X alloys were determined to be diecastable. Mg-Al-Sr-Ca alloys were observed to have the added

advantage of low tendency to burning and oxidation. Further investigation of the diecastability is underway.

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Tensile and Compressive Creep of Magnesium-Aluminum-Calcium Based Alloys: *Alan A. Luo*[1]; Bob R. Powell[1]; [1]General Motors R&D Center, Mats. & Proc. Lab., MC: 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

In this paper, both tensile and compressive creep properties of the newly developed Mg-Al-Ca-based ACX alloys are reported to be significantly better than those of AE42 alloy. The tensile creep behavior of ACX alloys in this study obeys a power-law type of constitutive equation. The stress and temperature dependencies of the secondary creep rate of ACX alloys were also studied. The results suggest that the improved creep resistance in ACX alloys can be primarily attributed to the thermal stability and the interfacial coherency of the (Mg,Al)2Ca phase in the microstructure of the alloys.

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Creep and Bolt-Load Retention Behavior of a Die Cast Magnesium-Rare Earth Alloy: *Ian Patrick Moreno*[1]; J. Wayne Jones[2]; John E. Allison[3]; [1]University of Michigan, Mats. Sci. and Eng., 2300 Hayward St., 3062 H. H. Dow Bldg., Ann Arbor, MI 48109-2136 USA; [2]University of Michigan, Matls. Sci. Dept.; [3]Ford Motor Company, Ford Res. Lab., Mats. Sci. Dept.

Creep and bolt load retention behavior of the die-cast magnesium alloy, EZ31, is discussed. Primary alloying additions of this relatively new alloy are rare earth elements and zinc. Bolt load retention tests were conducted at preloads of 14 to 28kN and at temperatures ranging from 125 to 175°C. The microstructure of EZ31 consists of alpha-Mg grains, some containing a Mg-Mn phase within the matrix, and a Mg-RE grain boundary phase. When compared to other Mg systems, such as AZ91D, AE42, AM50, and AM50+Ca alloys, EZ31 retained the greatest fraction of the initial preload at all test temperatures and preloads. Bolt load retention was not significantly influenced by temperature. Tensile and compressive creep tests were conducted at 125-175°C and stresses of 50-150MPa. The creep behavior of EZ31 was similar to that of AE42. The stress and temperature dependence of steady state creep have been quantified and will be related to microstructure.

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The Mg-Zn-Al Alloys and the Influence of Calcium on Their Creep Properties: *Zhan Zhang*[1]; Réal Tremblay[1]; Dominique Dubé[1]; [1]Laval University, Dept. of Min., Metall. and Matls. Eng., Cite Universitaire, Ste-Foy, Quebec G1K 7P4 Canada

Mg-Zn-Al and Mg-Zn-Al-Ca are promising alloy systems for the development of new cast alloys with greater creep properties than AZ91 alloy, tensile proterties near AZ91, acceptable castability, and density less than 2.00 g/cm3. In this work, the properties and microstructure of many Mg-Zn-Al and Mg-Zn-Al-Ca alloys were studied in order to determine the key factors responsible for the improved creep behavior and optimize the compositions. The solidification microstructure of Mg-8-14%Zn-2-6%Al alloys was characterized, and relationship between the constituents and creep properties were established. Three intermetallic phases, τ (Mg32(Al,Zn)-49), ϕ (Al2Mg5Zn2), and ε (MgZn), were identified. The results showed that the phase τ plays an important role in the improvement of creep resistance. The influence of calcium on the microstructural changes of Mg-Zn-Al alloys and their corresponding creep properties were also presented. It was found that calcium can modify the intermetallic phase τ and improve the thermal stability of these alloys.

Materials Processing Fundamentals IV

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

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Session Chair: Yogeshwar Sahai, Ohio State University, MSE Dept., Columbus, OH 43210-1179 USA

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Self-Similar Growth of a Compound Layer in Thin-Film Binary Diffusion Couples: Huifang Zhang[1]; *Harris Wong*[1]; [1]Louisiana State University, Mech. Eng. Dept., Baton Rouge, LA 70803 USA

Diffusion controlled growth of a compound phase AnB between two parallel thin films of material A and B is important in electronic materials processing and in synthesis of high-temperature materials using multilayer films. Previous models of the growth rate do not solve the diffusion equation, and thus do not utilize fully the predictive capability. This talk presents a self-similar solution of the diffusion equation with the nonlinear Kirkendall effect included. It is found that the intrinsic diffusion coefficients of A and B in AnB are simultaneously determined from the positions of the interfaces without using the concentration profile in the compound phase. This provides a simpler method for measuring intrinsic diffusion coefficients. Implications of this complete solution will be discussed.

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Determination of Drop Size Distribution in a Continuous Mixer: *Maria Cristina Ruiz*[1]; Ximena Muller[1]; Rafael Padilla[1]; [1]University of Concepcion, Dept. of Metall. Eng., Edmundo Larenas 270, Concepcion, Chile

In the present study, the size distribution, Fo(v), of organic drops produced in a continuous-mixing vessel has been determined experimentally for various conditions. The organic phase used was a 1:1 mixture of a salicylaldoxime (LIX 860N-IC) and a ketoxime (LIX 64-IC) in an aliphatic diluent (Escaid 103). The aqueous phase was a copper sulfate solution. The results indicated that an increase in stirring speed, extractant concentration and pH produced smaller drops. The effect of the stirring speed was the most significant increasing the average volume of drops by 57% for a change from 300 to 400 rpm. On the contrary, an increase in the dispersed phase fraction, copper sulfate concentration, and retention time produced larger drops. Changes in temperature from 23 to 30°C did not affect significantly the drop sizes. In all cases, the experimental drop size distributions could be accurately represented by a lognormal distribution.

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Effect of Cooling Rate on the Formation of Austenite Grains in Fe-C-Mn Alloys: *Ho-Jung Shin*[1]; Sin-Myoung Kang[1]; Seon-Hyo Kim[1]; [1]Pohang University of Science and Technology, Dept. of Mats. Sci. and Eng., San 31, Hyoja-dong, Nam-gu, Pohang, Kyungbuk 790-784 Korea

The alloy of Fe-C-Mn is cooled from 1873 to 1473K at different cooling rates, in order to test a new model developed for estimating the size and morphology of austenite grain. The model was proposed by systemizing the relation between the driving force(undercooling) and energy barrier(misfit strain energy on ¥ä/¥ãinterface) for ¥ä/¥ã transformation. The model implies that the formation of austenite grains on ¥ä/¥ãinterface is much governed by the specific orientation index system relation depending on the cooling rates. The modeling results are well coincident with the experimental ones.

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Water Modeling for Flow Characteristics of Molten Steel in RH Refining Process: *Ji-He Wei*[1]; Neng-Wen Yu[1]; Yang-Yi Fan[1]; Sen-Long Yang[1]; [1]Shanghai University, Dept. of Metal. Mats., 149 Yan Chang Rd., Shanghai 200072 PR China

The flow characteristics of molten steel during the RH vacuum circulation refining were investigated on a water model with 1/5 scale of a 90t multifunction RH unit. The circulation rate was more accurately measured using a direct method. The fluid flow pattern and field in the ladle as well as the effects of technological and geometric factors, including gas top blowing operation, were examined. The results indicated that the circulation rate of molten steel in the unit can be fairly precisely calculated by the expression: Ql= 0.0333Qg0.26Du0.69Dd0.80 (t/min), where Qg gas flow rate (Ndm3/min), Du and Dd up- and down-snorkel (cm). The circulation rate will increase to a saturated (limited) value with the increase in gas flow rate, which is 30.9 t/min for the case of Du and Dd=30cm. There are a main loop and a number of small eddies and vortexes, and a liquid-liquid two phase flow between the down-snorkel liquid stream and the liquid around it, all of which will strongly influence the mixing and mass transfer phenomena in the ladle during the refining.

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A Continuum Scalar Model of Facets: Tinghui Xin[1]; *Harris Wong*[1]; [1]Louisiana State University, Mech. Eng. Dept., Baton Rouge, LA 70803 USA

Facet formation and evolution are important in many processes, such as crystal growth, solidification, and grain growth during annealing. Current models of facets usually choose a particular form of anisotropic surface energy and then compute the interfacial profile. This approach works fine if the surface energy anisotropy is weak and the interfacial profile is smooth. However, when the anisotropy increases, the surface profile starts to intercept itself. This non-uniqueness makes it difficult to model strong anisotropy in interfacial evolution. We have developed a new scalar model of facets that eliminates the non-uniqueness of interfacial profile. Planar facets and sharp corners can be easily handled by the new model. In addition, it can be incorporated directly into different theories of interfacial evolution. The new model also allows a simple proof of Wulff's theorem. In this talk, we will present the new model and discuss its applications.

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Study on Mathematical Modeling of Decarburization during AOD Refining Process of Stainless Steel: *Ji-He WEI*[1]; De-Ping Zhu[1]; [1]Shanghai University, Dept. of Metal. Mats., 149 Yan Chang Rd., Shanghai 200072 China

The competitive oxidation of the elements in molten steel such as C, Cr, Mn, Si and the changes in decarburization rate and bath temperature with carbon content during the AOD refining process of stainless steel were examined. The mass and heat balances of the AOD process and system were conducted and the heat losses were more accurately estimated. A new mathematical model for the AOD refining process of stainless steel was developed and applied to the industrial process in an 18t AOD vessel. The parameters, including the distribution ratios of oxygen gas among C, Cr, Si, Mn in molten steel and the activity coefficients of the components in slag melt, were more reasonably determined. The results indicated that the changes in carbon content and bath temperature predicted by this model are in fairly good agreement with the plant data. The effects of technological and operative factors on the refining process were analyzed using the model.

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Curing Kinetics of a Modified Bismaleimide: *Ying Xiong*[1]; [1]Nanyang Technological University, School of Mech. and Prod. Eng., Mats. Lab., Nanyang Ave., Singapore 639798 Singapore

Bismaleimide (BMI) has been widely used in aromatic, semiconductor industry, due to its high thermal stability than epoxy and better processibility than the condensation-type polyimides. It bridges the temperature performance gap between epoxies and polyimides. But its disadvantages are poor solubility in ordinary solvent (i.e., acetone), high processing temperature, and brittleness of the cured resin. As a result, many modification methods have been applied to improve the toughness and solubility. In order to improve the procesibility and toughness, several modification methods are always used simultaneously in the same formulation. It makes the curing process very complicated. Kinetic modeling curing process is critical to cure the resin properly. But few works concentrate on this complicated BMI curing system. In this paper, kinetics of a commercial modified BMI has been monitored by differential scanning calorimetry (DSC). Isothermal method is used to establish the kinetic model for the curing process. Analyses results demonstrate that, due to multiple curing reactions overlapping in the curing process, activation energy changes with the conversion. Kinetic model also changes from nth-order model to autocatalytic model.

Materials & Processes for Submicron Technology: Characterization Techniques

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

Tuesday PM — Room: 226
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Bhushan Sopori, NREL, 1617 Cole Blvd., Golden, CO 80401 USA; Manabu Ishimaru, Osaka University, 8-1 Mihogaoka, Ibaraki, Osaka 567-0047 Japan

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2:00 PM Invited

Non-Contact Characterization of Dielectric and Interface Properties Using COCOS and SILC-Probe Metrologies: Andrew M. Hoff[1], [1]University of South Florida, Elect. Eng. Dept., Tampa, FL 33620 USA

COCOS, corona oxide characterization of semiconductor, and SILC-Probe, stress induced leakage current, metrology methods are relatively new non-contact methods for on-line characterization of dielectric and interfacial properties of thin films used in silicon IC's. Both methods are based upon the application of electric fields to dielectric/semiconductor structures with the aid of corona charge produced with a high-voltage discharge in air. The use of corona charge allows films to be characterized immediately following their growth, avoiding the fabrication of electrical test device structures. Important parameters such as flatband voltage, Tox, Cox, Dit, and Dit spectra are obtained directly from COCOS measurements and do not require assumptions as in capacitor based determination of these quantities. The SILC method provides important information regarding the susceptibility of a dielectric/substrate structure to defect formation with increasing charge injection. The background physics of each measurement technique will be discussed along with representative examples of their utility in silicon thin film research and development efforts.

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Electrical Properties of Ru and RuO2 Gate Electrodes for Dual Metal Gate Si-CMOS with Hi-K ZrO2 Dielectrics: *Huicai Zhong*[1]; Veena Misra[1]; Greg Heuss[1]; [1]North Carolina State University, Dept of Elect. Eng., Box 7911, Raleigh, NC 27695 USA

As silicon CMOS devices are scaled below 100nm, the evaluation of metal gate becomes important to decrease gate depletion effect and improve interface between gate electrode and dielectrics.

Ru (Ruthenium) and RuO2 are attractive gate electrodes because of their large workfunctions (~5eV), low resistivity and excellent thermal/chemical stability. In this work, we have studied the electrical and thermal stability of Ru and RuO2 electrode on SiO2, ZrO2 and ZrSiO4 gate dielectrics. Thermal and chemical stability of the electrodes was studied at annealing temperatures up to 800°C in N2 and subsequently forming gas annealing. XRD and XPS were measured to study grain structure and interface reactions. Electrical properties were evaluated on MOS capacitors. The role of oxygen inside dielectrics was studied by comparing EOT change as a function of annealing temperature for capacitors with as-deposited and 800°C pre-annealed ZrO2 and ZrSiO4. For capacitors with Ru gate on 8000C pre-annealed ZrO2, excellent stability of equivalent oxide thickness was detected. Flatband voltage and gate current as a function of annealing temperature were also studied, which indicate that Ru and RuO2 are promising gate electrodes for P-MOSFETs.

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Microstructure and Mechanical Properties of LIGA Nickel MEMS Structures: Winston O. Soboyejo[1]; Seyed Allameh[1]; J. Lou[1]; T. Bucheit[2]; [1]Princeton University, Dept. of Mech. & Aerosp. Eng., 1 Olden St., D404 Engr. Quad., Princeton, NJ 08544 USA; [2]Sandia National Laboratories, Albuquerque, NM 87185 USA

The microstructure and mechanical properties of LIGA nickel MEMS structures are discussed in this paper. The hierarchies of microstructural features in LIGA nickel structures are elucidated via atomic force microscopy, transmission electron microscopy, orientation imaging and scanning electron microscopy techniques. The results of micro-tensile and micro-buckling experiments on well characterized LIGA nickel MEMS structures are then presented. The effects of specimen length scale are then examined within the context of anisotropic elasticity models and strain gradient plasticity concepts. The underlying dislocation substructures associated with plasticity length-scale effects are elucidated via transmission electron microscopy.

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3:30 PM

Properties of Sputter-deposited Tantalum Silicides Films on Silicon: *Lei Jin*[1]; Dentcho Ivanov[1]; James Grow[2]; Oktay Gokce[1]; N. M. Ravindra[1]; [1]New Jersey Institute of Technology, Dept. of Phys., 161 Warren St., Newark, NJ 07102 USA; [2]New Jersey Institute of Technology, Chem. and Chemic. Eng., 161 Warren St., Newark, NJ 07102 USA

Tantalum silicide films were sputter deposited on p- and n- type silicon substrates. The thicknesses of the films considered in this study were 200, 600 and 1000 angstroms. The TaSi2/Si structures were annealed at temperatures in the range of 400 to 900ºC. The measured sheet resistance was found to decrease with increase in annealing temperature and decrease with increase in film thickness. X-ray diffraction results show changes in the morphological structure of the films from amorphous to crystalline after annealing. Oxidation experiments performed in the temperature range of 500-900ºC, in steam ambients for durations in the range of 0.5 to 1.5 hours, show no oxide formation of the films.

Properties of Nanocrystalline Materials: Physical Properties and Characterization

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

Program Organizers: Sung H. Whang, Polytechnic University, Dept. of Mech. Eng., Brooklyn, NY 11201 USA; Horst W. Hahn, Techische Hchschule Damstadt, Darmstadt D-64287 Germany; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552 USA

Tuesday PM — Room: 223
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: S. H. Whang, Polytechnic University, NY USA; Reza Mirshams, University of North Texas, Denton, TX 76203 USA

2:00 PM Invited

Preparation, Characterization and Properties of Pd Supported on Nanocrystalline Oxide Catalysts for Methane Oxidation: *Gar B. Hoflund*[1]; Johannes Seydel[2]; Horst W. Hahn[2]; [1]University of Florida, Dept. of Chem. Eng., Gainesville, FL 32611 USA; [2]Darmstadt University of Technology, FB 21-Matls. Sci. Dept., Thin Films Div., Petersenstrasse 23, Darmstadt 64287 Germany

Nanocrystalline oxides including CeO_2, ZrO_2 and TiO_2 have been shown to be excellent catalysts for methane oxidation under lean conditions. The bare oxides exhibit considerable activity and the addition of Pd greatly enhances their activities. On a mass basis the Pd/nano catalysts are significantly more active than the corresponding Pd/poly catalysts, and, in fact, they are the most active methane oxidation catalysts that have ever been made. In comparing nano and poly catalysts, there are two considerations: surface area and activity per specific area. Although the nano catalysts have higher surface areas, their activity per surface area has been shown to be lower. This result is consistent with surface characterization studies which demonstrate that the surface chemistry is different for nano and poly catalysts. This negative result implies that there is a significant margin for improving nano catalysts if the surface chemistry can be made more similar to the poly catalysts. In this study the same nano-oxides have been made using several different preparation techniques, and the resulting Pd/nano catalysts are being tested for catalytic activity and characterized using multiple techniques. This study will result in improved methane oxidation catalysts and a better understanding of the factors which are important with respect to the surface chemistry.

2:25 PM Invited

Optical Properties of Caged and Free Nanoparticles: Roland Schmechel[1]; *Heinz von Seggern*[1]; [1]Darmstadt University of Technology, Electr. Mats., Petersenstr.23, Darmstadt 64287 Germany

Optical properties such as photoluminescence emission spectra, excitation spectra, lifetimes and quantum efficiencies of nanocrystalline GaN and europium doped Y_2O_3 were investigated in different caging hosts such as porous MCM-41, silica and alumina with a pore size ranging between 2.7nm and 80nm. The structural properties of the particles and hosts were determined by x-ray diffraction and transmission electron microscopy. In case of GaN, only results on filled MCM-41 with a mean pore size of 2.7nm will be reported. A bandgap shift of about $\delta E_g = 0.255$ eV compared to bulk GaN is measured and correlates nicely with an estimate of the theoretically expected shift by quantum confinement. In case of $Y_2O_3:Eu^{3+}$, a comparison of free standing nanopowders and nanoparticles in different hosts (MCM-41, porous silica,porous alumina, aqueous solution) with commercially available bulk lamp phosphors of about 5µm grain size will be reported. A strong dependence of the charge transfer process on the surrounding of the nanoparticle is detected.

2:50 PM Invited

Quantitative Lorentz Microscopy of Magnetic Nanocrystalline Materials: *Marc De Graef*[1]; [1]Carnegie Mellon University, Mats. Sci. and Eng., Roberts Engineering Hall, Rm. 130, 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

Recent advances in Lorentz microscopy now make it possible to obtain quantitative information about the magnetic microstructure of solids and nanocrystalline materials at high spatial resolution. The fundamental physical parameter which underlies image formation in the transmission electron microscope is the phase of the electron wave, which for a magnetic object, contains information about the magnetization state of the material. Using a non-interferometric phase reconstruction technique one can reconstruct the phase of the electron wave, starting from conventional Fresnel or out-of-focus images. The gradient of the phase then results in a map of the product of the in-plane component of the magnetic induction and the local sample thickness. In this contribution we will first describe the details of the phase reconstruction method, and then apply the method to a variety of material systems.

3:15 PM Invited

Three Dimensional Atom Probe Studies of Nanocrystalline Metallic Materials: *Kazuhiro Hono*[1]; [1]National Research Institute for Metals, Mats. Phys. Div., 1-2-1 Sengen, Tsukuba 305-0047 Japan

Nanocrystallization processes of amorphous alloys are widely used for processing nanocrystalline soft magnetic materials, nanocomposite hard magnets and nanocrystalline ultahigh stregth materials. This talk overviews nanocrystallization proesses of various amorphous alloys studied mainly by the three dimensional atom probe (3DAP), complemented by high resolution electron microscopy (HREM) and small angle x-ray scattering (SAXS). The final nanocrystalline and nanocomposite microstructures obtained via a crystallization route from amorphous precursors are very sensitive to alloy compositions, microalloyed elements and heat treatement conditions. Using 3DAP, the distributions of solute atoms in the course of crystallization processes of Fe, Al, and Zr based amorphous alloys are visualized with an atomic resolution, and the overall microstructures are examined by TEM. The factors influencing nanocrystalline microstructures and the structure-property relationship of nanocrystalline! Alloys will be discussed based on these results.

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Melting Points of Nanostructured Materials: *K. Lu*[1]; [1]Institute of Metal Research Chinese Academy of Sciences, State Key Lab. for RSA, Shenyang 110015 China

As melting is normally initiated at solid surface or interfaces, the melting point for nanostructured materials is significantly deviated from the equilibrium melting point for bulk materials. In this talk, experimental and computer simulation studies of the melting points for various kinds of nanostructured metals (nano-granular particles and thin films) will be presented. It shows that the melting point of nano-granular particles can be either depressed or elevated depending upon the interface structure between the particle and the matrix. An evident particle size dependence of the melting point was identified. More interestingly, we found, for the first time, the melting point of Pb thin films confined by Al layers can be substantially elevated. The superheating phenomenon in thin films provides a unique opportunity for study the nature of melting of solids. And meanwhile, it is crucial for the technological applications of nanostructured materials with novel properties and performance.

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Photoelectrode Properties of Nanocomposite Thin Films Based on Interfacing Nanosized Noble Metal and Titanium Dioxide: *Jong-Won Yoon*[1]; Takeshi Sasaki[1]; Naoto Koshizaki[1]; [1]National Institute of Materials and Chemical Research (NIMC), Agency of Indus. Sci. and Techn., MITI, 1-1 Higashi, Tsukuba, Ibaraki 305-8565 Japan

Photoelectrochemical nanocomposites based on coupling TiO_2 matrix with the nanosized noble metal (Ag, Au, Pt) showed promising photoelectrode properties. The M/TiO_2 (M=Ag, Au, Pt) nanocomposite thin films were deposited on ITO glass substrates using co-sputtering method. TiO_2 in rutile form is a dominant crystalline phase for as-deposited nanocomposite films. Along with heat treatment up to 600°C, XRD peaks of rutile phase as well as those of noble metal increased in intensity and decreased in width, indicating the growth of crystallites. From the TEM observation, the platinum particle size ranged from 1 to 2 nm in as-deposited Pt/TiO_2 nanocomposite and increased to 5-10 nm by heat-treatment at 600°C. The anodic photocurrents of Pt/TiO_2 were observed not only in UV range but also in visible light range. Photocurrent in visible region can be attributed to the interfacial states between homogeneously dispersed noble metal particles and TiO_2 matrix.

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The Effect of DC-Electric-Field on II-VI Semiconductor Nanocrystal Embedded in Indium Tin Oxide Film: *Aiko Narazaki*[1]; Takeshi Sasaki[1]; Naoto Koshizaki[1]; Toshiaki Hirano[2]; Katsuhisa Tanaka[3]; Kazuyuki Hirao[2]; [1]National Institute of Materials and Chemical Research (NIMC), Agency of Indust. Sci. and Techn., MITI, 1-1 Higashi, Tsukuba, Ibaraki 305-8565 Japan; [2]Kyoto University, Dept. of Mat. Chem., Grad. Sch. of Eng., Sakyo-ku, Kyoto 606-8501 Japan; [3]Kyoto Institute of Technology, Fac. of Eng. and Design, Matsugasaki, Sakyo-ku, Kyoto 606-8585 Japan

The DC-electric-field effect on both the crystallization of II-VI semiconductor nanoparticle and its optical second-order nonlinearity has been investigated. The II-VI semiconductor nanoparticles of CdS or CdSe doped in indium tin oxide (ITO) thin films were fabricated via pulsed laser deposition as well as r.f. magnetron sputtering. As for the as-deposited films containing CdSe, the X-ray diffraction patterns indicate that CdSe crystallites are precipitated in an amorphous ITO matrix with (111) in zinc blende structure or (002) in wurtzite structure oriented. It was also observed that the CdSe crystallite grew with keeping its initial orientation when a DC voltage of 50V/cm was applied in the direction parallel to the film surface. The application of the electric field effectively enhanced the second-order nonlinearity, leading to an increase in the second-harmonic intensity by two orders ofmagnitude compared to that of the as-deposited films.

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Aluminum Titanate from Sol-Gel Nanosized Oxides: Sintering Behavior and Compound Formation Under Electric Field Application: Lia A. Stanciu[1]; Vladimir Kodash[1]; *Joanna R. Groza*[1]; Andrei Jitianu[2]; Oana Scarlat[2]; Maria Zaharescu[2]; [1]University of California-Davis, Mats. Sci. Dept., Davis, CA 95616 USA; [2]Institute of Physical Chemistry, Matls. Sci. Dept., 221 Splaiul Independentei, Bucharest, Sector 6 77208 Romania

Aluminum titanate (Al2TiO5) is an attractive ceramic material due to a combination of high temperature resistance and low thermal expansion. Processing of dense parts from initial Al2O3 and TiO2 powders is difficult because of a competing reaction, reduced final theoretical density, highly anisotropic structure, and low thermal stability of aluminum titanate at temperatures under 1180°C. A nanometer grain size of the initial precursors may alleviate some of these problems, such as enhancing the reaction rate and sintering, and control of microcracks. The sol-gel synthesis of binary Al2O3-TiO2 precursor was performed by hydrolysis and condensation of aluminum and titanium alkoxides at 70°C under a nitrogen flow and subsequent calcination. The amorphous powders were characterized by infrared spectroscopy, TEM, and DTA/TG analysis. A field activated sintering technique (FAST) was applied to simultaneously sinter and react sol-gel amorphous powders to form Al2TiO5. Densities close to theoretical and conversion to aluminum titanate (up to 99.9%) have been achieved by FAST sintering at 1050-1200°C for 10 minutes. For comparison, conventional sintering at 1300°C for 2 hours resulted in 88.9% aluminum titanate and approximately 75% density. Adsorbate elimination, surface activation and enhanced ion diffusion explain the enhanced sintering and compound formation by an external field application.

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Parameter Effects on the Stoichiometry of Nanoparticle Aggregated Oxide Films: *Naoto Koshizaki*[1]; Leszek Zbroniec[1]; Takeshi Sasaki[1]; [1]National Institute of Materials and Chemical Research (NIMC), Agency of Indust. Sci. and Techn., MITI, 1-1 Higashi, Tsukuba, Ibaraki 305-8565 Japan

In this report we characterize the cobalt oxide and iron oxide nanoparticle aggregated films prepared by laser ablation technique. The off-axis configuration and gas condensation process were adopted for deposition. Sintered iron oxide targets were ablated by the ArF excimer (193 nm) under various pressures of Ar, O_2, He, N_2, Ne and Xe. It was found that both the ambient gas type and its pressure strongly affect the crystal structure and composition of the deposition product. Depending on the ablation parameters the product of ablation was comprised of highest oxidized phase and lower oxide. The relative abundance of these oxides changed drastically with pressure, energy and ambient gas. These results can be explained in terms of ambient gas confinement effect of energetic species ejected by laser irradiation.

Sampling, Sensors & Control for High Temperature Metallurgical Processes: Smelting Applications

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

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

Tuesday PM — Room: 230
February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Adrian C. Deneys, Praxair, Appl. Res. and Dev., Tarrytown, NY. 10591 USA; Derek Fray, University of Cambridge, Dept. of Mats. Sci. and Metall., Cambridge CB2 3Q2 UK

2:00 PM

Imaging the Solidification of Molten Metal by Eddy Currents: *Minh Hoang Pham*[1]; Yingbo Hua[1]; *Neil Boon Gray*[2]; [1]The University of Melbourne, Elect. and Electr. Eng., Grattan St., Parkville, Victoria 3010 Australia; [2]The University of Melbourne, Chem. Eng. Dept., Grattans St., Parkville, Victoria 3010 Australia

This paper presents a technique for imaging the extent of solidification of molten metal flowing in a pipe based on the concept of eddy current. A mathematical model has been derived which relates the distribution of solidification to the scattered field induced in a solenoid external to the pipe. Based on the model, the shape and the thickness of metal solidification can be determined. The model is described by a set of integral equations which are solved numerically using the moments method. An iterative algorithm is developed to reconstruct the solidification distribution image. Experimental measurements have shown that the eddy current technique is sufficiently sensitive for practical purposes.

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Monitoring Method of the Condition of Slag-Cleaning Furnace in Mitsubishi Process: *Akira Kaneda*[1]; Nozomu Hasegawa[1]; [1]Mitsubishi Materials Corporation, Cen. Res. Instit., 1-297 Kitabukuro-cho, Omiya, Saitama 330-8508 Japan

Mitsubishi continuous process employs an electric furnace for settling matte from slag. Electrodes are immersed into the slag layer in the slag-cleaning furnace and the melt temperature is maintained by Joule's heat. Electrodes are immersed to the depth where the electric current is kept at a set value, and they are raised or lowered automatically to compensate for the deviation from it. Because electric resistance of the bath is varied with the matte level and the composition and temperature of the slag, anomalous situations in the furnace, such as rise of matte level or local abnormal state of melt can be detected by monitoring the immersion depth of the electrodes and its change with time. In this paper, relations between state of the furnace and electrode behavior are discussed based on the plant data, and the new and simple monitoring method of the condition of the furnace is proposed.

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Recent Improvements for Stable Operation of the Tamano Type Flash Smelting Furnace: Koji Noda[1]; Soichiro Tanaka[1]; Makoto Hamamoto[1]; *Masashi Kato*[1]; [1]Hibi Kyodo Smelting Company, Ltd., Tamano Smelter, 6-1-1 Hibi, Tamano, Okayama 706-0027 Japan

Tamano Smelter of Hibi Kyodo Smelting Company, Ltd. is operating with the Tamano Type Flash Smelting Furnace method which is a flash smelting furnace without slag cleaning furnace but which can discharge a slag equivalent to that from the slag cleaning furnace and with the development of coke combustion technology, the smelter exhibits a low operation cost under high productivity operation, at present the production has reached 263,000mtpy of anode copper. This paper outlines recent improvements for stable operation of the Tamano Type Flash Smelting Furnace, such as an automatic sampling system of granulated slag and subsequent analysis and a computer operation guide system.

3:15 PM Break

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Fault Diagnosis System for the Outokumpu Flash Smelting Process: *Eija Vapaavuori*[1]; Sirkka-Liisa Jamsa-Jounela[1]; Ilkka Kojo[2]; [1]Helsinki University of Technology, Lab. of Proc. Cont. and Auto., P.O. Box 6100, Espoo FIN-02015 HUT Finland; [2]Outokumpu Engineering Contractors, Box 862, Espoo 02201 Finland

Fault diagnosis systems have attracted growing interest in a number of engineering fields. The number of applications has increased and successful results have been widely reported. This paper presents and outlines an integrated fault diagnosis system for flash smelting. The system monitors the states of the process, classifies the type of the feed material, and also performs fault detection and diagnosis on the process equipment. If abnormalities in the states of the process or condition of the process equipment are detected, the system suggests suitable recovery actions to the process operators. The monitoring and classification are performed by using Kohonen Self-Organizing Maps. Fault detection on the process equipment supports the process monitoring and it is performed on the basis of the symptoms and knowledge stored in the rule base and process equipment knowledge base. The results of the monitoring of the process states and the fault detection on the process equipment are integrated in a decision making module by using decision rules. The decision making is carried out by using the forward chaining with the Rete-algorithm as its reasoning strategy.

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Real Time Determination of Metal Losses in Furnace Dusts by X-Ray Fluorescence: *Gerhard A. Meyer*[1]; Paul Mink[2]; [1]EDAX, Inc., 6161 Busch Blvd., Suite 305, Columbus, OH 43229 USA; [2]CORUS-IJmuiden, Hoogovens Corporate Services, B.V., P.O. Box 10000, IJmuiden 1940 CA, The Netherlands

A novel instrument for the determination of iron, titanium, zinc, calcium, and other metals real-time directly in the fumes emitted from pyrometallurgical processes has been developed. Based on X-Ray Fluorescence, the instrument can detect numerous metals simultaneously in the fume every 6 seconds and report their concentration on a mass per unit time basis real time. The use of the metal fume analyzer has proven a useful tool for dynamically monitoring changes in metallic content of fume present in the exhaust ducts of various metal producing processes. In these applications, the fume chemistry is correlated with changing process parameters for the purposes of: Reducing metal fume; Optimizing converter processes and practices; Slag management; waste-gas cleaning system optimization, and; Reducing emissions to the atmosphere. Actual Real

time elemental concentration profiles from different lots of steelmaking will be presented. Changes in the pyrometallurgical practice that results in lowered losses will also be discussed.

Second Global Symposium on Innovations in Materials Process & Manufacturing: Sheet Materials: Primary Processing Developments

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

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

Tuesday PM
February 13, 2001

Room: 228
Location: Ernest N. Morial Convention Center

Session Chairs: Iver Anderson, Ames Laboratory, Ames, IA 50011 USA; Anthony Rollett, Carnegie Mellon University, Pittsburgh, PA USA

2:00 PM Invited

Thin Strip Casting of Steel: a Process Comes of Age: *Anthony D. Rollett*[1]; Kanchan Kumari[1]; Paretosh Misra[1]; Alan W. Cramb[1]; [1]Carnegie Mellon University, Mats. Sci. & Eng., Wean Hall 4315, 5000 Forbes Ave., Pittsburgh, PA 15213-3890 USA

Thin strip casting shows promise as a processing route that connects casting directly to cold rolling. Bessemer's original twin-roll configuration from 1857 has proved to be the most reliable process for producing sheet of acceptable quality at thicknesses less than five millimeters. The process is already well established as a technically viable process for stainless steels. Carbon steels have proved to be more challenging because of the different solidification characteristics. Recent developments have demonstrated the viability of this processing route for this important class of sheet materials. This paper discusses an example of a carbon steel sheet cast in a thin strip caster with composition similar to a 1006 grade, which has been characterized for microstructure, texture and mechanical properties. The overall quality is good with uniform thickness and no apparent cracking or macrosegregation. The as-cast structure shows acicular and Widmanstätten ferrite in addition to the polygonal ferrite structure because of the higher cooling rate associated with the strip casting process. Since the initial microstructure of the strip cast steel is different from conventionally processed steel, its response to thermo-mechanical processing is also different. Detailed texture analysis of the deformed and annealed samples shows that moderate development of the gamma-fiber, {111} parallel to ND. The results from cold rolled and batch annealed material are very promising in terms of ductility and r-value, and open a door to a wide range of future applications.

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Improving Microstructure and Mechanical Properties of Near Net Shape: *Dierk Raabe*[1]; R. Kaspar[1]; [1]Max-Planck-Institut, Max-Planck-Str. 1, 40237 Duesseldorf, Germany

Sheet steel is manufactured by continuous casting, hot rolling, hot band annealing, cold rolling, and a final annealing treatment. Both, strip casting and thin slab casting technology offer the ability of bypassing the hot rolling processing stage or at least some of it by solidifying liquid steel on the surface of two rotating water cooled rolls and producing thin strips or thin slabs respectively. Strip casting and thin slab casting provides main advantages compared to the conventional process. First, it can supply steel bands which have mauch smaller starting thickness than those from conventional processing. Second, strip and thin slab cast steels exhibit weak initial crystallographic textures and weak through-thickness texture gradients, yielding favorable strength and deep drawing properties of the final sheets. Third, the high solidification rates obtained in these processes entails refined microstructures. The paper will give a detailed overview of the microstructures and the crystallographic textures obtained after strip and thin slab casting and further processing such as warm rolling, cold rolling and recrystallization annealing.

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Flexible Rolled Blanks for Automotive Application: *Carsten Greiserf*[1]; [1]Ford Forschungszentrum GmbH, Manuf. Sys., Suesterfeldstr. 200, Aachen 52072 Germany

The newly developed flexible rolling concept satisfies the ongoing demand for vehicle weight reduction in combination with improved passive safety. For this concept an advanced rolling gap control was installed in a cold mill. With that technology blanks with a continuous change of thickness can be rolled. These blanks are designated by a load optimised thickness and strength profile with a good formability. When annealed, the formability of the flexible rolled blank is comparable to a cold strip but superior to tailor welded blanks. In this study the formability of flexible rolled blanks and tailor welded blanks made of a deep drawing quality and a microalloyed high strength grade was investigated. Possible automotive applications are crash relevant and outer body parts.

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Processing of Bulk Nanostructured Copper by Repetitive Corrugation and Straightening (RCS): *Yuntian T. Zhu*[1]; Jianyu Huang[1]; Honggang Jiang[1]; Terry C. Lowe[1]; [1]Los Almaos National Lab, Mats. Sci. & Techn. Div., MS G755, Los Alamos, NM 87545 USA

A new process, Repetitive Corrugation and Straightening (RCS), has been developed to create bulk, nanostructured copper. In this investigation, a high purity (99.99%) copper bar measuring 6mm x 6mm x 50mm with an average grain size of 765 mm was used as the starting material. It was repetitively corrugated and straightened for 14 times with 90° rotations along its longitudinal axis between consecutive corrugation-straightening cycles. The copper was cooled to below room temperature before each RCS cycle. The grain size obtained after the RCS process was in the range of twenty to a few hundred nanometers, and microhardness was increased by 100%. This work demonstrates the capability of the RCS process in refining grain size of metal materials. The RCS process can be easily adapted to large-scale industrial production and has the potential to pave the way to large-scale structural applications of nanostructured materials.

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Surface Porosity Formation in Deposits Sprayed onto Flat Substrates by the Uniform Droplet Spray Process: Sukyoung Chey[2]; Jean-Pei Cherng[1]; Jung-Hoon Chun[1]; [1]Massachusetts Institute of Technology, Lab. for Manuf. and Produc. , Cambridge, MA 02139 USA; [2]Currently at i2 Technologies, 909 E. Las Colinas Blvd., Irving, TX 75039 USA

The porosity of spray-formed deposits at the substrate interface was investigated by using the uniform droplet spray (UDS) process in two steps. First, the formation of individual splats was analyzed to find the processing conditions for the ideal splat geometry. Second, the effect of multiple-splat interactions on surface porosity formation were studied by comparing splat solidification rate and droplet deposition rate. Experiments were performed with pure tin droplets, 416 mm and 271 mm in diameter, sprayed onto heated stainless steel and glass substrates. Results showed that slower splat solidification and higher droplet flux reduce surface porosity. A minimum surface porosity of 1.6% was achieved using the UDS process.

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Graphitization in Sheet Steels: *Jinhong Yang*[1]; [1]CDI Corporation, 18800 NW Rock Creek Circle, Apt. 200, Portland, OR 97229 USA

During the last two decades, finely dispersed, graphite nodules were observed in the microstructure of some cold rolled and annealed sheet steels. Graphitization has been a serious problem for sheet steel suppliers, because it impaired paintability and coating adherence of steel, reduced the tensile strength of steels. In order to explore the graphite formation mechanism, the characteristics of graphite and carbide phases in cold worked and annealed low alloy sheet steels were investigated in this study. Also, graphite dissolution kinetics was demonstrated as a function of austenitizing temperature and time.

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Alloy Design and Industrial Processing of Fe-40Al Sheets: *Seetharama C. Deevi*[1]; *D. H. Sastry*[1]; [1]Chrysalis Technologies, Inc., Res. Cen., 4201 Commerse Rd., Door 17, Richmond, VA 23234 USA

Intermetallics based on iron aluminides are attractive candidates for high temperature applications due to their high strength to weight ratio, and excellent oxidation and corrosion resistances. Environmental embrittlement, low room temperature ductility, and lack of economical processing techniques hampered the commercial utilization of iron aluminides based on FeAl in cast and wrought forms. As part of our program on intermetallics, we carried out alloy design of FeAl with the addition of boron, carbon, and molybdenum to optimize the room temperature ductility and high temperature strength of FeAl alloys. Alloys with an optimum combination of properties were powder processed to obtain thin sheets of FeAl by roll compaction, tape casting, and plasma spray techniques. To date, over 12,000 lbs. of excellent sheets of FeAl were commercially manufactured by roll compaction followed by sintering/annealing techniques using water atomized FeAl powders. In this presentaion, we will discuss the evolution of micro structure during the rolling process, and the importance of intermediate and final sintering/annealing steps for the recovery and recrystallization processes. In addition, we summarize the physical and mechanical properties of FeAl alloys, and these will be compared to some of the commercial iron based and nickel based alloys.

Structural Biomaterials for the 21st Century: Bone and Hydroxyapatite

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 Engineering Deparatment, Austin, TX 78712-1063 USA

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February 13, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Donald Lesuer, Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

2:00 PM Keynote

Clinical Significance of Calcium Phosphate Cement in Repairing of Bone Fracture: *Shigeo Niwa*[1]; Keizo Morikawa[1]; Yasuhito Aizawa[1]; Keiji Sato[1]; [1]Aichi Medical University, Dept. of Orthoped. Surg. Fac. of Med., 21 Karimata Yazone, Aichi-gun 480-1195 Japan

Recently, the remarkable development of technology in material science has brought an excellent outcome in treatment of bone fracture. There are many fixation devices of Titanium alloy etc., which have good histocompatibility to the bone and toughness, elasticity. But fracture related to osteoporosis almost doubled in the last decade. In the treatment of osteoporote fractures there are many problems, such as difficulty with rigid fixation by metal devices due to the fragility of the micro architecture of cancellous bone substitute material. A world wide use is to fill cancellous bone defects in fracture treatment. Resorbable calcium phosphate material is applicable in paste form as bone substitute for the osteoporotic fractures. This calcium phosphate cement, which we developed, has many advantages, which are higher compression strength, more normal cancellous bone, and early bone conduction activity.

2:30 PM Invited

Synthesis and Characterization of Porous Hydroxyapatite and Hydroxyapatite Coatings: *T. G. Nieh*[1]; B. W. Choi[1]; A. F. Jankowski[1]; J. Koike[2]; [1]Lawrence Livermore National Laboratory, P.O. Box 808, Mail Stop L-369, Livermore, CA 94550 USA; [2]Tohoku University, Sendai, Japan

A technique is developed to construct bulk hydroxyapatite (HA) with different cellular structures. The technique involves the initial synthesis of nanocrystalline hydroxyapatite powder from an aqueous solution using water-soluble compounds and then followed by spray drying into agglomerated granules. The granules were further cold pressed and sintered into bulks at elevated temperatures. The sintering behavior of the HA granules was characterized and compared with those previously reported. Resulting from the fact that the starting HA powders were extremely fine, a relatively low activation energy for sintering was obtained. In the present study, both porous and dense structures were produced by varying powder morphology and sintering parameters. Porous structures consisting of open micro-cells were constructed. Sintered structures were characterized using scanning electron microscopy and x-ray tomography. In the present paper, hydroxyapatite coatings produced by magnetron sputtering on silicon and titanium substrates will also be presented. The mechanical properties and interfacial adhesion of the coatings, measured by nanoindentation techniques, will be discussed. In addition, the interface microstructure examined using TEM will be described. Acknowledgement: This work is performed under the auspices of the U.S. Department of Energy through contract # W-7405-Eng-48 with Lawrence Livermore National Laboratory.

2:50 PM

Crystallinity Control of Hydroxyapatite and the Related Calcium Phosphates by Mechanical Grinding (MG) Method: *Takayoshi Nakano*[1]; Atsuyuki Tokumura[1]; Yukichi Umakoshi[1]; [1]Osaka University, Dept. of Mats. Sci. and Eng. and Grad. Sch. of Eng., 2-1, Yamada-oka, Suita, Osaka 565-0871 Japan

Crystallinity of hydroxyapatite (HAp) and the related calcium phosphates for regenerating hard tissue was controlled by MG method and the subsequent heat treatment. The HAp, Fluorapatite (FAp), Calcium-deficient apatite (CDAp), alpha- and beta-tricalcium phosphates, Tetracalcium phosphate and Octacalcium phosphate were used as initial materials. The variations in crystallinity and crystal structure were examined by the XRD method during the MG and the following heat treatment. Assignment of the infrared spectra was also performed by a Fourier transform infrared spectroscopy. Crystallinity based on crystal size and crystal elastic strain decreased with grinding time and the decreasing rate depended on type of calcium phosphates. Crystallographic diffraction peaks, for example, disappeared more rapidly in HAp than those in CDAp and FAp. The recovery process of crystallinity in the milled powders for 72h was investigated. The crystallinity of the MG powders recovered even at low temperatures and in some cases different crystal structure appeared during the heat treatment.

3:10 PM

Mechanical Integrity of Hydroxyapatite Coatings on Titanium Implants: *Yang Leng*[1]; Chenge Zhang[1]; [1]Hong Kong University of Science and Technology, Mech. Eng. Dept., Clear Water Bay, Kowloon, Hong Kong, China

Hydroxyapatite (HA) coated Ti alloy is a promising material for biomedical implants. The successful applications of such implants rely on fundamental understandings of mechanical behavior of the HA coatings. Experiments reveal the conventional cyclic bending tests cannot effectively evaluate the fatigue resistance of the HA

coatings on Ti substrates in both air and simulated body fluid (SBF). Alternative approaches including the Hertzian indentation and a new shear test method are employed. The fatigue tests of the coatings under the cyclic Hertzian indentation reveals that the fatigue damages in the HA coatings are severer in SBF than in air, and thin HA coatings exhibit less fatigue damages than the thick ones. The newly developed testing method for the coatings not only generates similar shear loading as the conventional ones, but also it is easy for alignment and for adapting coating thickness variation. Using the new test method, the static interfacial shear strength is evaluated and the coating resistance to shear loading is characterized. A fatigue mechanism of interfacial micro-flaw coalescence is suggested based on the fact that the interfaces between the coating and substrate are not fully bonded.

3:30 PM Break

3:40 PM Keynote

Fatigue of HAPEX-a Structural Bone Replacement Material: *K. Elizabeth Tanner*[1]; William J. McGregor[1]; Peter T. Ton That[1]; Ian M. Ward[2]; William Bonfield[3]; [1]Queen Mary University of London, IRC in Biomed. Mats., Mile End Rd., London E1 4NS UK; [2]University of Leeds, IRC in Poly. Sci. and Techn., Leeds LS2 9JT UK; [3]University of Cambridge, Dept. of Metall. and Mats. Sci., Pembroke St., Cambridge CB2 3QZ UK

HAPEX is a composite of hydroxyapatite in polyethylene and has been developed as a bone replacement material. It is clinically used in middle ear implants. To increase its mechanical properties hydrostatic extrusion has been used to orient the polyethylene. HAPEX has been fatigue tested at 37C in saline, to mimic the physiological environment, in tension-compression, torsion, combined tension-compression and torsion and in bending. S-N curves have been developed for these loading modalities. Isotropic HAPEX has a fatigue limit in the region of 5MPa and the addition of torque reduces the fatigue limit while increasing the phase angle increases it. Hydrostatically extruded HAPEX has increased static and dynamic mechanical properties and does not fracture under fatigue loading, but undergoes dynamic creep. HAPEX is more fatigue resistant in compression than tension as failure occurs at the filler polyethylene interface.

4:00 PM Invited

Anisotropic Elastic Moduli from Nanoindentation and Ultrasonic Velocity Measurements in Human Tibial Cortical Bone: *J. Gregory Swadener*[1]; Jae-Young Rho[2]; Jaofang Fan[2]; George M. Pharr[1]; [1]Oak Ridge National Laboratory, Mets. and Cer. Div., P.O. Box 2008, MS-6093, Oak Ridge, TN 37831 USA; [2]University of Memphis, Dept. of Biomed. Eng., Memphis, TN 38152 USA

Many biological materials are anisotropic. Cortical bone is one of these materials, and it contains microstructures, such as osteon lamellae, which are on the order of a few microns across. Nanoindentation is an effective technique to determine the mechanical properties of microstructures at the micron scale. However, the effects of anisotropy on nanoindentation have not generally been addressed. This study presents a method, which accounts for the effects of anisotropy on elastic properties measured by nanoindentation. The method is used to correlate elastic properties determined from earlier ultrasonic velocity measurements with recent nanoindentation results in human tibial cortical bone. The results from nanoindentation and ultrasonic velocities agree within experimental uncertainty. Research at the Oak Ridge National Laboratory SHaRE user facility was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:20 PM Invited

Application of Fracture Mechanics to the Study of Crack Propagation in Bone: *Jeffery C. Gibeling*[1]; Debbie R. Shelton[1]; Craig L. Malik[2]; [1]University of California, Dept. Chem. Eng. & Mats. Sci., One Shields Ave., Davis, CA 95616-5294 USA; [2]Hewlett Packard Company, Mailstop 1034D, 1000 NE Circle Dr., Corvallis, OR 97330 USA

The cortical or compact bone tissue that constitutes the material found in the dense regions of long bones can be treated as a ceramic matrix composite. Here, the matrix is composed of crystalline hydroxyapatite and the fibers are the osteon structures that provide nutrient pathways to the bone tissue. The osteons are oriented along the longitudinal axis of the bone, and it is clear that they also play an important mechanical role in determining the properties of the bone. Cortical bone is subject to fatigue damage accumulation in vivo, and this damage may lead to catastrophic crack propagation and even complete failure under repeated loads. Prevention of fatigue-related injuries necessitates knowledge of the mechanics and mechanisms of crack propagation in bone. In addition, the proper design of implantable biomaterials requires a thorough understanding of the mechanical properties of the surrounding bone. The application of fracture mechanics principles to the study of cortical bone is presented. Although bone is transversely isotropic, linear elastic methods are appropriate for crack propagation perpendicular and parallel to the longitudinal axis of the bone. The application of standard fracture and fatigue test methods to this material using compact type specimens is described. The only significant complication in these experiments is the need to keep the bone wet and at body temperature throughout the tests. Selected results for the propagation of cracks transverse to the longitudinal axis of equine cortical bone are presented. It is shown that cortical bone exhibits stable crack propagation and rising R-curve behavior as expected by analogy to fiber reinforced ceramic composites. The average crack growth initiation toughness is 4.5 MPa-m1/2. In addition, fatigue crack growth can be described by the usual Paris law with a stress exponent of approximately 10. This value is again within the range expected for fiber reinforced ceramics. In addition, the data reveal a threshold stress intensity factor of about 2 MPa-m1/2. Crack propagation under both fracture and fatigue conditions is also shown to be sensitive to variations in microstructure.

4:40 PM

Fracture Characteristics of Bovine Compact Bone with Relating Microstructure: *Kei-ichi Fukunaga*[1]; Mitsuo Niinomi[1]; Takashi Kodama[1]; Hirokazu Tajima[1]; [1]Toyohashi University of Technology, Dept. of Product. Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Aichi 441-8580 Japan

The characteristics concerning bones are poorly understood, but should be driving the requirement for developing biomaterials. There is still very little knowledge about fracture toughness and about the effect of microstructure on fracture toughness of bone. The effect of microstructure on fracture characteristics of compact bovine bone was investigated with the cooperation of fractography in present study. The fracture toughness tests were carried out on specimens which were machined from bovine femurs to make the crack propagation direction in the longitudinal or circumferential direction. The effects of anisotropy and moisture on static fracture toughness, KQ, were investigated. KQ of samples in the circumferantial directions is greater than that in longitudinal directions. The fracture toughness of wet samples is significantly higher than that of dry sample. The osteons were sheared in a zig-zag manner in wet samples by the longitudinal load while the crack propagated by flat shearing of osteons in dry samples.

5:00 PM

Micromechanisms of Crack Nucleation and Growth in Dental Ceramics: T. Chang[1]; K. Barbee[2]; R. Wang[2]; R. Seghi[3]; *W. O. Soboyejo*[1]; [1]Princeton University, Princeton Mats. Instit. and the Dept. of Mech. and Aero. Eng., Princeton, NJ 08544 USA; [2]The Ohio State University, Dept. of Mech. Eng., Columbus, OH 43210 USA; [3]The Ohio State University, Coll. of Dentist., Columbus, OH 43210 USA

This paper presents the results of recent studies of the micromechanisms of crack nucleation and growth in dental ceramic crowns. These include leucite-reinforced glass matrix composites and model multilayered structures with equivalent elastic properties to crown/dentin assemblies. The micromechanisms of crack nucleation are elucidated via Hertzian indentation studies under monotonic and cyclic loading. The mechanisms of fatigue crack growth are also examined under different loading conditions that simulate the range of stress states associated with occlusal contact.

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

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

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

★★★

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

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

Alumina & Bauxite: Bayer Process Development

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

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

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

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

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

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

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

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

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

10:00 AM Break

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

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

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

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

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

11:10 AM

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

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

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

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

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

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

8:30 AM

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

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

9:00 AM

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

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

9:30 AM

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

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

10:00 AM Break

10:15 AM

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

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

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

10:45 AM

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

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

11:15 AM

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

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

Aluminum Reduction Technology - Stream I: Modeling and Magnetics

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

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

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

8:30 AM

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

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

9:00 AM

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

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

9:25 AM

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

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

9:50 AM

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

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

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

10:15 AM Break

10:25 AM

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

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

10:50 AM

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

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

11:15 AM

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

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

11:40 PM

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

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

Aluminum Reduction Technology - Stream II: Sampling & Sensors

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

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

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

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

8:30 AM

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

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

9:00 AM

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

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

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

9:25 AM

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

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

9:50 AM

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

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

10:15 AM Break

10:25 AM

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

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

10:50 AM

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

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

11:15 AM

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

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

11:40 AM

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

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

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

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

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

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

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

8:30 AM Introductory Comments by Program Organizers

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

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

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

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

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

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

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

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

10:20 AM Break

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

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

10:55 AM
Panel Discussion with Questions from Audience

Cast Shop Technology: Inclusions & Alloying

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

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

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

8:30 AM

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

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

8:55 AM

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

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

9:20 AM

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

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

9:45 AM

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

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

10:10 AM Break

10:20 AM

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

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

10:45 AM

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

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

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

11:10 AM

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

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

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

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

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

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

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

8:30 AM

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

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

9:00 AM

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

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

9:30 AM

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

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

10:00 AM

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

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

10:30 AM

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

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

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

11:00 AM

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

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

11:30 AM

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

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

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

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

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

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

8:30 AM

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

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

8:55 AM

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

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

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

9:20 AM

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

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

9:45 AM

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

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

10:10 AM Break

10:20 AM

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

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

10:45 AM

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

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

11:10 AM

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

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

11:35 AM

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

10:15 AM Break

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

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

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

11:00 AM Invited

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

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

11:25 AM Invited

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

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

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

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

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

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

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

8:30 AM Invited

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

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

9:00 AM

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

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

9:20 AM

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

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

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

9:40 AM

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

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

10:00 AM Break

10:20 AM Invited

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

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

10:50 AM

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

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

11:10 AM

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

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

11:30 AM

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

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

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

11:50 AM

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

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

General Abstract Sessions: Solidification Processing

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

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

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

8:30 AM

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

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

8:55 AM

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

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

9:20 AM

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

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

9:45 AM

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

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

10:10 AM Break

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

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

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

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

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

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

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

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

General Abstract Sessions: Composites

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

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

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

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

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

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

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

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

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

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

9:45 AM

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

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

10:10 AM Break

10:20 AM

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

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

10:45 AM

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

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

11:10 AM

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

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

11:35 AM

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

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

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

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

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

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

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

8:30 AM

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

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

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

8:55 AM Invited

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

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

9:20 AM

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

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

9:40 AM

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

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

10:00 AM Break

10:15 AM

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

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

10:35 AM

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

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

10:55 AM

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

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

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

11:15 AM

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

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

11:35 AM

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

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

International Symposium on Deformation and Microstructure in Intermetallics: Creep

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

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

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

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

8:30 AM Invited

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

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

9:00 AM Invited

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

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

9:30 AM Invited

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

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

10:00 AM Break

10:20 AM Invited

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

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

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

10:50 AM Invited

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

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

11:20 AM

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

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

11:40 AM

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

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

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

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

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

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

8:30 AM Keynote

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

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

9:15 AM Invited

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

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

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

9:45 AM

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

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

10:15 AM Break

10:30 AM

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

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

11:00 AM

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

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

11:30 AM

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

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

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

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

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

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

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

8:30 AM Invited

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

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

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

8:55 AM

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

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

9:15 AM Invited

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

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

9:35 AM

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

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

9:55 AM

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

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

10:15 AM Break

10:30 AM

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

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

10:50 AM

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

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

11:10 AM

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

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

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

11:30 AM

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

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

11:50 AM

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

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

Lightweight Alloys for Aerospace Applications: Advances in Joining

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

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

Wednesday AM — Room: 213
February 14, 2001 — Location: Ernest N. Morial Convention Center

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

8:30 AM Invited

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

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

9:00 AM

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

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

9:30 AM

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

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

9:55 AM

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

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

10:15 AM

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

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

10:35 AM

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

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

11:00 AM

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

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

11:25 AM

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

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

11:45 AM

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

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

Magnesium Technology 2001: Physical Metallurgy

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

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

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

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

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8:30 AM

Application of Digital Image Analysis Technique to Quantify

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

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

8:55 AM

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

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

9:20 AM

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

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

9:45 AM

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

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

10:10 AM Break

10:20 AM

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

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

10:45 AM

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

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

11:10 AM

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

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

11:35 AM

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

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

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

Materials Processing Fundamentals V

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

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

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

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

8:30 AM

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

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

8:55 AM

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

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

9:20 AM

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

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

9:45 AM

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

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

10:10 AM Break

10:20 AM

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

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

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

10:45 AM

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

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

11:10 AM

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

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

Materials & Processes for Submicron Technology: Device Characterization and Integration

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

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

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

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

8:30 AM Invited

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

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

9:00 AM Invited

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

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

9:30 AM

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

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

9:50 AM

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

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

10:20 AM Break

10:40 AM Invited

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

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

11:10 AM Invited

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

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

Modeling of High Temperature Alloys: Process Modelling

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

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

Wednesday AM — Room: 221
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Session Chair: David Furrer, Ladish, Research, Cudahy, WI 53110 USA

8:30 AM Opening Remarks:

Lesh Patel; Special Metals Corporation

8:35 AM

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

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

9:00 AM

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

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

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

9:25 AM

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

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

9:50 AM Break

10:10 AM

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

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

10:35 AM

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

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

11:00 AM

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

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

11:25 AM

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

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

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

Properties of Nanocrystalline Materials: Modeling and Microstructure

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

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

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

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

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

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

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

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

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

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

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

10:10 AM Break

10:30 AM

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

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

10:50 AM

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

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

11:10 AM

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

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

11:30 AM

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

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

11:50 AM

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

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

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

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

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

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

8:30 AM

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

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

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

9:00 AM

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

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

9:25 AM

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

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

9:50 AM

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

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

10:15 AM Break

10:25 AM

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

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

10:50 AM

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

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

11:15 AM

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

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

11:40 AM

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

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

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

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

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

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

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

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

8:30 AM

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

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

8:55 AM

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

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

9:20 AM

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

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

9:40 AM

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

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

10:00 AM Break

10:20 AM

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

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

10:40 AM

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

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

11:00 AM

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

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

11:20 AM

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

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

Teaching and Learning Solid State Diffusion

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

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

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

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

8:30 AM

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

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

8:55 AM

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

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

9:20 AM

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

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

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

9:45 AM Break

10:10 AM

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

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

10:35 AM

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

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

11:00 AM

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

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

11:25 AM

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

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

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

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

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

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

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

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

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

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

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

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

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

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

3:50 PM Break

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

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

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

Abstract text is unavailable.

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

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

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

5:20 PM

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

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

Aluminum Reduction Technology - Stream I: Fundamentals

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

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

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

2:00 PM

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

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

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

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

2:50 PM

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

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

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

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

3:40 PM Break

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

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

4:15 PM

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

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

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

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

Aluminum Reduction Technology - Stream II: Advanced Control

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

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

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

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

2:00 PM

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

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

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

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

2:50 PM

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

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

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

3:15 PM

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

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

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

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

4:15 PM

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

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

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

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

Bauxite Residue Treatment: New Development: Session 1

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

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

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

2:00 PM

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

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

2:25 PM
Filtering Dike: Cruz de Jesús Silva[1]; [1]CVG-Bauxilum, Manejo de Lodo, Zona Industrial Matanzas, Puerto Ordaz, Bolivar 8015 Venezuela

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

2:50 PM
Construction of a Pilot Plant for the Neutralization of Water Pond: Ricardo Alfredo Galarraga[1]; [1]CVG-Bauxilum, Manejo de Lodo, Zona Industrial Matanzas, Puerto Ordaz, Bolivar 8015 Venezuela

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

3:15 PM
Recovery and Utilization of Iron from Red-Mud: *Brajendra Mishra*[1]; Anthony Staley[1]; David Kirkpatrick[2]; [1]Colorado School of Mines, Metall. and Matls. Eng., 1500 Illinois St., Golden, CO 80401 USA; [2]Kaiser Aluminum and Chemical Corporation, P.O. Box 3370, Gramercy, LA 70052 USA

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

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

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

4:05 PM
Smelting Reduction of Red Mud for the Recovery of Iron and Titania: Slag/Metal Equilibrium Studies: Animesh Jha[1]; Srikanth Srinivasan[2]; Amitava Bandopadhyaya[3]; Thomas C. Alex[3]; [1]University of Leeds, Dept. of Mats., Clarendon Rd., Leeds LS2 9JT UK; [2]National Metallurgical Laboratory Madras Centre, CSIR Madras Complex, Post TTTI, Tharamani, Chennai, Tamilnadu 600 113 India; [3]National Metallurgical Laboratory, Ferr. Proc. Div., P.O. Burma Mines, Jamshedpur, Bihar 831 007 India

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

Carbon Technology: Cathode Performance

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

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

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

2:00 PM Introductory Comments

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

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

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

2:50 PM

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

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

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

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

3:40 PM Break

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

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

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

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

Cast Shop Technology: Degassing and Alkali Metal Removal

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

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

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

2:00 PM

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

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

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

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

2:50 PM

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

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

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

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

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3:50 AM

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

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

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

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

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

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

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

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

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

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

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

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

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

2:00 PM

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

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

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

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

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

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

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

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

4:00 PM

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

4:10 PM

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

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

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

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

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

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

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

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

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

2:00 PM Invited

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

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

2:25 PM Invited

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

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

2:50 PM Invited

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

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

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

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

3:50 PM Invited

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

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

4:10 PM Invited

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

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

4:35 PM Invited

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

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

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

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

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

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

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

2:00 PM Invited

Texture, Deformation Mechanisms and Forming Properties of Hexagonal Alloys: *Marie Jeanne Philippe*[1]; [1]Université de Metz,

LETAM, Ile du saulcy BP 80794, Metz, Cedex 1 57012 France

The mechanical properties of hexagonal alloys are strongly anisotropic because of the associated sharp texture and the anisotropy of the HCP monocrystal. The present paper reviews the following aspects associated with forming of Zinc, Titanium and Zirconium Alloys: (1) initial texture and texture evolution during deformation; (2) active deformation mechanisms; (3) mechanical properties associated with (1) and (2), and (4) modeling and prediction of mechanical properties. Deformation can be accommodated by both glide and twinning, and their relative contribution depends on the alloying elements, the grain size, the grain orientation, and temperature and strain rate conditions. Yield stress and ductility can be directly related to the deformation mechanisms activated. The present work demonstrates the qualitative correlation between items (1) to (3) above, and the forming properties for all the alloys studied. A correlation is made between deformation texture and the composition of the alloys. We show that it is possible to model the texture evolution and to predict the mechanical properties of the alloys studied taking into account the initial texture and the active deformation mechanisms. Predictions and experimental results are compared and the most important deformation parameters are identified and discussed. As a conclusion, we identify the next steps required to make progress in this type of work.

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Identification of Material Parameters in Yield Functions for Textured Titanium Sheets: *Chi-Sing Man*[1]; Ying Zhang[2]; Yu Xiang[3]; Mojia Huang[1]; [1]University of Kentucky, Dept. of Math., 715 Patterson Office Tower, Lexington, KY 40506-0027 USA; [2]Xiamen University, Dept. of Mats. Sci., Xiamen, Fujian 361005 China; [3]University of Kentucky, Dept. of Chem. and Mats. Eng., 177 Anderson Hall, Lexington, KY 40506-0046 USA

Man has recently proposed a hierarchy of yield functions which, in increasing sophistication, would take account of the effects of crystallographic texture on the plastic flow behavior of weakly-textured orthorhombic sheets of hexagonal metals. In this paper we examine: (i) whether the two yield functions lowest in the hierarchy would be adequate for describing the angular dependence of the yield stress and of the r-value for commercially pure titanium sheet in uniaxial tension tests; (ii) whether the experimental data on the angular dependence of the uniaxial yield stress and of the r-value would suffice for the identification of material parameters in the two yield functions.

2:50 PM

Role of Twinning in the Constitutive Response of Zirconium: *Carlos N. Tomé*[1]; George C. Kaschner[1]; Thomas A. Mason[1]; [1]Los Alamos National Laboratory, Mats. Sci. and Techn., MST-8, Mail Stop G755, Los Alamos, NM 87545 USA

Polycrystal models have been traditionally used for understanding and describing texture evolution during plastic forming. More recently, they have been used for understanding the hardening of the aggregate, through the correlation between the macroscopic stress and the internal hardening mechanisms at the grain level. In hcp materials, twinning is an important deformation mode and its contribution to texture and hardening has to be accounted for. While the former has been incorporated in polycrystal models, very little has been done to account for the hardening contribution. In a recent study[1] we use tension and compression results of rolled Zr tested along different directions to adjust a constitutive law. In doing so we account for the twinning contribution to texture using a Predominant Twin Reorientation scheme, and for its contribution to hardening by treating the twinning activity in a similar way as we treat the slip activity. Although the resulting constitutive description is successful in describing the material response at moderate strains (~20%), two conclusions are extracted from this study concerning the hardening contribution from twinning: the latter is more relevant to the constitutive law than the associated texture contribution, and an approach different than the one for slip is required to describe it. In the present work we extend a model originally proposed by Karaman et al.[2] for Hadfield steel, and reformulate the constitutive response of Zr using a twinning model that accounts for directional barriers to dislocation motion posed by the twin lamellae in the grains, and also for the evolution of twin fraction with deformation in the grains. In addition to the tensile and compressive tests, we use Orientation Image Microscopy (OIM) of bent Zr beams to elucidate the evolution of twinning with deformation and as a way of checking the consistency of the model. [1]C.N. Tomé, P.J. Maudlin, R.A. Lebensohn and G.C. Kaschner, Acta materialia, submitted. [2] I. Karaman, H. Sehitoglu, A.J. Beaudoin, Y.I. Chumliakov, H.J. Maier and C.N. Tomé, Acta materialia 48 (2000) 2031.

3:10 PM

Texture Effects on Dynamic Shear Response in Ti-6Al-4V plates: *Bimal Kad*[1]; Scott Schoenfeld[2]; [1]University of California-San Diego, Struct. Eng., 409 University Ctr., La Jolla, CA 92093-0085 USA; [2]US Army Research Laboratory, Impact Phys., Terminal Effects Div, Bldg. 4600, AMSRL-WM-TD, Aberdeen Proving Ground, MD 21005-5069 USA

Due to the asymmetric and anisotropic nature of crystallographic slip and twinning in HCP materials. the bulk mechanical properties are strongly affected by the orientation distribution of the crystals in the polycrystal aggregate. In this presentation we examine the in-plane shear response of Ti-6Al-4V alloys that are nominally textured via routine deformation and thermal processing schedules. For rolled plates, a 2-dimensional constitutive model for slip and twinning (treated here as pseudo-slip) is derived for the HCP single crystal, and embedded into a finite element model representing a spatial distribution of single crystals. The polycrystal mechanical response is examined with respect to macroscopic shear loading as may take place during a dynamic punch through process.

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Creep Mechanisms and Anisotropic Creep in Zr Alloys: *K. L. Murty*[1]; [1]North Carolina State University, P.O. Box 7909, Raleigh, NC 27695-7909 USA

Zirconium alloys are commonly used for cladding radioactive UO_2 in water reactors and possess hcp structure at the reactor operating temperatures. Zr alloyed mainly with Sn and Fe (referred to as Zircaloys) is widely used while recently Nb-additions are considered for long-term application with improved corrosion resistance. Creep characteristics of these thin-walled tubing materials are evaluated for predicting the dimensional changes of these structures in-service. Zircaloy-4 that finds application in the pressurized water reactors exhibit deformation behavior characteristic of Class-M (also known as Class-II) alloys with climb of edge dislocations as the dominant mechanism while Nb additions are shown to lead to alloy class (Class-A or Class-I) behavior with the stress exponent (n) of around 3 (identified as the viscous glide due to solute locking). At low stresses, both materials exhibit Newtonian viscous creep behavior with n=1 identified to be Coble creep. Crystallographic textures developed in these ma terials during the thermomechanical processing result in highly anisotropic deformation, and the effects of stress level and alloying on creep under equibiaxial loading will be described. The significance of transitions in creep mechanisms (as lower stresses are encountered) on the predictability of dimensional changes in-service will be highlighted.

4:20 PM

Diffraction Studies of Twinning in Pure Zirconium: *Partha Rangaswamy*[1]; Donald W. Brown[1]; George C. Kaschner[2]; John F. Bingert[3]; Luc L. Daemen[4]; [1]Los Alamos National Laboratory, Mats. Sci. Techn., MST-8, MS H805, Los Alamos, NM 87545 USA; [2]Los Alamos National Laboratory, Mats. Sci. Techn., MST-8, M/S G755, Los Alamos, NM 87545 USA; [3]Los Alamos National Laboratory, Mats. Sci. Techn., MST-6, M/S G770, Los Alamos, NM 87545 USA; [4]Los Alamos National Laboratory, LANCE-12, Lujan Center, M/S H805, Los Alamos, NM 87545 USA

An experimental study using x-ray and neutron diffraction was performed to quantify the twinning volume fraction in pure zirconium deformed under compression. Strongly textured clock-rolled pure zirconium was deformed to a maximum strain of 25% with the loading direction nearly parallel to the c-axis of the hexagonal close packed unit cell. The deformation was performed at applied strain rates between 0.001 and 3500/s, and test temperatures between 77

and 298K. Using both x-rays and neutrons, diffraction peak intensities were recorded as a function of strain in the various samples. The objective of this study was to demonstrate the effectiveness of using diffraction as a method to correlate the diffraction intensity changes with the plastic contribution of the macroscopic strains. The overall goal is to quantify the volume fraction of twinning and use it as a calibration for verifying volume fractions determined through orientation imaging microscopy.

4:40 PM

The Role of the HCP Phase on the Tensile Mechanical Properties of Two-Phase Co-27Cr-5Mo-0.05C Alloy: *Armando Salinas-Rodriguez*[1]; Antonio Mani-Medrano[1]; [1]Centro de Investigacion y de Estudios Avanzados del IPN , Carr. Saltillo-Monterrey km 13, P.O. Box 663, Saltillo, Coahuila 25000 Mexico

Isothermal aging of solution treated Co-27Cr-5Mo-0.05C alloy produces two-phase, FCC-HCP, microstructures. The amount of the HCP phase produced by aging at a given temperature exhibits a sigmoidal dependence on time. Alloys with varying amounts of HCP phase were used to investigate the effects of the phase distribution on their room temperature tensile properties and hardness. The results showed that the yield strength and hardness increase linearly with the increase in the amount of HCP phase. In contrast, the ductility remains constant for 15<%HCP<55. At larger or smaller amounts of HCP phase the ductility decreases linearly. It was also found that the amount of HCP phase, as measured by X-ray diffraction, increases significantly as a result of plastic deformation within the region of nearly constant ductility. Finally, the tensile fracture strength was found to be independent on the amount of HCP present at the start of the deformation. It is proposed that the tensile ductility of this material depe nds on the ability of the metastable FCC phase to transform to HCP dynamically during plastic straining. The region of slow decrease of the ductility with increasing %HCP was attributed to the dynamic softening associated with the strain induced transformation.

5:00 PM

The Basal Slip in Alpha-Zirconium: Experimental Evidences and the Role in Texture Formation: *Margarita Isaenkova*[1]; Yuriy Perlovich[1]; [1]Moscow State Engineering Physics Institute (Technical University), Met. Sci. Dept., Kashirskoe Shosse 31, Moscow 115409 Russia

Up to now the real participation of the basal slip in the plastic deformation of alpha-Zr is significantly underestimated. Several X-ray diffractometric methods were elaborated to follow reorientation of alpha-Zr grains by the plastic deformation of commercial Zr-based alloys. For grains with different initial orientations the trajectories of basal axes were constructed for the cases of gradual rolling of an originally textureless sample and transverse rolling of a sample with the previously developed texture. The obtained trajectories by intermediate grain orientations correspond to the preferential action of the basal slip, as it follows from the known models, connecting modes of texture formation with concrete mechanisms of plastic deformation. The basal slip results in formation of the intermediate quasi-stable rolling texture with basal axes, declined from ND to RD by 15-25°. Its stability up to deformation degrees of 50-60% is conditioned by the mutual equilibrium of opposite reorientation effects, accompanying action of basal and pyramidal slip systems. Only by the further increase of the deformation degree, this intermediate texture transforms into the final stable rolling texture with basal axes, declined from ND to TD by 30-50°.

General Abstract Sessions: Extraction and Processing

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Program Organizers: Thomas P. Battle, E. I. DuPont de Nemours & Company, Inc., White Pigments and Mineral Products, Edgemoor, DE 19809 USA; TMS, Warrendale, PA 15086 USA

Wednesday PM — Room: 212
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Mark E. Schlesinger, University of Missouri-Rolla, Dept. Metallur. Eng., Rolla, MO 65409-0001; Ibrahim Gaballah, INPL-CNRS, Vandoeuvre, France

2:00 PM

Chlorination: An Emerging Technology: *I. Gaballah*[1]; N. Kanari[1]; M. Djona[1]; E. Allain[2]; [1]Mineral Processing and Environmental Engineering Team, Rue du Doyen Marcel Roubault, B.P. 40, Vandoeuvre, Cedex 54501 France; [2]University of Missouri-Rolla, Sch. of Mines and Metall., Ctr. for Pyrometall., 215 Fulton Hall, Rolla, MO 65401 USA

Sodium hydroxide and chlorine are generated by the electrolysis of NaCl. Periodical imbalance of the consumption of these two products poses problems of storage for the operators. Chlorine surplus could be used for the extraction of valuable metals and for upgrading of raw materials. Extraction of tantalum and niobium from tantalo-columbite concentrates and tin slag, upgrading of chromite concentrate, recovery of valuable metals from spent catalysts by using chlorine technique are summarized. Thermodynamic and kinetics aspects of several chlorination reactions are outlined.

2:25 PM

Technology Selection for La Caridad Precious Metals Plant: *Irshad A. Rana*[1]; [1]Fluor Daniel, Mining, 43686 Skye Rd., Fremont, CA 94539 USA

Available and proven technologies were reviewed for the La caridad Precious Metals Plant to produce approximately 450 mt/year silver, 3 mt/year gold and assorted other metals from the anode slimes originating from the La Caridad Copper Refinery and some purchased materials. For the conventional process the technologies considered were autoclave leaching, roasting, Moebius parting plant, chemical precipitation of fine gold from slolution and oxygen assisted smelting and refining. The Phelps Dodge process was considered which consists of slimes leaching, sulfatization roast, silver leaching/electrowinning, gold leaching and solvent extraction/ precipitaion. Also evaluated was the complete hydrometallurgical wet chlorination process in which a series of leaching and precipitation steps are used to produce refined precious metals. A flowsheet was finally selected, plant built during 1999 and commissioned in year 2000.

2:50 PM

Production of Pure Metals and Semiconductive Materials: *A. V. Tarasov*[1]; Yu O. Mamayev[1]; [1]State Research Center of Russian Federation, State Rsch. Instit. on Non-Ferrous Mets., 13 Acad. Korolyov St., Moscow 129515 Russia

Issues relating to production of bismuth, antimony and tellurium of high purity for application relating to synthesis of low-temperature thermoelectric alloys based on bismuth and antimony chalcogenides have been discussed. Experimental investigations of physical and chemical aspects of the behavior of individual impurities of their combinations have been carried out using different crystallization methods. Process parameters for production of high-purity materials have been optimized. The necessity for integrated use of distillation and zone re-crystallization techniques for production of high-purity materials has been confirmed.

3:15 PM Break

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Hydrometallurgical Technologies of a Complex Silver-Gold Concentrate and its Calcine by Chlorine and Thiosulfate: *Shengming Xu*[1]; Jingming Xu[1]; Ru'an Chi[1]; Guocai Zhu[1]; [1]Tsinghua University, Inst. of Nuclear Energy Techn., Beijing, 102201 China

Leaching of a complex silver-gold concentrate and its calcine was investigated using chlorine and thiosulfate for extraction of gold and silver. The results of elementary experiments showed that the concentrate needed to be pre-treated prior to leaching. In order to eliminate the carbonaceous materials and decompose the sulfides, a series of roasting tests was firstly carried out; the roasting conditions of the concentrate were also chosen. The results showed that the additive had a favorable effect on extractive yield of silver in the temperature range of 400º-650º. Silver minerals in the concentrate will be dissociated, then oxidized into sulfate or chloride with a different additive. Following chlorine leaching of gold, the calcine was leached out by thiosulfate again. The extractive yields of gold and silver would excess 95% and 97%, respectively. This new route seems to be more environmental and economical than the traditional cyanide process.Keywords: Silver concentrate; Roasting; Leaching; Chlorine; Thiosulfate

3:55 PM

Thiourea Leaching of Gold and Silver from A Carbonaceous Silver Concentrate: *Shengming Xu*[1]; [1]Tsinghua University, Insti. of Nuclear Eng. Techn., Beijing 102201 China

An innovative process for thiourea leaching of gold and silver from a carbonaceous flotation concentrate assaying 17.4 g/t Au, 4301 g/t Ag and 6.71 % C, and containing different sulfide minerals, was investigated. According to the results of chemical phase analysis of gold and silver, the concentrate can be classified as refractory ore because silver is difficult to extract by traditional cyaniding or thiourea process. A thiourea carbon-in-pulp process, however, is suitable for the extraction of gold prior to silver preferentially. The influence of leaching temperature, leaching time, thiourea concentration, pH value and activated carbon added amount on extractive yield of gold was firstly investigated, and extractive yield of gold can reach 92% in this stage. In order to recover the residual silver, the leached gold residue further ground in a vibration miller for 10 min. was subsequently oxidized by a dilute ferric chloride solution, and most of silver minerals were dissolved into the solution. Thus, total extractive yield of silver will increase from 60% to 95%. Keywords: carbonaceous silver concentrate; thiourea carbon-in-pulp process; ferric chloride leaching

General Abstract Sessions: Composites/New Products and Processes

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Wednesday PM Room: 229
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: James C. Foley, Ames Laboratory, Metall. and Ceram. Prog., Ames, IA 50011 USA; Funsho Ojebuoboh, ASARCO, Denver, CO 80216 USA

2:00 PM

Metal/Polymer Interface and Solder Joint Reliability of a Wafer Level CSP: *Hun Han*[1]; Jin Yu[1]; [1]KAIST, Mat. Sci. and Eng., 373-1 Kusong-dong, Yusong-gu, Taejon 305-701 Korea

Wafer level chip scale package (WLCSP) has the highest potential compared with flip chip package (FCP), because they provide the benefits of real chip size package with low cost and high reliability. We use low modulus polymers (stress buffer layer; SBL) which were designed to relax the thermal strain generated at the solder joint. However, reliability of metal/polymer interface and solder joint are still critical problems to the WLCSP. We studied the reliabilities of metal/SBL interfaface and solder joint according to variation of the polymer surface treatments and the metal structure. To measurement of adhesion property on metal/SBL, peel strength was evaluated. In addition, the effects of under bump metallurgy (UBM) on the reliability solder joint at the WLCSP will be discussed.

2:25 PM

HVOF Application of Nickel and Nickel Alloy Jackets to Tungsten Heavy Alloy Penetrators: *John Vincent Kelley*[1]; [1]Army Research Laboratory, Weapons Mats, Div,, AMSRL-WM-MC, Bldg. 4600, Aberdeen Proving Ground, MD 21005 USA

There has been an increased desire to replace depleted uranium (DU) anti-armor penetrators with tungsten heavy alloy (WHA). Unfortunately, WHA does not perform ballistically nearly as well as DU. One effort to improve ballistic performance of WHA examines high velocity oxy-fuel (HVOF) coatings as a jacket material. The differences in the coefficients of thermal expansion (CTE) make it difficult to achieve adequate adhesion of most coatings to tungsten. Further compounding the problem are residual stresses inherent in thermally sprayed coatings. Early attempts to apply 0.010-0.020 inches of Ni to tungsten substrates yielded immediate disbonding and/or cracking of the coatings. This work is an investigation of modifications to parameters and coating thicknesses to minimize heat transfer to achieve better adhesion. The use of materials with a CTE nearer to that of tungsten as a bond coat was also examined as a method for increasing the overall adhesion of the coating system.

2:50 PM

Fatigue Behavior of Ceramic Matrix Composites with Nondestructive Evaluation (NDE) Techniques: *Jeongguk Kim*[1]; Peter K. Liaw[1]; Hsin Wang[2]; You-Tae Lee[3]; [1]University of Tennessee, Mats. Sci. and Eng., Knoxville, TN 37996-2200 USA; [2]Oak Ridge National Laboratory, Mets. and Ceram. Div., Oak Ridge, TN 37831 USA; [3]Kyongdo College, Dept. of Auto., Kyungbook 757-800 Korea

High-cycle fatigue behavior of continuous fiber reinforced ceramic matrix composites (CFCCs) was investigated with the aid of nondestructive evaluation (NDE) techniques. The NDE methods used for this investigation include ultrasonic testing (UT), infrared (IR) thermography, and acoustic emission (AE) techniques. Prior to fatigue testing, UT and IR thermography were used to characterize the initial defect distribution of CFCC samples, i.e., developing ultrasonic C-scans and thermal diffusivity maps, respectively. A qualitative correlation between the C-scan and thermal diffusivity map has also been obtained. During fatigue testing, AE sensors and an IR camera were used for in-situ monitoring of progressive damages of CFCC samples. A stress versus cycles to failure (S-N) curve has been provided to predict the lifetime of CFCC samples as functions of initial defects and progressive damages. UT and IR thermography were conducted on fractured samples after testing to compare progressive damages with the init ial defects. Microstructural characterization using scanning electron microscopy (SEM) was performed to investigate fracture mechanisms of CFCC samples. In this study, NDE techniques were used to facilitate a better understanding of fracture mechanisms of CFCCs during high-cycle fatigue testing.

3:15 PM

Oxide Dispersion Hardened Platinum Materials for High Temperature Applications: *Bernd Fischer*[1]; Dietmar Freund[2]; Rainer Volkl[2]; David F. Lupton[2]; [1]FH Jena-University of Applied Sciences, Dept. of Mats. Tech., Carl-Zeiss-Promenade 2, Jena D-07745 Germany; [2]FH Jena-University of Applied Science, Dept. of Mats. Sci., Carl-Zeiss-Promenade 2, Jena D-07745 Germany

In spite of their high prices platinum materials are well suited to being used as high temperature materials for varied technical applications. This paper reports on the fabrication, structure, properties and use of a new class of oxide dispersion hardened platinum materials (Pt DPH materials) with improved essential properties. The production follows a new route. By the addition of oxide-forming

elements (for example zirconium and yttrium) during the melting process it is possible to precipitate dispersoids in the course of the working process by means of internal oxidation. The materials Pt DPH, Pt-10%Rh DPH and Pt-5%Au DPH are available. The new oxide dispersion hardened platinum materials are excellent to process (forming, welding). Because of the dispersoids they have excellent grain stability in long-term use at the highest temperatures. Mechanical high temperature properties (stress-rupture strength, creep data) are given in the unwelded and welded state for the temperature range 1200°-1600°C and for the material Pt-10%Rh DPH up to 1700°C. In accordance with the practical use of the materials, the results of stress-rupture tests with periodical change of temperature and the effects of notches with special geometry are discussed. Under all test conditions the new oxide dispersion hardened platinum materials have not only an increased stress-rupture strength but also a good ductility even at the highest temperatures. These facts apply in particular to the welded state. Finally the paper reports on first tests of the new platinum materials for components in the glass industry. The excellent chemical stability of the materials in aggressive glass melts has been investigated. Compared with the previously known platinum materials, the Pt DPH materials show improved properties which lead to a considerable increase in the time to failure for components in the glass industry.

3:40 PM Break

3:50 PM

New Process for the Synthesis of Potassium Ferrate ($K_2Fe^{VI}O_4$): *N. Kanari*[1]; I. Gaballah[1]; C. Mathieu[2]; N. Neveux[2]; O. Evrard[2]; [1]Mineral Processing and Environmental Engineering Team, CNRS UMR 7569, ENSG INPL, BP 40, Vandoeuvre, Cedex 54501 France; [2]Universite Henri Poincare Nancy I, Lab. de Chimie du Solide Minerale, BP 239, CNRS UMR 7555, Vandoeuvre, Cedex 54506 France

Potassium ferrate is a superoxidant material containing iron in hexavalent state (Fe^{VI}). The conventional synthesis's methods are based on wet processing leading to ferrate decomposition and consequently low synthesis efficiency. A new process for the dry synthesis of potassium ferrate is developed and already patented. It consists to the reaction of iron sulfate with an oxidant in a postassium hydroxide medium. Synthesis is achieved in a rotary reactor at room temperature and the overall reaction was exothermic. The potassium ferrate (Fe^{VI}) synthesis' efficiency is about 60%. Kinetics aspects of the potassium ferrate synthesis are detailed.

4:15 PM

The Challenge for Lead-free Brass in Casting Alloys: *Funsho Ojebuoboh*[1]; [1]ASARCO, Inc, 495 E. 51st Ave., Denver, CO 80216 USA

Copper-based alloys, i.e., brasses and bronzes, are the preferred and traditionally alloys for sand-cast plumbing components, and more recently, permanent mold castings for finer water fixtures. As it is for other engineering alloys, lead is a particularly important additive to copper alloys where it imparts the key attributes of machinability and pressure tightness. To truly gain the benefits of these attributes, manufacturers use as much as 8% lead in sand castings. However, after the US Safe Drinking Water Act of 1986, the industry (ingot manufacturers through end-users) has endeavored to reduce the level of lead in the value chain. This paper examines EnviroBrass, a family of lead-free brasses, with particular attention to the development of the alloys, alloy properties, and obstacles to their adoption for drinking-water applications.

4:40 PM

Synthesis of Open Cell Metal Foams by Plasma Enhanced, Electron Beam Directed Vapor Deposition: *Douglas Ted Queheillalt*[1]; Yasushi Katsumi[1]; Haydn N.G. Wadley[1]; [1]University of Virginia, Mats. Sci. & Eng., 116 Engineers Way, P.O. Box 400745, Charlottesville, VA 22904-4745 USA

Open cell, reticulated metal foams have been synthesized by a plasma enhanced, electron beam-directed vapor deposition (EB-DVD) process and their physical and mechanical properties evaluated. The deposition process uses an open cell, reticulated polymer or vitreous carbon foam template upon which is deposited various metal and alloy coatings. The electron beam evaporated flux was entrained in a rarefied transonic gas jet and propagated along the flow stream lines through the template structure. During vapor transport, the atoms pass through a plasma created by a hollow cathode arc discharge system. These combined technologies lead to high deposition rates of atoms with high energies, promoting enhanced coating quality. After vapor deposition, the template may be removed by thermal decomposition resulting in ultra-lightweight metal foams composed of a three-dimensional open cell, reticulated structure possessing triangular ligaments with either hollow or carbon cores.

High Temperature Coatings - IV: Ceramic Coatings

Sponsored by: Materials Processing and Manufacturing Division, ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee, Surface Engineering Committee

Program Organizers: Narendra B. Dahotre, University of Tennessee Space Institute, Center for Laser Applications, Tullahoma, TN 37388 USA; Janet Hampikian, GA Institute of Technology, School of Materials Science & Engineering, Atlanta, GA 30332-0245 USA

Wednesday PM, February 14, 2001 — Room: 219, Location: Ernest N. Morial Convention Center

Session Chairs: Narendra B. Dahotre, University of Tennessee Space Institute, Dept. of Mats. Sci. and Eng., Tullahoma, TN 37388; Jeff Th De Hosson, University of Groningen, Dept. of Appl. Phys., Groningen 9747 AG The Netherlands

2:00 PM Invited

Properties of WC/C Coatings Investigated with Cross-Sectional TEM and Nanoindentation: *Jeff Th De Hosson*[1]; Nuno J. Carvalho[1]; [1]Un. of Groningen, Dept. Appl. Phys., Nijenborgh 4, Groningen 9747 AG The Netherlands

This paper concentrates on the interplay between microstructure and properties of coatings studied with cross-sectional transmission electron microscopy and nano-indentation techniques. Coatings of multilayer tungsten carbide/carbon (WC/C) were deposited by physical vapor deposition (PVD) onto steel substrates. The coatings are composed by a chromium columnar interlayer to improve the adhesion to the substrate, an intermultilayer of WC and carbon, and an interlaminar amorphous WC/C multilayer structure. The mechanical properties were evaluated with respect to the hardness and the effective Young's modulus. The hysteresis loops are analyzed and discussed in detail. Moreover a new technique for cross-sectional transmission electron microscopy of the nano-indentations was developed to gain a better view of the interplay between the mechanical properties, the microstructure and the chemical composition of the system. The information obtained by this technique is also correlated with the load-displacement data from a nano-indentation cycle to reveal detailed deformation mechanisms

2:25 PM Invited

Electrophoretic and Electrolytic Deposition of Ceramic Coatings: *Igor Zhitomirsky*[1]; [1]McMaster University, Dept. of Mats. Sci. and Eng., 1280 Main St. West, Hamilton, Ontario L8S 4L7 Canada

Two processes were used to prepare oxide coatings by electrodeposition: the electrophoretic process (EPD) that is based on the use of suspensions of ceramic particles and the electrolytic process (ELD) that starts from solutions of metal salts. ELD enables formation of nanostructured thin films. EPD is an important tool for preparation of thick ceramic films and body shaping. The feasibility of electrolytic deposition of various ceramic materials has been demonstrated. It was shown that chemical problems related to ELD of some important ceramic materials could be solved by use of specific precursors. An important finding was the possibility of electrochemical intercalation of cationic polyelectrolytes with inherent binding prop-

erties into ELD deposits. Advanced solvent-binder-dispersant systems were developed for deposition of consecutive ceramic layers of controlled thickness in multilayer EPD processing. Thin and thick films were deposited as monolayers or laminates on various substrates, including metals, conducting ceramics, carbon fibers and mats, platinized silicon wafers. The results presented involve the examination of the microstructure, composition and crystallization behavior of the deposits. Deposition yield was evaluated under various experimental conditions. Mechanisms of EPD and ELD, new developments and applications of the two methods are discussed.

2:50 PM

Yttria Stabilized Zirconia/Alumina Coatings Deposited by Combustion Chemical Vapor Deposition: David W. Stollberg[1]; Monique McIntosh[1]; W. Brent Carter[1]; *Janet M. Hampikian*[1]; [1]Georgia Institute of Technology, Mats. Sci. and Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Yttria stabilized zirconia/alumina (YSZ/alumina) thin films were grown on sapphire substrates by combustion chemical vapor deposition, using yttrium and zirconium 2-ethylhexanoate and aluminum acetylacetonate as precursors. These compounds were dissolved in toluene and burned in a flame to produce five different compositions of YSZ/alumina: 15, 30, 45, 62.8 (the eutectic composition) and 80 mol% alumina. The mechanical properties of these films were investigated as a function of alumina content using nanoindentation. The highest fracture toughness of the films was found to occur at alumina compositions between 30 and 45%. The thermal stability of some films deposited at the eutectic composition was investigated as a function of temperature and annealing time. The as deposited microstructure exhibits particle sizes at the nanometer level, characterized by transmission electron microscopy. Particle sizes increased on average to approximately 0.4 microns after annealing for 5 hours at 1500ºC.

3:10 PM

High Temperature Oxidation of VC Coated H13 Steel: *Swapnil V. Shah*[1]; Narendra B. Dahotre[1]; [1]University of Tennessee Space Institute, Dept. Mats. Sci. & Eng., Cen. for Laser Appl., 411 B.H. Goethert Parkway, Tullahoma, TN 37388 USA

ASISI H13 is the most widely used material for die-casting dies. High temperature oxidation of the die material during service affects die lifetime. Hard ceramic coating like VC can significantly increase the die life. In the present study attempts are made to coat H13 by VC using laser surface engineering technique. Oxidation behavior of coated H13 steel at high temperature (up to 1000ºC) is studied along with detailed microstructural and mechanical properties evaluation.

3:30 PM

Near Net Shape Forming of Hafnium-Based Ceramic Components: Synthesis and Characterization: *Arvind Agarwal*[1]; Tim McKechnie[1]; Samuel J. Causey[2]; Mark M. Opeka[3]; [1]Plasma Processes, Inc., 4914 D Moores Mill Rd., Huntsville, AL 35811 USA; [2]Southern Research Institute, 757 Tom Martin Dr., Birmingham, AL 35211 USA; [3]Naval Surface Warfare Center, Carderock Div., Code 681, 9500 McArthur Blvd., West Bethesda, MD 20187-5700 USA

Ultrahigh temperature applications such as combustion chamber liners, rocket thrusters, thermal protection systems for carbon/carbon composites and leading edges of the spacecraft require materials, which are protective and oxidation resistant at temperatures higher than 20000C. Refractory ceramics such as hafnium diboride (HfB2), hafnium carbide (HfC) and hafnium nitride (HfN) are candidate materials because of their high melting points, low coefficient of thermal expansion, high erosion and oxidation resistance. Conventionally, processing of these ceramics has been difficult and very expensive due to their intrinsic brittle nature. Near net shape forming of these ceramics using vacuum plasma spray technique offers a cost effective method to fabricate structural components. In the present investigation, it has been demonstrated that HfB2, HfC and HfN can be spray formed to near net shapes. Microstructural characterization for density and grain size distribution has been performed using optical and scannin g electron microscope (SEM). Phase identification and chemical characterization have been carried out using x-ray diffraction (XRD) and energy dispersive spectroscopy (EDS).

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4:05 PM

Environmental Barrier Coatings For Ceramic Matrix Composites: Evaluation After Exposure To Simulated Combustor Environments: *Karren L. More*[1]; Peter F. Tortorelli[1]; Larry R. Walker[1]; James R. Keiser[1]; Harry E. Eaton[2]; Gary D. Linsey[2]; Joshua B. Kimmel[3]; Jeffrey R. Price[3]; [1]Oak Ridge National Laboratory, Met. and Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6064 USA; [2]United Technologies Research Center, East Hartford, CT; [3]Solar Turbines, Inc., San Diego, CA USA

Recent gas turbine engine tests utilizing SiC/SiC composite combustor liners as well as laboratory exposures of similar materials to elevated water-vapor pressures and temperatures have demonstrated rapid oxidation/degradation of these materials under typical engine operating conditions. In order to achieve the lifetime goals for SiC-based composite materials in engine applications such as combustor liners, protective coatings for the composite materials will be required. Oxide-based environmental barrier coatings (EBCs) have been developed specifically for these SiC/SiC composite materials. The EBCs have been exposed for >6000 h in the laboratory under conditions chosen to best simulate the key degradation mode in combustion environments and are also currently in an engine test which has been running for >10,000 h. This work will focus on the microstructural evaluation of several EBC and composite systems and their stabilities after exposures in both laboratory facilities and gas-turbine engines. Research sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Power Technologies, Continuous Fiber Reinforced Ceramic Composite Program, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

4:25 PM

Oxidation-Resistant CVD Mullite Coatings with Controlled Composition and Microstructure: *Svetlana M Zemskova*[1]; J. Allen Haynes[1]; Kevin M. Cooley[1]; [1]Oak Ridge National Laboratory, Met. & Ceram. Div., P.O. Box 2008, MS 6063, Oak Ridge, TN 37831-6063 USA

Application of silicon-based ceramics as high-temperature structural materials requires the development of environmentally-resistant coatings due to rapid degradation in high temperature water vapor. The CVD method of fabricating dense, high-purity, crystalline mullite (3Al2O3x2SiO2)coatings on silicon nitride was utilized in this study. The textured coatings were generally comprised of crystalline grains (0.3-0.5μm) with a columnar structure. Coatings with thickness of less than 2 μm, provided excellent corrosion protection for silicon nitride exposed to low-velocity, high pressure (10 atm) steam at 1200ºC.

4:45 PM

The Spectral Emittance and Stability of Coatings and Textured Surfaces for Thermophotovoltaic (TPV) Radiator Applications: *Brian Vern Cockeram*[1]; Dorothy P. Measures[1]; Jim L. Hollenbeck[1]; [1]Bechtel Bettis, ZAP 08D/MT, P.O. Box 79, West Mifflin, PA 15122 USA

Coatings and surface modifications are needed to improve the surface emissivity of materials under consideration for TPV radiator applications. The wavelengths of photons emitted from the surface of the TPV radiator should ideally match the range of wavelengths that correspond to the bandgap of the TPV cells for optimum efficiency. Vacuum plasma spray coatings (ZrO2+18% TiO2+10% Y2O3, ZrC, Fe2TiO5, ZrTiO4, ZrO2+8% Y2O3+2% HfO2, and Al2O3+TiO2) and a chemical vapor deposited coating of rhenium whiskers have been shown to be thermally stable and have produced a desired increase in the surface emissivity of refractory metals and nickel-base materials [1]. These coatings are further evaluated by measuring the spectral emissivity before and after long-term vacuum anneals. The microstructure of the coatings is evaluated to characterize the thermal stability of the coatings after long-term vacuum annealing. 1B.V. Cockeram, D.P. Measures, and A.J. Mueller, Thin Solid Films, 355/356 (1999), pp. 17-25.

5:05 PM

Friction and Wear Propertiesof CVD-Coated Diamond at Room Temperature: *Ali Soleman Al-Watban*[1]; [1]Riyadh Technical College, P.O. Box 53699, Riyadh 11593 Saudi Arabia

In this paper, the room temperature friction and deformation properties of a CVD coated polycrystalline diamond were studied when softer metallic and ceramic sliders were used. The experiments were conducted at relatively low sliding speeds (10 mm/min), in a vacuum of 0.0001 mbr after having cleaned the specimen surfaces by out-gassing them for about 30 min at 800ºC. As a result of increasing the number of traversals significant wear of the coated diamond by softer metallic sliders (aluminum and mild steel) was observed. With ceramic sliders, it is shown that the multiple traversals result in the formation of wear groove. For reasons of comparison, the same technique has been applied to type I single crystal diamond.

5:25 PM

Laser Processing of Ultrahard Coatings: *Ashok Kumar*[1]; [1]University of South Florida, Dept. of Mech. Eng., Center for Microelec. Res., Tampa, FL 33620 USA

The development of superhard coatings with high level of hardness, wear resistance and toughness is an important area of research with numerous applications. Our research is focused on the development of carbide and nitride coatings by the Pulsed Laser Deposition (PLD) method. The PLD is a vapor deposition process that combine near atomic-level control of the surface composition with the high kinetic energies of the condensing vapor produced by the laser ablation. Thin film coatings of carbides (titanium carbide, silicon carbide and boron carbide) and nitrides (titanium nitride, silicon nitride, and aluminum nitride and carbon nitride) were deposited on Si (100) substrates using PLD method. The structural and microstructural properties of these films have been characterized using x-ray diffraction, scanning and transmission electron microscope techniques. The mechanical properties of the films were evaluated to measure the hardness and modulus values. Microlaminate made of alternate layers of TiN and TiB2 films is likely to offer promises of exceptionally high hardness and modulii and, therefore, can be employed for future protective coatings. Single layer of TiN, TiB2 and TiB2/TiN microlaminate coatings with varying thickness were initially deposited on Si(100) and oxidized Si(111) substrates by pulsed laser deposition techniques and then characterized by x-ray diffraction, transmission electron microscopy and nano-indentation methods. Analysis of the resulting data revealed that the elastic modulus and hardness of multilayer coatings are superior to monolithic coatings of either of the two constituent films. It is suggested that the smooth nature of the interface between TiN and TiB2 is responsible for the improved hardness. This work was supported by NASA EPSCoR program.

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Effect of Enamel Coating on The Corrosion of Titanium Alloys: *Yuming Xiong*[1]; *Fuhui Wang*[1]; *Weitao Wu*[1]; *Yan Niu*[1]; [1]Institute of Corrosion and Protection of Metals, State Key Lab. for Corr. and Prot., Wencui Rd. 62, Shenyang, Liao Ning 110016 China

The air oxidation and corrosion induced by the synergistic effect of solid NaCl deposits and water vapor of the titanium alloys with an enamel coating have been investigated. The results revealed that the enamel coating markedly improved the oxidation resistance of the alloy at 600-800ºC in air, since the coating possesses a good thermal chemical stability and a thermal expansion coefficient matching well with titanium alloys. The coating showed good corrosion resistance to solid NaCl deposits in mixtures of oxygen with water vapor at 600ºC. The enamel coating can also improve long-term oxidation of the alloy in air at 600ºC, and acts as the barrier for suppressing the migration of oxygen into the substrate, thus prevents the alloys from oxygen embrittlement.

International Symposium on Deformation and Microstructure in Intermetallics: Microstructure II

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Physical Metallurgy Committee, Jt. Mechanical Behavior of Materials

Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Peter M. Hazzledine, UES Inc., Dayton, OH 45432 USA

Wednesday PM — Room: 220
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Robert A. Varin, University of Waterloo, Canada; Sung H. Whang, Polytechnic University, Dept. of Mech. Eng., Brooklyn, NY 11201 USA

2:00 PM Invited

Complex Faults in Boron-Containing B2 Iron Aluminides: *Sharvan Kumar*[1]; Lixin Pang[1]; [1]Brown University, Div. of Eng., Box D, Providence, RI 02912 USA

Complex faults have been observed on the {001} planes in an Fe-40Al-0.7C-0.5B alloy in the as-extruded condition. Six variants of such faults can occur; these faults are characterized by a missing aluminum plane and an in-plane shift of 1/2<001>. Boron is claimed to segregate at such faults. These faults exhibit APB and stacking fault contrast when examined using a superlattice and fundamental reflections respectively. A variety of heat treatments were conducted to understand the origin and evolution of these faults as well as conditions under which they occur. These results will be presented and possible formation mechanisms will be discussed.

2:30 PM

Relating Dislocation Core Observations with Yielding Behavior in Ni_3Ge-Fe_3Ge $L1_2$ Alloys: *Kevin J. Hemker*[1]; T. John Balk[1]; [1]Johns Hopkins University, Mats. Sci. and Eng. and Mech. Eng., 200 Latrobe Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

Ni_3Ge-Fe_3Ge has been found to be a model system for relating the macroscopic mechanical behavior of $L1_2$ intermetallic alloys with atomic level dislocation core structures. Increases in iron content cause a gradual transition from anomalous to normal behavior, and remarkable low temperature strengthening. Transmission electron microscopy (TEM) and image simulations have been combined to determine the operative deformation mechanisms and to perform detailed measurements of superdislocation dissociations. The transition in yield strength behavior has been correlated with a transition from octahedral glide and cross-slip locking to cube glide, while low temperature strengthening coincides with enhanced cross-slip. The propensity for cross-slip has been related to a significant drop in cube plane antiphase boundary energy. It has also been noted that low temperature strengthening and the transition to cube glide are both consistent with an increase in complex stacking fault energy.

3:00 PM

Dipolar and Multipolar Defects in Al-Rich Gamma-TiAl and the Motion of Ordinary Dislocations: *Patrick Veyssiere*[1]; Fabienne Gregori[2]; [1]LEM, Cnrs-onera, Bp 72, Chatillon Cedex 92322 France; [2]LPMTM, Institut Galilee, 99 Av. J. B. Clement, Villetaneuse 93430 France

The generation and the organisation of single and multipolar prismatic loops is studied in Al-rich γ-TiAl single crystal strained to 2% at room temperature. In the near-[153] load orientation slip takes place on one single slip plane and mostly by ordinary dislocations. It is shown that the partial annihilation of mixed dipoles by local cross-slip contributes to the population of prismatic loops on which mobile dislocations are pinned. These are in turn annihilated by impacting mobile dislocations forming new loops. As the pinning/annihilation process repeats itself, prismatic loops gradually aggregate in certain locations of the sample forming loose walls. On the other hand, elastic interactions between ordinary dislocations and the longest prismatic loops may give rise to multipoles comprising a limited number of branches the extremities of which are aligned

with the screw direction. Elongated prismatic loops and multipoles may interact with impacting dislocations forming trailing-like configurations on both mixed and screw segments. This process is at the origin of a significant fraction of the dipole-decorated ordinary dislocations that constitute one of the essential characteristics of the deformation microstructure of γ-TiAl deformed in the low temperature domain of the flow stress anomaly. These processes are at variance from trailing mechanisms considered so far in order to explain dipole-decorated dislocations and to design models of the flow stress anomaly.

3:20 PM

Slip and Twin Interactions with Polytwin Interfaces in L10-ordered Intermetallics: *Jörg M.K. Wiezorek*[1]; William A. Soffa[1]; [1]University of Pittsburgh, Dept. Mat. Sci. & Eng, 848 Benedum Hall, Pittsburgh, PA 15261 USA

Polytwinned (PT), lamellar microstructures readily form via solid-state transformations in alloys based on intermetallic phases with the ordered tetragonal L1o-structure, e.g. TiAl and FePd. The PT-composition planes are common {111} in TiAl and common {101} in FePd. The mechanical properties of PT-grains are anisotropic with "hard" and "soft" orientations. For "hard" oriented grains shear transfer across PT-interfaces must occur and appears to be of particular importance to the development of quantitative descriptions of the behavior of this type of compounds. The deformation modes active in these L1o-phases involve slip of super- and ordinary dislocations, as well as typically significant amounts of ordered twinning. Methods of scanning and transmission electron microscopy have been used to investigate details of these transfer processes. Using crystallographic analyses based on the Thompson-tetrahedron adapted to the L1o-structure possible mechanisms for shear transfer across the distinct configurations at {111}- and {101}-conjugated PT-interfaces have been identified. These theoretically derived transfer mechanisms are compared to experimental observations in TiAl and FePd. Results of this study are related to the physical and mechanical behavior of PT-L1o-intermetallics.

3:40 PM

Twinning Processes in Creep-Deformed Lamellar TiAl: *Luke L. Hsiung*[1]; T. G. Nieh[1]; [1]Lawrence Livermore National Laboratory, Chem. and Mats. Sci., L-369, P.O. Box 808, Livermore, CA 94511 USA

Deformation twinning in creep-deformed lamellar TiAl has been found to be intimately related to the motion, pile-up and dissociation of interfacial (Shockley partial) dislocations. Since the interfacial (Shockley partial) dislocations are energetically less favorable to undergo cross-slip or climb, under normal conditions they can only move conservatively along interfaces. Consequently, the pile-up configuration once generated cannot be easily dissipated and thus remain in place even at elevated temperatures. The dislocation pile-up eventually leads to the emission of deformation twins from the interfaces into γ lamellae. In addition to the {111}<112]-type twinning, {112}<111]-type twinning has also been identified. The critical shear stresses for different twinning processes are evaluated based upon the pile-up and core dissociations of interfacial dislocations. This work was performed under the auspices of the U.S. Department of Energy through contract #W-7405-Eng-48 with Lawrence Livermore National Laboratory.

4:00 PM

Deformation Structure during Creep Deformation in Soft Orientation PST Crystals: *Hee Young Kim*[1]; Kouichi Maruyama[1]; [1]Tohoku University, Grad. Sch. of Eng., Dept. of Mat. Sci., Aobayama 02, Sendai 980-8579 Japan

The creep deformation behavior and microstructural evolution of polysynthetically twinned (PST) crystals Ti-48Al were investigated. The soft orientation with the lamellar plates oriented 35° to compression axis were deformed at 1150K under the applied stress of 100-400 MPa. Deformations took place in the maximum shear stress direction in the soft orientation. The dislocation structures in γ domains of six different orientation variants were examined by transmission electron microscopy, and operative slip and twinning systems were analysed. The macroscopic plastic strain and strain compatibility at domain and lamellar boundaries were discussed with associated the slip systems in each domain. The refinement of lamellae occurred by mechanical twinning during creep deformation. The effect of applied stress and strain on activity of twining was investigated quantitatively during creep deformation.

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Fracture Initiation in Gamma-TiAl Related to Crystallographic and Microstructural Features Studied in 4-point Bending: Benjamin Andrew Simkin[1]; Martin A. Crimp[1]; Thomas R. Bieler[1]; [1]Michigan State University, Dept. of Matls. Sci. and Mech., 3536 Engineering, East Lansing, MI 48824-1226 USA

Sub-critical surface fractures in 4-point bend samples of a duplex gamma-TiAl based alloy (Ti-48%Al-2%Nb-2%Cr) are characterized according to their relation to microstructural and crystallographic features of the material. Some fractures appear to initiate in the vicinity of Ti3Al particles due to apparent stress concentrations. Other fracture initiation sites occur at grain boundaries, and often are associated with the impingement of deformation twins. These initiation sites are characterized via electron channeling contrast imaging (ECCI) and electron backscatter patterns (EBSP). Using these methods, the local grain orientation, crack orientation relative to the principal stress axies, and the deformation defects in the vicinity of the crack are determined. An attempt is made to correlate macroscopic texture to the eventual plastic strain to failure.

International Symposium on Shape Casting of Aluminum: Science and Technology: Microstructural Evolution During Solidification and Heat Treatment

Sponsored by: Light Metals Division, Materials Processing and Manufacturing Division, Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Aluminum Committee, Non-Ferrous Metals Committee, Solidification Committee, Jt. Mechanical Behavior of Materials

Program Organizers: John E. Allison, Ford Motor Company, Scientific Research Laboratory, Dearborn, MI 48124-2053 USA; Dan Bryant, Chester, VA 23836-3122 USA; Jon Dantzig, University of Illinois, Department of Mechanical & Industrial Engineering, Urbana, IL 61801-2906 USA; Ray D. Peterson, IMCO Recycling, Inc., Rockwood, TN 37854 USA

Wednesday PM — Room: 224
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Murat Tiryakioglu, Western Kentucky University, Dept. of Manuf. Sci., Bowling Green, KY 42101-3576 USA; Arne K. Dahle, The University of Queensland, Dept. of Min., Brisbane, Australia

2:00 PM Keynote

Formation of Shear Defects in Pressurised Casting Processes: *D. H. StJohn*[1]; A. K. Dahle[1]; [1]University of Queensland, CRC for Cast. Mets. Manuf. (CAST), Dept. of Min., Mins. and Mats. Eng., Queensland, Australia

Shear defects are formed in a casting when partially solidified material has to flow over extended distances. They are therefore often apparent in casting processes such as semi-solid casting and in those where pressure is being applied (e.g. in pressurised casting processes such as squeeze and high-pressure die-casting). On complete solidification the shear defects often take the form of bands of segregation, porosity and tears or a combination of these defects. The properties of the casting are thus adversely affected. This paper outlines the cause of shear defects in terms of the development of mechanical strength during solidification. It is shown that the development of shear strength during solidification can be sub-divided into four regimes that have distinct rheological properties. Identification of these regimes provides a basis for development of a simulation program for predicting the location and severity of shear defects. Strategies to control the formation of these defects

can be suggested based on the new concept of rationalising the origin and causes of these defects.

2:45 PM Invited

Understanding Eutectic Formation in Hypoeutectic Al-Si Alloys: *Arne K. Dahle*[1]; Kazuhiro Nogita[1]; Stuart D. McDonald[1]; Jacob W. Zindel[2]; [1]The University of Queensland, Dept. of Min., Mins. and Mats. Eng., Brisbane, Qld 4072 Australia; [2]Ford Motor Company, Ford Res. Lab., Mats. Sci. Dept., Dearborn, MI 48121 USA

Commercial hypoeutectic Al-Si foundry alloys contain between 50 and 90 vol% eutectic. Solidification of the eutectic is the last major solidification event and therefore the critical stage where casting defects, such as porosity, form in these alloys. Thermal analysis, microstructural inspection, quenching, and electron back-scattering diffraction (EBSD) mapping have been used to study the effects of a range of different modifying elements and levels on eutectic nucleation and growth. The results show that eutectic solidification can occur by three distinctly different nucleation and growth modes, in isolation or sometimes together, but controlled by the modifier element used and the concentration added. Furthermore, it has been found that eutectic nucleation, and the resulting macroscopic growth mode, is independent of the plate-like to fibrous transition in silicon morphology upon modification. The eutectic nucleation mechanism and resulting macroscopic growth pattern dramatically affect the mushy zone permeability and pressure drop and therefore controls both porosity content and distribution.

3:15 PM

Evolution of the Eutectic Microstructure during Solidification of Hypoeutectic Al-Si Alloys: *Hema Vardhan Guthy*[1]; Makhlouf Makhlouf[1]; [1]Metals Processing Institute-Worcester Polytechnic Institute, 100 Institute Rd., Worcester, MA 01609 USA

Commonly used Al-Si casting alloys are typically hypoeutectic with silicon ranging from 7 to 11wt%. Although these alloys generally possess a relatively short freezing range, good castability and desirable overall properties, their mechanical properties are influenced by porosity, shrinkage and segregation, which in turn are determined by the last stage of solidification, namely eutectic formation. In this investigation, the evolution of the eutectic microstructure in hypoeutectic Al-Si alloys was studied using thermal analysis and metallographic techniques. Quench experiments of various Al-Si alloys of commercial purity and of high purity were performed. The resulting microstructure was characterized using optical and scanning electron microscopy. In unmodified alloys, both the eutectic silicon and the eutectic aluminum originate from the primary aluminum. Eutectic aluminum grows continuously out of the primary aluminum without any re-nucleation while the eutectic silicon nucleates on the primary aluminum. On the other hand, in Sr-modified alloys, the eutectic silicon nucleates on the primary aluminum while the eutectic aluminum nucleates on the eutectic silicon. Moreover, equiaxed eutectic grains were observed in Sr modified alloys with impurity elements segregated along the grain boundaries but no specific morphology was observed in eutectic grains of unmodified alloys.

3:45 PM Break

4:00 PM Invited

Modelling of Microsegregation in Shape Cast Aluminum Alloys: *N. Saunders*[1]; B. Boutwell[2]; [1]Thermotech, Ltd., Surrey Technology Centre, The Surrey Research Park, Guilford, Surrey GU2 7YG UK; [2]AEAT Technology, Inc., 241 Curry Hollow Rd., Pittsburgh, PA 15236-4696 USA

In recent years, thermodynamic modelling has reached the stage where phase equilibria can be modelled with a high degree of accuracy for many of the major types of Al-alloys. Such modelling is also readily extendable to predicting non-equilibrium solidification behaviour under so-called "Scheil-Gulliver" conditions and highly accurate predictions for fraction solid, heat of solidification and the phases formed during solidification can be obtained. It is now of interest to see what effect a more explicit treatment of diffusion in the solid state would make and, to this end, the software package DICTRA will be used to model the solidification of an Al-Si-Cu based alloy. A potential advantage of using DICTRA is that annealing schedules after solidification can be taken into account and the effect of subsequent heat treatment on the reduction in micro-segregation of the cast product can be modelled. The presentation will (i) provide a review of previous solidification modelling and (ii) present results from a DICTRA simulation applied to an Al-Si-Cu based casting alloy.

4:30 PM

The Effect of Ti Additions on the Grain Size and Ageing Properties of Aluminium Alloy A356: *Mark Alan Easton*[1]; Joseph Barresi[2]; David H. StJohn[3]; [1]Monash University, Dept. of Mats. Eng., Wellington Rd., P.O. Box 69M, Clayton, Victoria 3800 Australia; [2]Comalco Research and Technical Services, Edgars Rd., Thomastown, Victoria 3074 Australia; [3]University of Queensland, Dept. of Min., Mins. and Mats. Eng., Brisbane, Queensland 4072 Australia

Al-Si foundry alloys usually contain up to 0.2wt% titanium, added for grain refinement. It was found, using results published in the literature and from experiments, that additions of titanium up to 0.2wt% cause only a minor or negligible decrease in grain size in an A356 alloy for a wide variety of casting conditions. Boron additions in the form of stoichiometric TiB2 master alloys, are much more effective at reducing the grain size and therefore should be considered as the preferred grain refining addition. As well as grain size it is important to consider whether Ti in solution plays a role in the age hardening response of Al-Si alloys and these results are presented. Considerations for determining the optimum titanium concentration are discussed.

5:00 PM

Modeling Thermal Growth During Heat Treatment in Aluminum Castings: *Chris Wolverton*[1]; John Allison[1]; [1]Ford Motor Company, MD3028/SRL, P.O. Box 2053, Dearborn, MI 48176 USA

Macroscopic, irreversible dimensional changes are notorious for occuring during heat treatment of aluminum alloys. These dimensional changes (often called "thermal growth" since they typically involve an expansion of the material rather than a contraction) can deleteriously affect the performance of a given alloy. We have investigated the thermal growth in cast 319 Al, determining that the precipitate phase transformation to the Al2Cu (Θ') phase is responsible for the growth. In conjunction with experimental data, we have used a variety of computational tools (first-principles quantum-mechanical calculations, computational thermodynamics methods, and microstructural evolution models) to construct a model of thermal growth. The model is capable of quantitative prediction of the temperature-, time-, and composition-dependence of thermal growth in 319, and accounts for multiple Cu-containing precipitate phases. Although we have constructed the model for 319 Al, this type of model should be generally applicable for thermal growth in other alloys as well.

Lead-Free Solder Materials and Soldering Technologies VI: Reliability, Electromigration, Applications

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Sung Kang, IBM, TJ Watson Research Center, Yorktown Heights, NY 10598 USA; Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Hareesh Mavoori, Lucent Technologies, Bell Laboratories, Murray Hill, NJ 07974 USA; Ronald W. Smith, Materials Resources International, North Wales, PA 19454 USA

Wednesday PM Room: 227
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Srinivas Chada, Motorola, APTC Dept., Plantation, FL 33322 USA; Ray Fournelle, Marquette University, Mats, Sci, & Eng., Milwaukee, WI USA

2:00 PM Invited

Pb-free Solders for Power Die Attach: *J.N. Lalena*[1]; Martin W. Weiser[1]; Nancy F. Dean[1]; [1]Honeywell Electronic Materials, 15128 E. Euclid Ave., Spokane, WA 99216

Power semiconductor devices such as IGBTs, MOSFETS, Bipolar Transistors, Rectifiers, and Thyristors are normally fabricated by soldering a Si die to a Cu leadframe that serves as a heat spreader. These die are often quite large (0.8 mm square is common) and have high heat output (>100 W is not uncommon). The solders used for these devices must have both good thermal conductivity to dissipate the heat and very good resistance to thermomechanical fatigue due to the large CTE difference between Si and Cu. Several high-Pb and a couple of high-Sn solder alloys have dominated this application for many years. The electronics industry is in the process of adopting Pb-free solders for board level assembly. Most of the proposed solders are based upon the Sn-Ag, Sn-Cu, and Sn-Ag-Cu eutectics which melt between 210 and 230ºC. As a result these solders are reflowed at 235 to 270ºC depending upon the solder, process, and thermal mass of the system that is being assembled. Of the current high volume die attach solder alloys only the high-Pb solder alloys will survive these reflow temperatures-too much liquid phase forms in the high-Sn alloys. We have looked at a variety of systems to find Pb-free alloys that meet the stringent requirements of a die attach solder for power semiconductor devices. We will discuss the requirements in detail and how some of the most promising systems stack up against these requirements. Some alloys such as Zn4Al3Mg3Ga discussed in a previous year by Shimuzu et. al. have promising melting and wetting behavior, but have inadequate mechanical properties.

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Board Level Reliability of Pb-free and Pb-bearing Polymer-Core Solder Balls for CSP Application: *Seung Wook Yoon*[1]; Jong Heon Kim[1]; Shin Choi[1]; Ik Seong Park[1]; Heung Sup Chun[1]; [1]Hyundai Electronics Industries Company, Ltd, Semiconductor Group, Ichon, Kyunggi-do 467-701 Korea

Chip scale packages (CSP) have essential solder joint quality problems, and a board level reliability is a key issue in design and development of the CSP type packages. And many countries such as Japan and Europe, have proposed laws reducing or eliminating the use of Pb and other toxic substance in an effort to decrease landfill pollution and ground water contamination. This paper is focused on the results of investigations on polymer-core solder ball with Pb-bearing as well as Pb-free solder for CSP applications. The solder-joint reliability of a LFCSP(Lead-Frame CSP) and WLCSP(Wafer Level CSP) on printed circuit board (PCB) under thermal fatigue is studied. The solder joints are subjected to thermal cycling and their lifetime, crack initiation site, crack propagation mode are observed. Their solder joint life times were compared with conventional Sn-Pb eutectic solder and microstructures were also examined by OM, SEM and EDX. In order to find out the role of polymer core in solder joint, the stress/strain intensity factors at the solder joint region are investigated by fracture mechanics with FEM (finite element method)computer simulation.

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Ball Shear Strength and Microstructure of Eutectic Tin Lead and Lead Free Solder Ball After Reliability Test: *P. J. Zheng*[1]; S. C. Lee[1]; J. Z. Lee[1]; J. G. Hwang[1]; Jim C. L. Wu[1]; [1]Advanced Semiconductor Engineering, Inc., 26, Chin 3rd Rd., Nantz Export Processing Zone, Kaohsiung City, Taiwan

Currently, the alloy materials used as BGA solder ball are mostly eutectic tin lead materials such as 63wt%Sn/37wt%Pb and 62wt%Sn/36wt%Pb/2wt%Ag. Due to environmental pollution concern, e.g., landfill pollution and ground water contamination, many countries have legislated to reduce or eliminate the use of lead. This comes out the massive need of lead free alloy to be used in electronic components. However, because the mechanic property of lead free alloy is quite different from eutectic tin lead, the process parameters used in PBGA assembly flow need to be rebuilt. In addition, the reliability of the corresponding PBGA products also needs to be evaluated. Since the cost deviation between lead free and eutectic solder ball is still a key concern, how to select a suitable and cost-effective lead free material becomes an important issue. In the study, the shear strength and microstructure of lead free solder ball versus eutectic solder ball after reliability test is investigated. Two kinds of eutectic tin lead materials 63wt%Sn/37wt%Pb and 62wt%Sn/36wt%Pb/2wt%Ag and two kinds of lead free alloys 96.5wt%Sn/3.5wt%Ag and 95.5wt%Sn/4.0wt%Ag/0.5wt%Cu are selected to be solder balls in 618 PBGA (with 0.76 mm ball diameter). After the assembly, the samples are first preconditioned under JEDEC level 3 standard. Temperature cyclic test (TCT), thermal shock test (TST), temperature and humidity test (THT), pressure cook test (PCT) and high temperature storage test (HTST) are followed after the precondition test. The ball shear strengths of solder balls are respectively measured right after assembly, pre-conditioned and the end of each reliability test. Our preliminary result shows that 96.5wt%Sn/3.5wt%Ag possess highest shear strength. For example, at the end of 1000 cycles of TC test, the shear strength is 1500 g for 96.5wt%Sn/3.5wt%Ag, which is 1.5 times higher than other three materials. Optical microscope and SEM are used to observe the failure mode of shear test after each reliability test. SEM and EDX are applied to analyze the different intermetallic compounds (IMC) that formed at solder joint of the four solder ball alloys. The analysis results will be presented and discussed in the final paper.

3:00 PM

Chip-on-glass(COG) Mounting Using a Laser Beam Transmitting Glass Substrates: *Jong-Hyun Lee*[1]; Won-Yong Kim[2]; Dong-Hoon Ahn[2]; Yong-Seog Kim[1]; [1]Hong Ik University, Mats. Sci. and Eng., Mapo-Cu Sangsu-Dong 72-1, Seoul 121-791 Korea; [2]Yeonwoo Engineeing Company, Ltd., Choong-Ri #425, Koosung-Myun, Yongin-Si, Kyungki-Do 449-910 Korea

For Chip-on-glass(GOG) electronic packaging in liquid crystal display, anisotropic conductive film(ACF) is being used extensively. The reliability and reworkability of the joints need to be improved further for extended applications of the COG mounting method. In this study, COG mounting process was attempted by local heating of solder bumps using a laser beam transmitted through glass substrate. The laser beam from diode transmitted the glass was absorbed by a metal pad and consequently heated Sn-Ag-Cu solder or solder paste in contact with the pad. The pad materials consisted of adhesion layer (i.e. Cr or Ti) and solderable layer (i.e. Ni or Cu). The interfacial microstructure and mechanical properties of the joint were investigated at different energy input rates, radiation times and pad materials.

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Electroless Ni/Immersion Au Finish: Impact on Interconnection: *Polina Snugovsky*[1]; Peter Arrowsmith[1]; Marianne Romansky[1]; [1]Celestica, Proc. Eng. Dev., 844 Don Mills Rd., 33/178, Toronto,

Ontario M3C 1V7 Canada; [1]Celestica, Inc., Techn. Assurance Labs., 844 Don Mills Rd., 20/149, Toronto, Ontatio M3C 1V7 Canada

Electroless Nickel/Immersion Gold finish is widely used in the electronic industry. Weak lifted joints, which exhibit a dark nickel-like surface on the conductive pads were detected and named "black pad". Many reports in the literature in recent years described cases of poor solder joints on electroless Ni/immersion Au. Celestica also face the black pad issue, and accumulated some knowledge in recognizing and reworking assemblies showing this phenomena. The black pad case reported here has some unique features. The investigation and finding of black pad in SMT processes is described. The presentation and the manuscript will include: Black pad occurrence and failure mechanism in wire bonding; Effect of board construction processes on black pad formation; Thick Au and thin Ni appearance on defective pads; Au and Ni layer composition; Mechanism of defective electroless Ni/immersion Au deposition; Weak solder joint on thick Au-black pad; Comparison of intermetallic formation during SMT on good and bad pads: microstructure, kinetics; SMT parameters influence on solder joint formation on bad pads; Rework reliability

3:40 PM Break

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Electromigration in SnPb Solder Alloys: *Cheng-Yi Liu*[1]; Q. T. Huynh[2]; King-Ning Tu[2]; [1]Intel, Component Research, 5000 W. Chandler Blvd., Mail Stop Ch5-166, Chandler, AZ 85226 USA; [2]UCLA, Dept. of Mats. Sci. & Eng., Los Angeles, CA USA

Both thin film and thick film of SnPb solder samples were prepared for electromigration. The thin film samples were of 1 mm in thickness with Cu films as electrodes. They were tested at current density of 1 x 105 amp/cm2 and at room temperature, resulting in the formation of a large number of hillocks of Sn at the anode and voids at the cathode. The dominant interfacial diffusing species was found to be Sn. The thick film samples were SnPb solder lines reflowed in V-grooves on (001) Si wafer surfaces. The width and depth of the V-groove were 100 mm and 69 mm, respectively. The electrodes were Cu wires inserted in the two ends of the V-groove before solder reflow. These thick solder lines were also tested at current density of 1 x 105 amp/cm2 but at 150°C, resulting in the formation of a large lump of accumulation of both Pb and Sn at the anode and a large void at the cathode. The dominant bulk diffusing species was observed to be Pb. We shall discuss the different electromigration behaviors in these two kinds of samples. Besides those changes occurring at the anode and cathode, there was a substantial amount of microstructure evolution in the films and lines, owing to grain growth and phase separation. Also we will present the electromigration behavior as a function of SnPb alloy composition, as in the 95Pb5Sn and pure Sn samples.

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Whisker Formation Study of Lead-free Sn-Cu Plated Packages by Pressure Cook Test: *Jim C.L. Wu*[1]; Jeffrey C. B. Lee[1]; Y. S. Chou[1]; C. H. Chang[1]; [1]Advanced Semiconductor Engineering, Inc, 26, Chin 3rd Rd., Nantz Export Processing Zone, Kaohsiung City, Taiwan

Tin-lead (85%/15%) plating system has been widely used in the package production line for more than ten years. It offers the best in classes from the production efficiency and cost. Due to the environmental and human being health concerns, some of the area, for example, Europe and Japan, has legislated that lead is going to be reduced or totally eliminated before next decade. Therefore, to develop another plating technology and system toward lead-free package is of great interested not only from novel new technology availability but also from market share business-wised consideration. There are several popular alloy system candidates in the market, such as pure Sn, Sn-Ag, Sn-Bi and Sn-Cu. However, tin rich alloy has been reported that they have whisker growth latent risk in the lead finishing. Therefore, how to develop a time effective acceleration test and setup a reject criteria to screen the material, process and reliability evaluation are of significant importance. In this report, pressure cook test(PCT) was applied to study the whisker formation of Sn-Cu lead finishing during the reliability test. The test vehicles include PLCC-44L, QFP-208L and TSOP-48L. The impact of percentage of copper on lead formation and reliability tests is also investigated. The influences with various leadframe materials, chemicals, stress/forming and copper contents on the microstructure were also scrutinized by SEM, EDX, and X-ray. The preliminary results showed that the chemical and leadframe materials play important roles on whisker growth latent risk. Copper diffusion during the PCT test was also reported.

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Chemistry, Microstructure and Mechanical Properties of Sn-Bi/Cu Interface: *Jian-Ku Shang*[1]; Pi Lin Liu[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mats. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA

Bi-containing alloys are being explored as potential replacements to the Sn-Pb eutectic alloy in the solder interconnect. While most of the attention has been focused on the bulk solder alloys, the metallurgical compatibility of these alloys with the common metallizations such as copper and nickel has not been adequately investigated. In this study, the compatibility of the eutectic Sn-Bi alloy with copper metallization was investigated by examining the chemistry, microstructure, and mechanical properties of the Sn-Bi/Cu interface in the as-reflowed and aged conditions. While the mechanical properties of the Sn-Bi/Cu interface were slightly inferior to those of the eutectic Sn-Pb/Cu interface in the as-reflowed condition, drastic weakening of the Sn-Bi/Cu interface was found after the interface was artifically aged. Such a weakening effect was shown to result from the unique interfacial chemistry and microstructure developed by the aging treatment.

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The Characteristics of Vibration Fracture of Pb-Sn and Lead-Free Sn-Zn Eutectic Solders: *Chiang-Ming Chuang*[1]; *Truan-Sheng Lui*[1]; Li-Hui Chen[1]; [1]National Cheng Kung University, Mats. Sci. and Eng., Tainan, Taiwan 701

This work investigates the characteristics of vibration fracture of Sn-Zn lead-free solder and Pb-Sn solder. The frequency of all most fatigue studies about solders were below 1 Hz, in the past. However, when these electronic components include solder joints were assembled in the vehicles, the frequency of vibrant situation is very different. The experimental materials of rapid solidification were tested as tension and vibration during unstable state to stable state in room temperature. The experimental results show that the tensile strength of Pb-Sn solder and Sn-Zn lead-free solder after rapidly solidified decrease with aging time increasing, then tend to stable after a period days. The results of vibration test show that, in the same vibration force, Sn-Zn lead-free solder has smaller specimen's end deflection and higher crack propagation resistance. Oppositely, when the initial specimen's end deflection of two experimental materials was similar, the strain in the notch area was equal, the traditional Pb-Sn solder has batter crack propagation resistance.

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Electromigration Effect upon the Sn-0.7wt%Cu/Ni and Sn-3.5wt%Ag/Ni Interfacial Reactions: *Chih-ming Chen*[1]; Sinn-wen Chen[1]; Mei-yau Du[1]; [1]National Tsing-Hua University, Dept. of Chem. Eng., #101 Kuang-Fu Rd., Sec. 2, Hsin-Chu, Taiwan 300 Taiwan

Electromigration refers to the phenomenon that the passage of electric currents induces the movement of metallic atoms in metals. This study investigates the effect of electromigration upon the interfacial reactions between the promising lead-free solders, Sn-Cu and Sn-Ag, with Ni substrate. Sn-0.7wt%Cu/Ni and Sn-3.5wt%Ag/Ni couples reacted at various temperatures with and without the passage of electric currents were examined. Only one intermetallic compound Ni_3Sn_4 was found at the interfaces of all the couples reacted at 160°C, 180°C, and 200°C. The growth rates of the Ni_3Sn_4 phase were either enhanced or retarded by the passage of a 500 A/cm^2 electric current depending on its flow direction. The effectiveness of the passage of electric currents decreased with increasing temperatures.

Lightweight Alloys for Aerospace Applications: Deformation, Fatigue and Environmental Fracture-I

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee

Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Nack J. Kim, Center for Adv. Aerospace Materials, Pohang 790-330 Korea; Eui W. Lee, Naval Air Warfare Center, Code 4342, MS5, Patuxent River, MD 20670 USA; William Frazier, Naval Air Warfare Center, Aircraft Division, Patuxent River, MD 20670-1908 USA

Wednesday PM — Room: 213
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: O. S. Es-Said, Loyola Marymount University, Los Angeles, CA USA

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The Effect of Multi-Axiality on Creep/Fatigue Failure in Gamma Titanium Alloys at Elevated Temperatures: *Kamran Nikbin*[1]; [1]Imperial College, MED, Exhibition Rd., London SW13 9NA UK

The thermal efficiency of a gas turbine is only around 38 % with a gas entry temperature of 1200K, whilst at 1800K the efficiency rises to over 50 % [1], hence there is a strong demand for materials capable of operating at ever increasing temperatures. In response to this considerable research has and indeed is continuing to be performed on intermetallic materials such as g-TiAl. The low-pressure turbine section, where blade temperatures up to 750ºC and blade root temperatures of 500ºC are commonplace, is seen as a typical environment for this material. g-TiAl possesses some particularly attractive properties such as low density (approximately half that of nickel-based superalloys), and good creep resistance, however it presents the engineer with problems not associated with conventional materials. The paper will deal with and analyse the differences in behaviour, in creep and creep/fatigue under multi-axial stress state, of different batches of Gamma- Titanimum with similar composition and heat treatment.

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Dwell-Fatigue Behavior of Ti-6242 Alloy: *V. Sinha*[1]; M. Savage[1]; J. Tatalovich[1]; M. J. Mills[1]; J. C. Williams[1]; [1]The Ohio State University, Dept. of Matls. Sci. and Eng., 2041 College Rd., Columbus, OH 43210 USA

The current understanding of the dwell-fatigue behavior of a/b titanium alloys will be reviewed. The results of this review will be used to motivate a discussion of a recent research results obtained for a typical high temperature a/b titanium alloy, Ti-6242. This alloy is very extensively used for rotors in the compressor section as multi-stage spools of the aeroengines. A significant deficit in the fatigue life of this class of alloys has been reported in the literature under dwell-fatigue conditions at room temperature and high stress levels when compared with that under continuous cycling conditions. In this study, the effects of microstructure (i.e. lamellar/ Widmanstätten vs. globular) on the dwell-fatigue response of Ti-6242 are being investigated. Furthermore, the influence of microtexture (in the equiaxed a/b forged microstructure) on the dwell-fatigue life is also being examined. The globular microstructures of two kinds are investigated: (i) having randomly oriented a grains, and (ii) having maximum microtexture. The effects of hydrogen content are also examined in some detail. In the end, some possible crack initiation and propagation mechanisms are discussed and supported by scanning electron microscopy and orientation imaging microscopy results. (This work is being supported by The Federal Aviation Administration).

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Understanding Foreign Object Damage through Spatially Resolved Residual Stress Measurements: *B. L. Boyce*[1]; J. O. Peters[1]; J. M. McNaney[1]; R. O. Ritchie[1]; [1]University of California at Berkeley/LBNL, Mats. Sci. Div., MS 62-203, 1 Cyclotron Rd., Berkeley, CA 94720 USA

Foreign Object Damage, caused by the ingestion of debris into turbine engines, can significantly degrade the usable lifetime of structural components (most specifically fan blades). The current study uses synchrotron x-ray diffraction to examine the formation of residual stresses by the impacting process and the implication of these residual stresses on crack formation and propagation under cyclic loading. Moreover, the observed residual stress gradients will be used to asses the validity and limitations of numerical models.

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Deformation, Fracture and Fatigue in a High Strength Aluminum Alloy: *A. P. Reynolds*[1]; Kumar Jata[2]; [1]University of South Carolina, Dept. of Mech. Eng., 300 Main St., Columbia, SC 29208 USA; [2]Air Force Research Laboratory, AFRL/MLLM, 2230 Tenth St., WPAFB, OH 45433 USA

Tensile, fracture, and fatigue studies have been conducted on a dispersion strengthened aluminum alloy with sub-micron grains. Full-field, surface strain distributions were measured by digital image correlation (DIC) in selected cases. DIC was used to understand the strain distribution/partitioning in uniaxial tension and in the plastic zone at a crack tip.

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The Compressive Behavior of Ti-6Al-4V/TiC Layered Composites Examined Through Experiments and Modeling: C. L. Briant[1]; C. W. Bull[1]; K. S. Kumar[1]; *A. J. Wagoner Johnson*[1]; [1]Brown University, Div. of Eng., Box D, Providence, RI 02906 USA

The Ti-6Al-4V/TiC composite system is being studied in compression as a function of strain rate. Symmetric, three-layered structures have been successfully fabricated with equal layer thickness by diffusion bonding individual layers. The layers are made of either the monolithic material or a 10%TiC particulate reinforced composite. The layer-interfaces are void-free and the interfacial bond is excellent. Preliminary results show that the engineering stress of the layered structures is bounded by the engineering stress of the individual layers, despite the yield of only the monolithic layer. While the structures show similar hardening behavior as the individual layers, the strength and ductility are much greater than the individual monolithic and reinforced layers, respectively. The interface between layers constrains the softer material from flowing radially, causing the diameter of the cylindrical specimen to vary. Structure failure is postponed by crack deflection near the soft/hard layer interface. The contribution of the interface to the compressive strength of the structures will be modeled by altering the interfacial strength, or interface boundary conditions. Layer thickness and number of layers will also be varied to further improve properties.

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Influence of Heat Treatment on the High Rate Deformation Behavior of a Metal Matrix Composite: *Don Lesuer*[1]; Chol Syn[1]; Mary LeBlanc[1]; [1]Lawrence Livermore National Laboratory, L-342, Livermore, CA 94551 USA

Many potential applications of metal matrix composites involve use of the material under dynamic loads. In this paper we report on the influence of heat treatment on the high rate deformation of the 6090 aluminum alloy reinforced with 25% SiC particulate. Six heat treatments that changed the strength of the matrix alloy were considered a peak age temper, two underage tempers and three overage tempers. High rate testing was done in compression using the split Hopkinson pressure bar technique, and data was obtained at strain rates of 103 s-1 to 104 s-1. The underage and overage tempers produced lower flow stresses than the peak age treatment but all heat treatments produced comparably work hardening rates. The influence of the stress-strain behavior of the matrix on the stress-strain behavior of the composite has been analyzed in terms of

composite-strengthening, continuum-plasticity models. Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under contract No. W-7405-ENG-48

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Intergranular and Stress Corrosion Cracking Behavior of Al-Li-Cu Alloy AF/C458 After Artificial Aging: D. Mathur[1]; P. I. Gouma[1]; R. G. Buchheit[1]; [1]Ohio State University, Dept. of Mat. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

The stress corrosion cracking (SCC) behavior of AF/C458 (Al-2.05Li-2.70Cu-0.6Mg-0.3Zn-0.08Zr) in 1.8 inch plate form was studied as a function of artificial aging at either 150°C or 190°C for times ranging from 2.4 to 36 hours. SCC resistance of the alloy in the short-transverse and long-transverse orientations was evaluated by alternate immersion and constant extension rate testing (CERT) in 3.5% NaCl solutions. The alloy was also evaluated for susceptibility to a pre-exposure embrittlement phenomenon unique to Al-Li alloys. By all measures, the SCC behavior of this alloy is superior to AA 2090 and AA 8090. Resistance is likely derived from a variety of factors including controlled texture, grain size, high toughness, and controlled grain boundary precipitation. The experimental observations of SCC combined with a microstructural characterization by transmission electron microscopy supports the notion that when the alloy is SCC-susceptible, an anodic dissolution-based cracking mechanism involving selective dissolution of Zn-modified T_1 (Al_2(Cu,Zn)Li) precipitated at low and high angle boundaries is operative.

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Localized Corrosion Mechanisms and TEM Characterization of Al-Li-Cu Alloy AF/C458 After Interrupted Quenching from Solutionizing Temperatures: P. I. Gouma[1]; J. E. Kertz[1]; R. G. Buchheit[1]; [1]Ohio State University, Dept. of Mat. Sci. and Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Isothermal time-temperature-localized corrosion mechanism curves were determined for the Al-2.05Li-2.70Cu-0.6Mg-0.3Zn-0.08Zr alloy AF/C458 to understand the effect of slow or delayed quenching on localized corrosion mechanisms. Alloy samples were subject to a series of systematic interrupted quenching experiments conducted at temperatures ranging from 480° to 230°C for times ranging from 10 to 1000 seconds. Samples were then exposed to a oxidizing aqueous chloride environment to induce localized attack. The alloy exhibited pitting, intersubgranular attack (ISGA), or intergranular attack (IGA) depending on the time at temperature. Corrosion behavior was interpreted on the basis of a detailed microstructural evaluation by transmission electron microscopy, which clearly showed that IGA and ISGA were related to the precipitation of Zn-modified T_1 (Al_2(Cu,Zn)Li) at high and low angle boundaries resepctively. In situations where the T_B (Al_7Cu_4Li) phase was present on boundaries instead of T_1, IGA and ISGA susceptibility was comparatively diminished.

Magnesium Technology 2001: Forming

Sponsored by: TMS: Light Metals Division, Magnesium Committee and Reactive Metals Committee; International Magnesium Association; and ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee

Program Organizers: John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA; David Creber, Alcan International, Ltd., Kingston R&D Center, Kingston, Ontario K7L 5L9 Canada; Russell H. Jones, Battelle Pacific Northwest National Laboratory, Richland, WA 99352 USA; Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Ramaswami Neelameggham, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Eric A. Nyberg, Pacific Northwest National Laboratory, Materials Processing Group, Richland, WA 99352 USA; Mihriban O. Pekguleryz, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Kevin Watson, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H7R 1G5 Canada

Wednesday PM — Room: 203-205
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA

2:00 PM

Alloy Design and Microstructure Evolution of Thixoformable Mg-Ni Alloys: *Shaekwang Kim*[1]; Won Ha[1]; Chulhong Bae[1]; Youngjig Kim[1]; [1]Sung Kyun Kwan University, Sch. of Metall. and Mat. Eng., 300 Chunchun-dong Jangan-gu, Suwon, Gyunggi-do 440-746 Korea

The importance of processing of magnesium alloys in semisolid state is increasing rapidly. The understanding of the process is concerned primarily with the microstructure of semisolid alloys generated during either partial solidification or partial remelting, with their Rheological behaviors and with the modeling of this behavior for the purpose of numerical simulation of thixoforming processing. However, very little work has been done in the development of alloy composition tailored to thixoforming processing. The aim of this research is to develop thixoformable Mg-Ni alloys and to evaluate the microstructure evolution of them during partial remelting as functions of Ni content and isothermal holding temperatures. The characteristics of thixoformable Mg-Ni alloys are based on the fact that fraction and composition of liquid and fine solid globules without the network among solid globules can be simply controlled by normal solidification and reheating procedures.

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Microstructure Studies after Different Solution Treatments of a Thixo-Cast AZ91: *Enrico Evangelista*[1]; Marcello Cabibbo[1]; Emanuela Cerri[2]; [1]University of Ancona, Dept. of Mechanics, Via Brecce Bianche, Ancona I-60131 Italy; [2]University of Lecce, Dept. of "Ingegneria dell'Innovazione", Via Arnesano, Lecce I-73100 Italy

The microstructure of a thixoformed AZ91 Mg-Al-Zn consists of large alpha globules separated by quasi-eutectic phase (alpha+betha). Solution heat treatments at 395°C, 415°C and 435°C for different exposition times were carried out. Light Microscopy (LM) investigations showed that the alpha-Mg based areas are developed into individual grains, while the betha phase (Mg17Al12) particles are present only in the eutectic area. Transmission Electron Microscopy (TEM) revealed small Mg-rich particles inside the eutectic (divorced eutectic). The solution treatment induced the almost complete transformation of the original microstructure, produced by thixo-forming, in a more conventional structure of equiaxed grains respectively in 1hr, 3hrs, 24hrs for 435°C, 415°C and 395°C. From this time the eutectic is localised in the grain boundaries and the alpha-Mg based grains enriches in aluminium and zinc content

due, basically, to the simultaneous dissolution of the alpha-phase and reduction in eutectic quantity.

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Superplasticity in Coarse Grained Class I Solid Solution of HCP Mg-Al: *Tsutomu Ito*[1]; Junya Saeki[1]; Masahisa Otsuka[1]; [1]Shibaura Institute of Technology, Dept. of Mats. Sci. and Eng., 3-9-14 Shibaura, Minato-ku, Tokyo 1088548 Japan

Hexagonal close packed magnesium alloys have recently been expected as structural materials for transportation systems due to their excellent specific stiffness and specific strength. However, a limited number of their slip systems due to an anisotropy in hcp structure make them less ductile at room temperature than face centered cubic materials such as aluminum or copper alloys. On the other hand, in the case of elevated temperature, magnesium alloy should potentially exhibit high ductility because both pyramidal slip and prism one are activated in addition to basal glide. We have been investigated high temperature deformation behavior on three kinds of coarse-grained Mg-Al class I solid solution. The stress exponent was 3 and the activation energy for deformation was close to that for chemical interdiffusion. The characteristics are similar to those of class I solid solutions. The alloys showed enhanced ductility over 250% at 723K and initial strain rate around 1 x 10-4s-1. The result suggest that the alloys could have exhibited class I superplasticity. The mechanism of deformation and fracture will be discussed on the basis of microstructural observation.

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Production of a Superplastic Microstructure in Magnesium Alloys and their Application: *Claus Christian Kedenburg*[1]; Antonia Schram[1]; Ulrich Draugelates[1]; [1]Institute of Welding ISAF, Magnesium Dept., Agricolastrasse 2, 38678 Clausthal, Germany

Despite the increasing interest of the industry in lightweight materials during the last years an intensive industrial use of magnesium alloys due to the restricted cold-work-ability caused by its hexagonal lattice is still very limited. Considering this limitation a solution is provided by the process of superplastic forming of magnesium based alloys which, in contrast to other types of materials, is neither metalurgically developed nor process optimized. Presuppositions for the superplastic behavior of magnesium alloys are discussed in the introductory part of this presentation. Methods to quantify the superplastic behavior and the importance of the m-values derived thereof are explained in the following. Since a major precondition for superplastic forming is a very fine grain structure, various possibilities of grain-fining-procedures of magnesium-alloys are described with the help of parameter-lists and pictures of grain-structures. Finally, the results of the investigations (max. fracture elongation of 1050% at a constant strain rate of 1,6×10-4 s-1) are graphically displayed and possibilities of utilizing the superplastic characteristics, e.g. easy production of AM20, ZRE1 and QE22 magnesium alloy sheets with superplastic characteristics, are shown.

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Creep and Hot Working of Mg Alloys: *Hugh J. McQueen*[1]; W. Blum[2]; [1]Concordia University, Mech. Eng. Dept., 1455 Maisonneuve W., Montreal, Quebec H3G 1M8 Canada; [2]University of Erlangen-Nurenberg, Matls. Sci. Dept., Erlangen D91058 Germany

Die cast Mg alloy AZ91 was subjected to creep (<10-4s-1) in both compression and tension and to hot working (10-2-10 s-1) in torsion. Constitutive analysis showed that the dependencies of maximum deformation resistance (steady state creep rate at constant stress or steady state flow stress at constant strain rate) on temperature T were consistent across the entire range. The creep tests were concerned with the total strain within the operating life time and were compared to behavior of AS21. The torsion tests were concerned with the dependence of fracture strain on T and and were compared to the behavior of AZ31 and ZK60. Twinning took place at low strains to reorient grains not suited for slip and occurred much more profusely in the hot working. As a result of dynamic recovery, subgrains developed primarily near the grain boundaries, being much larger in creep than those in hot working consistent with the stresses. In the creep specimens, the particles underwent considerable change over the long periods. In contrast, during hot working the specimens underwent dynamic recrystallization that gave rise to considerably improved ductility and reduced strength at high T.

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Forging of Magnesium Using a Squeeze Cast Pre-Form: Gabriela Tausig[1]; *Nigel Jeffrie Ricketts*[2]; Stephen Ronald Peck[2]; [1]National Forge Operations, 465 Somerville Rd., West Footscray, Victoria 3012 Australia; [2]CSIRO, Manufacturing Science and Technology, Technology Court, Pullenvale, Queensland, Australia

The supply of magnesium billet for forging stock is limited to only a few suppliers. It is difficult to find and is expensive. It should ideally be DC cast and grain refined, but a number of suppliers provide billet in a cast form with a large grain size. An alternative to DC cast billet is a cast pre-form. In order to minimise shrinkage porosity, squeeze casting was used to provide porosity-free preforms in AZ31 magnesium alloy for forging trials. These trials were conducted at National Forge Operations and showed that the preforms via the squeeze casting route can produce good quality forgings in a single forging step. Forging of an automotive clutch hub at National Forge was able to be conducted more easily than the literature would suggest. A comparison of the modelling work and the results obtained is presented. Forging via a cast pre-form would appear to offer promise in the provision of forging stock for single-stage forging.

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Assessment of Equal Channel Angular Extrusion Processing of Magnesium Alloys: *S. R. Agnew*[1]; L. Chen[2]; Y. Lu[2]; M. Stoica[2]; D. Fielden[2]; P. K. Liaw[2]; [1]Oak Ridge National Lab, Oak Ridge, TN 37831 USA; [2]University of Tennessee, Knoxville, TN 37996 USA

Equal channel angular extrusion (ECAE) offers the potential to introduce very large strains into a metal workpiece without changing its cross-section. Hence, it could be an attractive technique for developing a fine-grained forging stock with good forming characteristics. In general, the mechanical properties of hexagonal close packed (hcp) metals respond even more positively to grain refinement than cubic metals. Furthermore, because ECAE is significantly slower than straight extrusion, die chilling is a strong concern for materials that must be processed hot. Magnesium alloys are ideal candidates to benefit from the ECAE technique, because they are hcp and their low melting point allows them to be processed isothermally at temperatures of 300ºC and below. An assessment of the technique has been made with the alloys AZ31B and ZK60. The mechanical properties of AZ31B appear largely unaffected by the process, however, the ZK60 alloy responds very favorably. Following ECAE, the ductility of ZK60 has been shown to increase by 2 to 3 times over the entire temperature range investigated (24-450deg C) with no significant change in strength. Possible explanations for the property enhancements and the development of crystallographic textures will be discussed.

5:05 PM

AM70-Magnesium Processed by Semi-Solid Casting: *Dierk Hartmann*[1]; Wolfram Wagener[1]; [1]EFU Gesellschaft für Ur-/Umformtechnik mbH, Simmerath (D)

The process of semi-solid casting (SSC) is defined by forming a metal with thixotropic behavior in a temperature range between solidus and liquidus. The thixotropic behavior is dependent on the type of alloy and on the value of shear stress that acts during the forming process. The viscosity of the material is reduced and the forces to fill the tool are relatively low and comparable to forces during a casting process. Combining the positive characteristic of forging (advanced microstructure) with the characteristics of casting (complexity), this alternative manufacturing process allows the production of parts with low porosity, for example, in a near-net-shape design. The reason semi-solid casting of magnesium is relatively unknown is the lack of a "standard" feedstock material and sufficient material property data. In this paper the use of hot extruded magnesium for SSC is described for the production of demonstrator components. Tensile test specimens were machined to achieve more information about mechanical properties of SSC-magnesium.

Materials Issues in Microelectronics - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Michael R. Notis, Lehigh University, Department of Materials Science, Bethlehem, PA 18015 USA; Sailesh Merchant, Lucent Technologies, Orlando, FL 32819 USA; Martin W. Weiser, Honeywell Electronic Materials, Spokane, WA 99216 USA; Jin Yu, KAIST, Department of Materials Science, Seoul, Korea

Wednesday PM
February 14, 2001

Room: 226
Location: Ernest N. Morial Convention Center

Session Chairs: Michael R. Notis, Lehigh University, Dept. of Mats. Sci. & Eng., Bethlehem, PA 18015 USA; Martin W. Weiser, Honeywell Electronic Materials, Spokane, WA 99216 USA

2:00 PM

Reactive Formation of a Solid Phase: *Francois M. D'heurle*[1]; [1]IBM Corporation, P.O. Box 218, Yorktown Heights, NY 10598-0218 USA

Fundamental aspects of the reaction of a solid with another solid, a liquid, or a gas in order to form a new solid layer containing one or several separate phases will be considered. For solid-solid reactions the usual linear or parabolic kinetics will be reviewed. The specific character of linear kinetics most prominent in the reaction of a solid with molecular gases will be described. In solid-solid reactions, e.g., in the formation of thin film silicides, one can often neglect the formation of terminal solid solutions in the two sources; this is not possible in solid-liquid reactions because (a) the solubility limits tend to be high, and (b) rapid diffusion in the liquid dominates the interaction. Althought the approach will be formal, practical illustrations pertinent to silicide formation, oxidation, and soldering will be highlighted.

2:30 PM Invited

Reactions at Materials Interfaces in Seimconductors: *Sailesh M. Merchant*[1]; [1]Lucent Technologies, Bell Laboratories, 9333 South John Young Parkway, Orlando, FL 32819 USA

Materials interfaces of semiconductor devices play an important role in determining device performance. An understanding of metallurgical and materials interactions at these interfaces is key to determining their reliability. This paper reviews the role of the metallurgist/materials scientist, who invokes simple principles of phase constitution, interdiffusion, phase transformations, mechanical and chemical interface stability, to understand and minimize these reactions. Examples are provided from various steps of semiconductor device processing where these materials phenomena are commonly observed. Reactions and instabilities at materials interfaces, such as during silicidation, interconnect formation, packaging and assembly of semiconductor devices, are reviewed.

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Silicidation Reactions of Ti/Ni Bilayer on Chemically Oxidized Si Surface: *Tan Wee Leng*[1]; Pey Kin Leong[2]; Chooi Simon[2]; Ye Jian-Hui[3]; Mangelinck Domanique[3]; Osipowicz Thomas[4]; [1]National University of Singapore, Dept. of Comp. and Elect. Eng., 10 Kent Ridge 117576 Singapore; [2]Chartered Semiconductor Manufacturing Ltd., 60 Woodlands Industrial Park D, St. 2, Singapore 738406 Singapore; [3]Institute of Material Research and Engineering, 4 Engineering Dr. 3, Singapore 117576 Singapore; [4]National University of Singapore, Dept. of Phys., 4 Engineering Dr. 3, Singapore 117576 Singapore

Interfacial reaction in a Ti/Ni/SiOx/Si system was studied in detail. The chemical oxide was prepared by treating a (100)Si surface chemically with NH4OH:H2O2:H2O solution which forms ~ 12Å of SiOx. A 300Å of Ni layer was then sputtered on the chemically prepared Si substrate, followed by a 50Å or 100Å sputter Ti deposition. Subsequently, the samples were annealed in RTP for temperature ranging from 500°C to 800°C. Material and chemical characterization using XRD, RBS and XPS depth profiling were carried out to identify the various phases of Ni silicide and to study the inter-diffusion of the different elements before and after annealing. It was found that with a 50Å Ti cap, no reaction occurs between Ni and Si up to a temperature of 750°C. In this temperature range, inter-diffusion between Ni and Si takes place with increasing annealing temperature but no reaction occurs. However, when a 100Å Ti cap was used, reaction between Ni and Si starts at 600°C, forming NiSi. At 750°C, our XRD results show the presence of both NiSi and NiSi2 phases. At 800°C and above, full conversion to NiSi2 took place. The possible mechanism responsible for the Ni-salicidation is proposed.

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Piezoelectric Actuation of Crack Growth Along Polymer-Metal Interface: Tianbao Du[1]; M. Zhang[2]; S. Seghi[1]; K. J. Hsia[2]; J. Economy[1]; *J. K. Shang*[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mats. Sci. and Eng., 1304 W. Green St., Urbana, IL 61801 USA; [2]University of Illinois at Urbana-Champaign, Dept. of Theor. and Appl. Mech., Urbana, IL 61801 USA

A new experimental technique for determining mechanical properties of the polymer-metal interface was developed by replacing the conventional mechanical testing machine with a piezoelectric actuator. Crack growth along an adhesive bond was found to depend on the magnitude of the applied electric field. The driving force for the crack growth was computed from the finite element analysis as a function of crack length, applied field, material properties and specimen geometry. Kinetics of the crack growth was correlated with the piezoelectric driving force. The resulting crack-growth behavior was compared with the results from the conventional mechanical testing technique. Work supported by the National Science Foundation under grant NSF CMS 98-72306.

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Estimations of the Interfacial Fracture Energy of a Cu/Cr/PI System by the T-Peel Test: *J. Y. Song*[1]; Jin Yu[1]; [1]KAIST, Center for Electronic Packaging Materials, 373-1 Kusong-dong Yusong-gu, Taejon, Korea

T-peel tests were conducted to determine the metal/polymer(m/p) adhesion strength of a Cu/Cr/PI structure with interface precracks between Cr and PI used in electronic packaging. Effects of the biased RF plasma pretreatment and the metal layer thickness on the peel strength (P) were investigated, and the energy dissipated by plastic bending (Ψ) and the interfacial fracture energy (Γ) are estimated. During the steady state peeling, the peel angle (ϕ) and the maximum curvature at peeled film bases were directly measured by using an optical camera, from which Ψ can be deduced from the elastic/plastic analysis of Kim and Aravas. P, Ψ and ϕ values vary with the plasma density (ρ) and the metal layer thickness. The interfacial fracture energy between Cr and PI which increases with ρ but is independent of m/p layer thickness were deduced. Later, Γ values were compared with the theoretical analysis by Moidu et. al. and Wei and Hutchinson.

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Shear Strength of a Cu-Ta Interface by Molecular Dynamics: *Pekka Heino*[1]; [1]Tampere University of Technology, Electronics, P.O. Box 692, Tampere FIN 33101 Finland

During last few years, the electrical and mechanical properties of copper have received a lot of interest in the electronics community, mainly because of its low electrical resistance. Recently we have studied mechanical properties of nanoscale pure copper connections \1\. However, a barrier layer between copper and the rest of the system is needed to prevent diffusion. In such disordered systems the interfaces are often the weakest spots. Thus, in this work we study the interface of copper and tantalum, which is often used as a barrier metal, because of positive heat of formation. We use molecular dynamics with embedded-atom potentials as means. We study the energy and shear strength of several low-index interfaces. A strong positive correlation is found indicating that low-energy interfaces are weak. In addition, some interfaces are formed by de-

positing Cu on different Ta surfaces and their microstructure is analyzed. 1. P. Heino and E. Ristolainen: Mechanical properties of nanoscale copper under shear, Microelectronics Reliability 40 (2000) 435-441

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Void Formation of Wire-Bond During High Temperature Aging: *Ker-Chang Hsieh*[1]; Hen-So Chang[1]; Theo Martens[2]; Albert Yang[2]; [1]National Sun Yat-sen University, Instit. of Matls. Sci. and Eng., Kaohsiung, Taiwan; [2]Philips Electronic Bldg. Elements Industries, Ltd., Tech. Dev. Div., 10, Chin 5th Rd. N.E.P.Z., P.O. Box 35-48, Kaohsiung, Taiwan

Voids are formed along with the growth of gold/aluminum intermetallic phases at wire-bond interfaces during high temperature aging. This phenomenon is known to degrade the bond.The purpose of this study is to clarify the factors that influence bondability and bond degradation, and to understand the mechanism of void formation. Samples were prepared under various wire-bonding conditions, Al-pad thickness, aging temperatures and aging times. The treated samples were examined, and the ball bond cross section microstructure is reported. Microstructure analysis was perfomed on a JEOL Superprobe JXA-8900R.

5:00 PM Invited

Materials and Process Challenges for Packaging Higher Density/Higher Frequency Microelectronics: *Mark Thomas McCormack*[1]; [1]Fujitsu Computer Packaging Technologies, 3811 Zanker Rd., San Jose, CA 95134 USA

Many issues are apparent upon review of the requirements put forth by the electronics industry's microelectronics packaging roadmaps. Among the most challenging are those that will require new or modified fabrication processes and materials sets. After discussing many of these issues in broad terms, selected examples that concentrate on solving higher density/higher frequency in package substrates will be discussed.

Materials Processing Fundamentals VI

Sponsored by: Extraction & Processing Division, Materials Processing and Manufacturing Division, Process Fundamentals Committee, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: P. N. Anyalebechi, ALCOA, Ingot & Solidification Platform, Alcoa Center, PA 15069-0001 USA; A. Powell, MIT

Wednesday PM Room: 218
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Ray Y. Lin, University of Cincinnati, Dept. of Mat. Sci. & Met. Eng., Cincinnati, OH 45221-0012 USA

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Optimization of Oxide Powder Extrusion Paste for Forming Alloy Honeycomb Precursors: *Joe K. Cochran*[1]; Kevin M. Hurysz[1]; Raymond H. Oh[1]; Wesley D. Seay[1]; [1]Georgia Institute of Technology, Mats. Sci. and Eng., 771 Ferst Dr., Atlanta, GA 30332-1320 USA

The extrusion of oxide powders pastes through a thin-wall honeycomb die results in a low density metallic structure following reduction. These pastes are a combination of two phases: a solid phase composed of the particular oxide or oxide mixture carried by a fluid solution of water, binder, and lubricant. The key to forming high quality, defect free extrudate lies in the optimization of paste properties and is contingent on solids loading and fluid-phase rheology. To extrude efficiently, the fluid must yield to the paste enough compliance to flow through the die, yet provide a high enough viscosity or yield stress at low shear rates to avoid deformation following extrusion. Measurements of viscosity, yield stress, and wall shear stress are used to characterize water-lubricant and water-binder solutions. These data are used to optimize the water-binder-lubricant solution and facilitate the extrusion of articles having complex geometry. This investigation will consider the paste compositions necessary to form high thermal conductivity (copper), structural (maraging steel), and elevated temperature (Inconel 617) alloys.

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Sulfidation of Chalcopyrite with Gaseous Sulfur: *Rafael Padilla*[1]; Marcial Torres[1]; Maria Cristina Ruiz[1]; [1]University of Concepcion, Dept. of Metall. Eng., Edmundo Larenas 270, Concepcion, Chile

The reaction of chalcopyrite with gaseous sulfur has been investigated in the range 350-450°C in order to transform this mineral into a more acid soluble copper sulfide and insoluble iron sulfide. The results of thermodynamic analysis of this system as well as the X-ray diffraction analysis of the reaction products showed that the in the temperature range 350-450°C the sulfidation reaction proceeded without formation of intermediate copper-iron sulfides according to: CuFeS2+1/2S2(g) = CuS+FeS2 The effects of temperature and time on the conversion of chalcopyrite were studied. Conversion of chalcopyrite reached about 80% very rapidly with increase in temperature up to about 400°C. The recovery of copper from the sulfidized samples was determined by leaching in oxygenated 1.2M H2SO4, 2M NaCl solution. Copper recoveries over 90 % were obtained from samples sulfidized for 30 min at temperatures lower than 400°C.

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Metallic Iron Reactions in Aqueous Systems: *Batric Pesic*[1]; Victor C. Storhok[1]; [1]University of Idaho, Coll. of Mines, McClure Hall, Moscow, ID 83844-3024 USA

Despite its ubiquitous nature and its role as of the most important metals to the mankind the dissolutions reactions of iron in aqueous solutions, surprisingly, have not been studied. There is an enormous body of literature on corrosion of iron in aqueous systems but all the reported studies were electrochemical in nature, i.e. corrosion studies. This paper will discuss the reactions of iron with water as a function of pH, temperature, surface geometry, size, application of external potentials, and finally the possible role bacterium, Thiobacillus ferrooxidans. Fundamental knowledge of chemical reactions of iron with water and water constituents is of importance to understanding the processes of corrosion of iron and its alloys, and also of importance to the development of hydrometallurgical processes involving iron in either solid or soluble form.

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Magnetite Stratification During Slag Reduction: *Gabriel Riveros*[1]; *Andrzej Warczok*[1]; [1]Universidad de Chile, Departamento de Minas, Av. Tupper 2069, Santiago, Casilla 2777 Chile

Smelting of copper concentrate into white metal produces highly oxidized slag containing from 15 to 25% of magnetite. Copper recovery from the slag requires effective magnetite reduction and phase separation. Slag reduction and cleaning in an electric furnace is commonly used. Results of slag reduction with graphite electrodes in a crucible simulated electric furnace showed strong tendency for magnetite stratification and formation of related gradient of copper content along the slag height. Microscopic and analytical examination of slag samples allowed for determination of various parameters, such as electrode immersion, current intensity and reverts addition, on distribution of magnetite content. Analysis of mechanisms of magnetite stratification in an electric furnace pointed out the role of the immersion of electrodes, current density, reverts and coke addition. Possibilities of formation of dead zones and built-up on the furnace hearth have been discussed.

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In-Situ Monitoring of the Sintering Process with Non-Contact Electromagnetic Acoustic Transducers: *James C. Foley*[1]; David K. Rehbein[1]; Daniel J. Barnard[1]; [1]Ames Laboratory, Metallurgy and Ceramics Program, 122 Metals Development, Ames, IA 50011 USA

In-situ characterizations of green state part density and sintering state have long been desired in the powder metal community. Recent advances in non-contact electromagnetic acoustic transducer (EMAT) technology have enabled in-situ monitoring of acoustic amplitude

and velocity as sintering proceeds. Samples were made from elemental powders of Al (99.99%), Al (99.7%), Ag, (99.99%), Cu (99.99%) and Fe (99.9%). The powders were pressed in a uniaxial die and examined with acoustic waves for changes in velocity and amplitude during sintering. The changes in acoustic properties were correlated with sample microstructures and mechanical properties. Evolution of a series of reverberating echoes during sintering is shown to provide information on the state of sintering, changes in sintering kinetics as well as having the potential for detection of interior flaws. This work is funded by a laboratory directed research and development grant and by DOE-BES under contract no.W-7405-Eng-82.

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A Model for Coupled Laminar Fluid Flow and Electrochemical Reactions in Electric Field Smelting and Refining of Steel: *David Michael Dussault*[1]; Adam Powell[2]; [1]MIT, Mats. Sci., 77 Massachusetts Ave., Rm 4-033, Cambridge, MA 02139 USA; [2]MIT, Mats. Sci. and Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139 USA

A model for coupled laminar fluid flow and electrochemical reactions is developed using the Navier-Stokes equations and the phase field method. Application to the electric field enhanced refining of steel is discussed. Because the process is limited by ferrous ion transport to the cathodic slag-metal interface, the Mullins-Sekerka instability gives rise to liquid iron fingers protruding into the slag. Two-dimensional numerical results are given which show that the model captures this instability.

Modeling of High Temperature Alloys: Alloy Modelling

Sponsored by: Structural Materials Division, High Temperature Alloys Committee

Program Organizers: Shailesh Patel, Special Metals, Huntington, WV 25705-1771 USA; Gerhard E. Fuchs, University of Florida, Department of Materials Science and Engineering, Gainesville, FL 32611-6400 USA

Wednesday PM Room: 221
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Gerhard E. Fuchs, University of Florida, Mats. Sci. & Eng., Gainesville, FL 32611-6400 USA

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Materials Properties Modelling for Ni-based Superalloys: *Nigel Saunders*[1]; Xiuqing Li[1]; [1]Thermotech, Ltd., Surrey Technology Centre, The Surrey Research Park, Guildford, Surrey GU2 7YG UK

Thermodynamic modelling has reached a status where predictions for the phases present in Ni-based superalloys can be made to a high degree of accuracy [1]. Although pertaining to equilibrium such calculations have already had significant application in industrial practice [2]. However, it is clear that the long term goal of such modelling should be to provide a tool that will predict more general materials properties, for example long term stability with regard to TCP phase (sigma, mu...) formation, more general TTT diagrams for NiFe-based superalloys, mechanical properties, thermo-physical and physical properties, etc.. The present paper will report on new work to achieve this goal, in particular with respect to work on TTT diagrams and preliminary results on mechanical properties. References [1] N. Saunders, Superalloys 1996 eds. R. D. Kissinger et al. (Warrendale, PA: TMS, 1996), 101 [2] N. Saunders, M. Fahrmann and C. J. Small, to be presented at Superalloys 2000, Seven Springs, PA, Sept.21-25 2000

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Modeling of the Partitioning and Phase Transformation Temperatures of a As-Cast a Third Generation Single Crystal Ni-base Superalloy: *Gerhard E. Fuchs*[1]; Brett A. Boutwell[2]; [1]University of Florida, Mats. Sci. & Eng. Dept., P.O. Box 116400, 116 Rhines Hall, Gainesville, FL 32611-6400 USA; [2]AEA Technology, Mats. & Chem. Proc. Assess., 241 Curry Hollow Rd., Pittsburgh, PA 15236-4696 USA

Cast single crystal Ni-base superalloys exhibit severe solidification segregation that must be subsequently removed by solution heat treatment. In order to understand how some of the elements in these alloys effect the solidification partitioning, the as-cast microstructure of the third generation single crystal Ni-base superalloy, CMSX-10 was examined. In addition, the solidification partitioning was calculated using Thermo-Calc. The solidus, liquidus and gamma-prime solvus temperatures were also calculated for the compositions of the base alloy, and the determined compositions of the the dendrite cores and the interdendritic regions. All of the calculated values were compared to the experimentally determined values. The results of this study and areas for future work will be discussed.

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Examining the Effects of Elastic Stress in Ostwald Ripening Through Numerical Simulation: *Katsuyo Thornton*[1]; Norio Akaiwa[2]; P. W. Voorhees[3]; [1]Massachusetts Institute of Technology, Dept. of Mats. Sci. and Eng., 77 Massachusetts Ave., 13-5130, Cambridge, MA 02139 USA; [2]National Research Institute for Metals, Tsukuba, Japan; [3]Northwestern University, Dept. of Mats. Sci. and Eng., 2225 N. Campus Dr., Evanston, IL 60208 USA

We examine the effects of elastic stress on the kinetics of coarsening in an elastically homogeneous, anisotropic solid. Our computational model simulates Ostwald ripening in a solid-solid system efficiently and accurately by taking advantage of powerful numerical methods such as the boundary integral method with the fast multipole method. We study large systems consisting of many thousands of particles to produce quantitative, statistically meaningful measures of the temporal evolution of the microstructure. Our numerical simulation indicates that the power-law exponent for the average particle size remains 1/3, while the rate constant depends on the elastic stress. We provide insight into the fundamental phenomena underlying the processes that govern the evolution of the microstructures in elastically stressed solids through both theory and the simulation.

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Nucleation of Gamma' and Gamma" in INCONEL 706 Utilizing Computational Thermodynamics and Diffusion Kinetics: *Brett A. Boutwell*[1]; Raymond G. Thompson[2]; [1]UES Software, Inc., 241 Curry Hollow Rd., Pittsburgh, PA 15025 USA; [2]University of Alabama at Birmingham, Dept. of Mats. & Mech. Eng., 1150 10th Ave. South, Birmingham, AL 35294-4461 USA

The nucleation and subsequent growth of gamma' and gamma" precipitates in INCONEL alloy 706 has a significant impact on the mechanical properties, and therefore the performance, of the alloy. The growth of such precipitates can be modeled using various models and numerical methods, such as those incorporated in the software package DICTRA. Nucleation, however, is a bit more difficult process to model given the large number of factors that can influence the alloy behavior. A model for predicting nucleation kinetics of coherent, homogeneous precipitates using data from computational thermodynamic and diffusion kinetic data was developed. The thermodynamic and kinetic values needed for the nucleation model were obtained from Thermo-Calc and DICTRA. A database of atomic mobilities was created to allow DICTRA to model the multicomponent diffusivities of alloy 706. The nucleation model was then used to predict the incubation time of gamma' and gamma" precipitation in INCONEL alloy 706 at several temperatures. The results of the model calculations were compared to T-T-T and T-T-H data for the alloy.

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Application of Computational Thermodynamics to Model Stray Grain Formation: *John M. Vitek*[1]; Stan A. David[1]; S. S. Babu[1]; [1]Oak Ridge National Laboratories, Oak Ridge, TN 37831-6096 USA

The paper will examine stray crystal formation in single crystal material. Extensive use of computational thermodynamics will be made to quantitatively identify some of the factors that influence the extent of stray crystals.

4:25 PM

Capitalizing on Computational Tools in the Development of a New Low-Cost Diesel Exhaust Valve Alloy: *Michael Gustav Fahrmann*[1]; Gaylord D. Smith[1]; [1]Special Metals Corporation, Huntington Alloys/Technology, 3200 Riverside Dr., Huntington, WV 25705 USA

Recently, computational tools such as Thermo-Calc have been made available to industry to predict phase equilibria in multi-component alloy systems. These computer codes, in conjunction with validated thermo-chemical databases, account to a large degree for the complexity of commercial alloys. An example is presented as to how this tool has actually been used to expedite alloy development for new low-cost diesel exhaust valve material. The current predictive capabilities of Thermo-Calc in conjunction with a commercial Ni-database are also demonstrated in this context.

4:50 PM

Calculation and Verification of Solidification Diagrams in Superalloys: *Wanhong Yang*[1]; Keh-Minn Chang[1]; Wei Chen[2]; Sarwan K. Mannan[3]; Shailesh J Patel[3]; [1]West Virginia University, Morgantown, WV 26506-6106 USA; [2]General Electric, Power Systems, Schnectady, NY USA; [3]Special Metals, 3200 Riverside Dr., Huntington, WV 25705 USA

Solidification is the most important process that determines the segregation and structure of a material. Niobium containing nickel base superalloys were studied using differential thermal analysis, interrupted quenching of solidifying metals and scanning electron microscopy/electron dispersive spectrometry. Liquidus temperature, elemental partitioning coefficient and solid fraction formation in the mushy zone was measured. The solidification was also modeled using the commercial software package Thermo-Calc and a Ni-database, which has been successfully used for solid state phase calculation of superalloys. Comparison of the experimental results with calculation generally yielded good agreement. The versatility of the thermodynamic calculation was also proved. However, large scattering in the liquidus temperature prediction and deviation from measured partitioning coefficient were observed.

Properties of Nanocrystalline Materials: Magnetic Properties

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Jt. Mechanical Behavior of Materials, Chemistry & Physics of Materials Committee

Program Organizers: Sung H. Whang, Polytechnic University, Department of Mechanical Engineering, Brooklyn, NY 11201 USA; Horst W. Hahn, Techische Hchschule Damstadt, Darmstadt D-64287 Germany; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552 USA

Wednesday PM — Room: 223
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Bhakta B. Rath, NRL, Washington, DC USA; Robert D. Shull, NIST, 855.11, Gaithersburg, MD 20899-8552 USA

2:00 PM Invited

Observation of Hybrid Domain Walls in Exchange-Coupled Ferromagnet/Antiferromagnet Bilayers Using the Magneto-Optic Indicator Film (MOIF) Technique: *R. D. Shull*[1]; A. J. Shapiro[1]; V. S. Gornakov[2]; V. I. Nikitenko[2]; N. Goekemeijer[3]; C. L. Chien[3]; [1]National Institute of Standards and Technology, Mats. Sci. and Eng. Laboratory, 100 Bureau Dr., MS8552, Gaithersburg, MD 20899-8552; [2]Institute of Solid State Physics, RAS, Chernogolovka 142432 Russia; [3]The Johns Hopkins University, Dept. of Phys. and Astron., Baltimore, MD 21218 USA

Microscopic domain processes have been observed at 300K in an exchange-coupled ferromagnet FM)/antiferromagnet (AF) bilayer of $Ni_{81}Fe_{19}$ (160 Å)/$Fe_{50}Mn_{50}$ (300 Å). The bilayer had been ac demagnetized above the Néel temperature of FeMn and cooled in zero field to 300K. Consequently, hysteresis loop measurements showed two loops shifted to opposite sides of the origin. Domain imaging (via the MOIF technique) revealed alternately directed domains separated by hybrid domain walls, consisting of both FM and AF "parts" extending through the bilayer. The AF domain walls remained intact even after the reversal of the FM magnetization (M); only the FM portion of the hybrid domain wall moved. Upon decrease of the field (H), the magnetization decrease was initiated in the FM adjacent to the AF interface. Also, a new type of asymmetry was observed in the remagnetization process for an off-axis H.

2:30 PM Invited

Synthesis, Processing and Magnetic Properties of Nanostructured Gamma Ni-Fe Alloys: *Jai-Sung Lee*[1]; Yun-Sung Kang[1]; [1]Hanyang University, Dept. of Metall. and Mats. Sci., 1271 Sa-1-dong, Ansan, Kyunggi-do 425-791 Korea

The nano-processing has been known to be a potential and promising way to improve the magnetic properties of the Ni-Fe alloy system, such as higher permeability and lower coercivity. In this paper we report on a new processing route for fabricating nanostructured (ns)gamma-Ni-Fe alloy and on its related magnetic properties. The processing of ns gamma-Ni-Fe alloy in this study, which consists of nano powder synthesis and its consolidation process, has been conducted using a mechano-chemical process (MCP) specially for powder synthesis. Regarding the processing, we focus on two kinetic issues occuring during processing of gamma-Ni-Fe nano alloy; in-situ alloying process and densification process of gamma-Ni-Fe nano powder. Magnetic properties of the ns gamma-Ni-Fe alloy were investigated as a function of grain size and the result was discussed in terms of microstructure and chemical property.

3:00 PM Invited

Magnetic Properties of Co Nanocrystals in CoB Amorphous Matrix: A. González[1]; A. Hernando[1]; [1]Instituto de Magnetismo Aplicado, P.O. Box 155, 28230 Las Rozas (Madrid), Spain

The magnetic properties and crystallization behavior of two amorphous compositions of the Co-B system, $Co_{80}B_{20}$ and $Co_{75}B_{25}$, have been studied. By comparing the results it is found that the excess of Co with respect to the stequiometric Co_3B in the $Co_{80}B_{20}$ sample has a major effect on the properties of the amorphous material and on the crystallization process. Transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM) and differential scanning calorimetry (DSC) have been used to characterize the different crystallization states of $Co_{80}B_{20}$ alloy. This composition undergoes a primary crystallisation which transforms the material into a two phase system composed of Co nanocrystals embedded in a Co-B ferromagnetic amorphous matrix. The role of the cobalt grains on the magnetisation processes and the coercive field is studied for temperature ranging from 5 to 300K. It is found that the hardening effect of the grains diminishes on increasing the temperature. This behavior has been analyzed in terms of the magnetic coupling between grain

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Magnetic Microstructure and Anisotropy in Nanocrystalline Feromagnets Measured by Small-Angle Neutron Scattering: *Joerg Weissmueller*[1]; [1]Institut fuer Nanotechnologie, Forschungszentrum Kalrsruhe, Gabh,Herman-von-Helmholtz-Platz 1, D-76334 Germany and Technische Physik, Universitet des Saarlandes, Saarbrfacken, Germany

Magnetic small-angle neutron scattering (SANS) studies provide the unique opportunity to probe the magnetic microstructure in the bulk of nanocrystalline ferromagnets with a spatial resolution of few nm to a few 100nm, complementing imaging techniques which are sensitive to the materials surface and to larger structures. Recent progress in the SANS data analysis has made it possible to obtain quantitative information on (i) the magnetic microstructure and its variation as a function of the applied magnetic field, H, (ii) the ferromagnetic exchange-stiffness constant, and (iii) the magnitude

and microstructure of the magnetic anisotropy. SANS data for the magnetically soft nanocrystalline transition metals Ni and Co show that the dominant structure of the magnetization are continuous fluctuations of the spin directions about the direction of the applied field, with correlation lengths that vary, as a function of H, from few nm to (at least) tens of nm. By contrast, for the magnetically hard rare earth Tb the magnetization is found to be 91 locked into the basal plane of each crystallite up to applied fields of several Tesla.

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Nanocrystalline Hard Magnetic Materials: *Oliver Gutfleisch*[1]; A. Bollero[1]; A. Kirchner[1]; K. H. Muller[1]; L. Schultz[1]; [1]Institute of Solid State and Materials Research, P.O. Box 270016, Dresden 01171 Germany

Recent developments that occurred in nanocrystalline rare earth-transition metal hard magnets are reviewed and particular emphasis is placed on ongoing research work at IFW Dresden. Principal synthesis methods used include mechanical alloying, melt spinning and hydrogen assisted methods such as reactive milling and the hydrogenation-disproportionation-desorption-recombination (HDDR) process. These processing techniques are applied to NdFeB-, NdFeC-, PrFeB-, SmFe- and SmCo-type systems with the aim to produce high remanence magnets with high coercivity. Concepts of maximizing the energy product in nanostructured magnets by either inducing a texture via anisotropic HDDR or hot deformation or enhancing the remanence via exchange coupling are evaluated. The latter phenomenon is observed in nanocomposite magnets consisting of soft and hard magnetic phases where the magnetic interaction leads to magnetically single demagnetization curves despite a multi-phase microstructure provided grain sizes are below a certain threshold and paramagnetic intergranular phases are absent. This has been realized for example in thermodynamically highly stable Sm_2Co_{17} compounds which have been grain refined via a mechanically induced reversible gas-solid reaction using severe hydrogenation conditions. Finally, highly textured, radially oriented $Nd_2(Fe,Co,Ga)_{14}B$-based ring magnets produced by backward extrusion with improved temperature coefficients of coercivity and an energy density $(BH)_{max}$ of 325kJ/m^3 are described.

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Nanocomposite Magnetic Material: *Pekka Ruuskanen*[1]; M. Karttunen[1]; J. Enqvist[1]; [1]VTT Technical Research Center of Finland, Dept. of Chem. Tech., P.O. Box 1402, Tampere 33101 Finland

A nanocomposite magnetic material was developed using iron nanoparticles with a diameter of less than 80 nm. The iron nanoparticles were mixed with a styrenic block copolymer using a Brabender Plasticorder batch mixer. After mixing, the resulting material was compacted in a hot press at a temperature of 180°C. Because it proved difficult to obtain an adequate packing density with the iron nanopowders in their original form, a procedure for modifying the surface of the nanoparticles was developed to improve wetting by the plastic melt. This procedure made it possible to achieve a packing density of about 40 vol%. The quality factor (Q) and the permeability (i) of the compacted samples were measured at different frequencies using commercially-available ferrite plate and iron particles with an average diameter of approximately 30 ìm as the reference material. It was found that the quality factor Q at higher fre quencies was much higher in the nanocomposite magnetic material than in the reference material. At a frequency of 8.2 MHz the quality factor Q was 175 in the polymer nanocomposite and 400 in the commercial ferrite. When the frequency was increased to 200 MHz, the quality factor Q was 70 in the polymer nanocomposite and 3 in the commercial ferrite. The permeability of the nanocomposite material remained at an almost constant value of 11 as the frequency was increased from 8.2 MHz to 200 MHz while the permeability of the commercial ferrite material fell from 400 to 33 over the same frequency range. The results obtained show that nanotechnology has potential as a way of developing magnetic materials for use in high-frequency applications.

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Structure Peculiarities and Magnetic Properties of Nanocrystalline Thin Films: *Vladimir Grigor'evich Shadrow*[1]; Anatolyi Vasilyevich Boltushkin[1]; Lyudmila Vasil'evna Nemtsevich[1]; [1]Inst. Solid State Physics, Acad. Sci. of Belarus, P. Brovki,17, Minsk, Belarus 220072 Belarus

Growth processes,structure peculiarities and properties of hard and soft magnetic Co and Fe based films have been investigated by means of EM, XRD, AFM and AGFM as well as post deposition treatment effect on the films structure and properties. A mechanism of nanocrystalline structure formation and its influence on the films properties is discussed.Intergranular magnetic interaction and magnetization reversal processes in the above films are investigated through remanence and delta M curves measurements and time dependence measurements.

Sampling, Sensors & Control for High Temperature Metallurgical Processes: Aluminum Reduction Technology - Advanced Control

Sponsored by: Light Metals Division, Extraction & Processing Division, Materials Processing and Manufacturing Division, Aluminum Committee, Pyrometallurgy Committee, Jt. Processing Modeling Analysis & Control Committee

Program Organizers: Adrian Deneys, Praxair, Inc., Tarrytown, NY 10591 USA; Derek Fray, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3Q2 UK; Matt Krane, Purdue University, Department of Materials Engineering, West Lafayette, IN 47907; Markus Reuter, Delft University of Technology, Applied Earth Science, Delft 2628 RX The Netherlands; Fiona Stevens McFadden, University of Auckland, Chemistry and Materials Engineering, Auckland, New Zealand

Wednesday PM — Room: 230
February 14, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Fiona Stevens McFadden, University of Auckland, Chem. and Mats. Eng., Auckland, New Zealand; Matthew John M. Krane, Purdue University, Dept. of Mats. Eng., West Lafayette, IN 47907 USA

2:00 PM

Control Electrochemical Cell Dynamics with Electrode Current Measurements: *James R. Barclay*[1]; [1]Universal Dynamics, #100-13700 International Place, Richmond, BC V6V 2X8 Canada

Knowing the electrical current distribution in a multiple-electrode, electrochemical cell, for example, a prebake aluminum reduction pot, provides significant insights into the cell dynamics. Metal pad movements, bath chemistry variations, anode spikes, bubble phenomena all affect the current distribution and efficiency of the cell. This paper describes a robust current sensing system that graphically shows cell dynamics. The current sensors are suitable for harsh reduction cell environments and are easily installed and maintained. The system can display real time data, analyze historical data and graphically replay cell events.

2:25 PM

Digital Processing of Anode Current Signals: An Opportunity for Improved Cell Diagnosis and Control: Graeme C. Barber[2]; *Jeffrey T. Keniry*[4]; Mark P. Taylor[3]; Barry J. Welch[4]; [1]Alumination Consulting Pty, Ltd., 2, Governors Dr., Mt Macedon, Vic 3441 Australia; [2]Consultant, 5, Fullwood Pde, Doncaster East, Vic 3109 Australia; [3]Comalco Aluminium Ltd., 12, Creek St., Brisbane, Qld 4000 Australia; [4]University of Auckland, Dept. of Chem. and Mats. Eng., Auckland, New Zealand

While digital signal processing (DSP) is now commonplace in many industrial applications, it has received surprisingly little attention or application in aluminium smelting. Despite advances in data acquisition and storage, line current and cell voltage remain the only signals that are continuously monitored for control of vital cell functions such as alumina feeding, thermal regulation and magnetic stability. But are we using these signals to their maximum potential for diagnosis and control of the process? A full complement of

anode signals has been studied from industrial cells using high frequency (50Hz) sampling, with subsequent processing in time and frequency domains using the Fast Fourier (FFT) technique. While the imprinting of the metal surface motion is a well-known observation under low frequency sampling, this work shows that the higher frequency signals associated with bubble formation and release from the anodes also provide an imprint for specific process events and cell behaviour. The potential applications of DSP of individual anode and composite cell signals in diagnosis and control are discussed.

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Application of Advanced Process Control to Aluminium Reduction Cells-A Review: *Fiona J. Stevens McFadden*[1]; Geoffrey P Bearne[2]; Paul C. Austin[3]; Barry J. Welch[1]; [1]Auckland University, Chem. and Mats. Eng., Private Bag 92019, Auckland, New Zealand; [2]Comalco Research, P.O. Box 315, Thomastown, Victoria 3074 Australia; [3]University of Auckland, Elect. & Electr. Eng., Private Bag 92019, Auckland, New Zealand

The aluminium electrolysis process is fundamentally unchanged since its advent in the late 1880's. The control of the process has however, developed since then, with the trend being to increased mechanisation and automation. Process computers were implemented in the mid-1960's and currently the process is controlled with a mixture of automated and manual systems. In terms of control algorithms, although there have been refinements in use, the principles of the control strategies have not changed substantially in the last 30 years and control of the process outputs is in general achieved using single-input/single-output control loops. Through the application of advanced process control, which draws on elements from disciplines ranging from control engineering, signal processing, statistics, decision theory and artificial intelligence, performance improvements have been gained in other process industries. For aluminium reduction cells optimal control has been investigated along with artificial intelligence techniques such as fuzzy logic control, expert systems and neural networks for identification, prediction and control. Process simulators (physio-chemically derived dynamic models) have also been developed for the development and tuning of control strategies.

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A Multivariable Control of Aluminum Reduction Cells: *Kevin L. Moore*[1]; Nobuo Urata[2]; [1]Utah State University, CSOIS/ECE, 4160 Old Main Hill, Logan, UT 84322 USA; [2]Kaiser Aluminum and Chemical Corporation, Ctr. for Tech., Prim. Al Bus. Unit, 6177 Sunol Blvd., Pleasanton, CA 94566 USA

This paper considers control of the aluminum reduction process, using a dynamic model developed from the literature and Virtpot, a model developed at Kaiser Aluminum. Analysis shows the process is controllable and observable, but not easily stabilizable using one input, and that short-term changes in measured voltage result primarily from changes in alumina concentration rather than anode-to-cathode distance (ACD). Next, a multivariable control strategy is developed to regulate cell voltage by adjusting feed rate rather than beam movement. We introduce the idea of a feed voltage, obtained by subtracting expected voltage deviations due to ACD changes and beam moves from the filtered voltage. Feed rate is adjusted to compensate for deviations of feed voltage from its target. Simultaneously, beam movements are made to compensate for the difference in expected anode consumption and metal pad rise, based on changes in feed period. Simulations show the effectiveness of the proposed control strategy.

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From Cell Models to a Virtual Potroom: *Laszlo G. Tikasz*[1]; Rung T. Bui[1]; Vincent Villeneuve[1]; Sylvain Doyon[1]; [1]University of Quebec at Chicoutimi, Dept. of Appl. Sci., 555 Univ. Blvd., Chicoutimi, Quebec G7H 2B1 Canada

This paper analyzes the 'virtual cell' concept in which dynamic models of aluminium electrolytic cells are used as training, operation support and research tools in aluminium reduction plants. A systematic approach is proposed to transform a chosen cell model into a virtual cell that exchanges data with its environment like a real cell does. Then, a method is given to clone a virtual cell to form a group of cells or even populate an entire 'virtual potroom'. The virtual cells-like the real ones-are identical by design but individual in performance. Every one of these virtual cells is under automatic control. The controllers, organized into a hierarchical scheme, can be real or virtual. Examples are given for various cell and controller arrangements simulating typical cell operations in a plant.

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Virtual Aluminum Reduction Cell: *V. V. Yurkov*; V. Ch. Mann; T. V. Piskazhova; K. F. Nikandrov

At the TMS Annual Meeting of 2000 the "Model of Process of Electrolysis" was presented. Applying this dynamic mathematical model it was possible to imitate the operation of the industrial cell as the first approximation. After the conference this work was continued. A number of active and passive experiments were conducted. Specially designed diagnostic equipment was used for measuring and recording into a database temperature regimes of the cell different units. At the same time in order to identify the model some calculations on the model were carried out (equations selected and coefficients adjusted). This paper presents the results of the performed work, describes a "virtual cell", created on the basis of the dynamic model and a "virtual control system", which is the mathematical twin of an electrolysis control system operated at KRAZ.

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Development of Fuzzy Expert Control Technique for Aluminum Electrolysis: *Jie Li*[1]; *Fengqi Ding*[1]; Zhong Zou[1]; Minjun Li[1]; *Yexiang Liu*[1]; *Youkang Bian*[2]; Zhiming Wu[2]; *Gang Liu*[2]; [1]Central South University of Technology, Dept. of Metall. Sci. and Eng., Changsha, Hunan 410083 China; [2]Qinghai Aluminum Corporation, Xining, Qinghai 810108 China

In order to upgrade the basic control unit called "cell controller" in process control systems of aluminum electrolysis, a new control algorithm called "Fuzzy Expert Controller (FEC)" was developed. Because all the knowledge and experience that can be collected from the field experts as well as all the information which can be sampled for the analysis and control of cell state are not precise, the FEC was designed as a rule-based system working with rules in which imprecise and precise propositions were mixed freely. Its self-regulation mechanism adjusted the universes of discourse of fuzzy variables according to the change of cell state and the transition of control modes, achieving the object of on-line modifying its operating points and dynamic and static performance. Application results on smelters showed that the control accuracy, robustness and stability were satisfactory, and remarkable effects of production increasing and energy saving were achieved.

Second Global Symposium on Innovations in Materials Process & Manufufacturing: Sheet Materials: Composite Processing

Sponsored by: Materials Processing and Manufacturing Division, Powder Materials Committee, Shaping and Forming Committee, Solidification Committee

Program Organizers: Mahmoud Y. Demeri, Ford Motor Company, Manufacturing Systems Dept., Northville, MI 48167 USA; Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; John Papazian, Northrup Grumman, Bethpage, NY 11714 USA; Klaus Siegert, University of Stuttgart, Institute for Metal Forming Technology, Stuttgart D-70174 Germany

Wednesday PM, February 14, 2001 — Room: 228, Location: Ernest N. Morial Convention Center

Session Chairs: Patrick Blanchard, Ford Motor Company, Ford Research Laboratory, P.O. Box 2053, MD3135, SRL, Dearborn, MI 48121-2053 USA; Carl Johnson, Ford Motor Company, Ford Research Laboratory, Dearborn, MI 48121-2053 USA

2:00 PM Invited

The Future of Thermoplastic Composites in Automotive Applications: *Carl Johnson*[1]; [1]Ford Motor Company, Ford Research Laboratory, P.O. Box 2053, MD3135, SRL, Dearborn, MI 48121-2053 USA

In recent years the automotive industry has expressed an interest in stamped thermoplastic composites as these materials can be formed into highly complex cosmetic and semi structural components. The fundamental material compositions take advantage of continuous fibers to improve component performance when compared to conventional injection or compression molded thermoplastics. The improvement in mechanical properties, in turn, provides the opportunity to design a new class of applications utilizing thin wall lightweight structures. Considering the distinct advantages of continuous fiber stamped thermoplastics, implementation of these materials within the auto industry has been limited. This is due, in part, to the absence of robust processing technology and a requirement for engineering design methods. This paper examines the state of progress in implementing this technology by considering a number of current applications. A discussion of the benefits and limitations of material configurations and processing characteristics is presented in the context of competing technologies. Finally, a vision of future research and development activities required for full implementation of this technology is discussed.

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Forming of Engineered Prepregs and Reinforced Thermoplastics: *Michael John Clifford*[1]; Andrew C. Long[1]; Patrick de Luca[2]; [1]The University of Nottingham, Sch. of Mech., Matls. Manufact. Eng. and Mgmt., University Park, Nottingham NG7 2RD UK; [2]ESI International, 20 Rue Saarinnen-SILIC 270, Rungis, Cedex F-94578 France

Composite materials, such as engineered prepregs and reinforced thermoplastics, have found widespread use in the aerospace industry. In addition, the recent development of low-cost materials suitable for medium/high volume production (e.g. commingled glass/polypropylene fabrics) has attracted much interest from the automotive sector. Manufacturing high-performance engineering components from sheet material requires careful design. For sheet metals this problem is usually addressed using numerical simulations. Therefore a pragmatic approach for composite sheet forming is to modify existing packages developed for metal stamping to take account of the complex rheology of composites. In this paper, attention is focused on modelling the various mechanisms that occur during forming of composite sheets. In particular, a model is proposed for the dominant mechanism, intraply shear, based on the matrix rheology and the fibre architecture. The results are used to model composite sheet forming using an explicit non-linear FE code (PAM-FORM).

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Characterization of Textile Composite Behavior: Experiments and Simulations: *Jian Cao*[1]; Xiongqi Peng[1]; *Julie Chen*[2]; Darin Lussier[2]; [1]Northwestern University, Dept. of Mech. Eng., 2145 Sheridan Rd., Evanston, IL 60208 USA; [2]University of Massachusetts-Lowell, Dept. of Mech. Eng., Lowell, MA 01854 USA

Textile composite materials possess superior mechanical properties, such as high specific-strength and high specific-stiffness, and great material architecture flexibility. It is essential to understand the mechanical behavior of textile composites during processing so that optimal design of products and manufacturing processes using these materials can be achieved. This paper presents experimental data using a shear frame test and a numerical procedure for obtaining the effective nonlinear elastic moduli of textile composites during forming. The objective is to develop an efficient material model that considers the microstructure of the textile composite, and can be implemented in the numerical simulation of the stamping process. In this model, the woven fabric is assumed to be nonlinear orthotropic and in a plane stress state. The comparisons between experiments of a forming test and numerical simulations will be presented.

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Consolidation During Non-Isothermal Stamp Forming of Carbon/PA12 Composites: *M. D. Wakeman*[1]; L. Zingraff[1]; P-E. Bourban[1]; J-A. E. Manson[1]; Patrick Blanchard[2]; [1]Swiss Federal Institute of Technology (EPFL), Composites and Polymer Technology Laboratory (LTC), Science Park Bldg. A (PSE-A), CH-1015 Lausanne, Switzerland; [2]Ford Motor Company, Ford Res. Lab., 2101 Village Rd., Mail Drop 3135, Dearborn, MI 48121 USA

Stamp forming of thermoplastic composite materials offers the potential for rapid processing of shell-like components. The highly non-isothermal process is suited to a variety of thermoplastic composite precursor materials, ranging from dry unconsolidated commingled yarns to fully impregnated products. Initial void contents have a strong effect on the subsequent stamping process whereby the consolidation accompanying the deformation and heat transfer processes during stamping is reduced with higher impregnation levels. Reduced void contents in the final product increase mechanical properties and are related to the initial material form. To investigate these phenomena, carbon fibre weave reinforced polyamide 12 sheets, in two material forms, have been studied. A commingled yarn system, with varying degrees of preconsolidation, and fully impregnated sheets produced by the impregnation of carbon weaves with anionically polymerized laurolactam have been examined. The void content during the stamping process has been mapped experimentally and compared to a non-isothermal consolidation simulation to show the importance of the initial material consolidation quality on the properties of the final stamped component.

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Integrated Virtual Prototyping for Composite Design, Analysis and Manufacturing: *Olivier Guillermin*[1]; [1]Composite Design Technologies, 486 Totten Pond Rd., Waltham, MA 02451 USA

Tight interaction between design, analysis and manufacturing is required to engineer a composite part. In most cases, the final part contains details and modifications that create significant differences between the design model, the analysis model, and the manufactured part. For example, dramatic changes in fiber orientation can occur which induce wrinkling and large thickness changes. Or ply drop-offs and staggers may change the laminate stack-up symmetry and balance. All of these issues can have a considerable effect on the performance of the part. This paper describes how the FiberSIM suite of CAD integrated software tools for composites can be used to address these issues. For the first time, the link between CAD software, analysis packages, and manufacturing equipment makes it possible for designers, analysts and manufacturing engineers to access the CAD master model of a composite part in its to-be-manu-

factured state. Engineers can verify at any time during the design and manufacturing process that the part meets the performance specifications. Practical case studies from industry will highlight how composite engineering is improved and risk is reduced through the use of a complete and detailed virtual prototype.

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Evolution and Management of Process-Induced Damage in Polymer Composite Panels: *A. Sherif El-Gizawy*[1]; *Yean-Der Kuan*[1]; [1]University of Missouri-Columbia, Mech. and Aero. Eng., E3412 Eng. Bldg. E., Columbia, MO 65211 USA

A numerical model describing the evolution of process-induced damage in molded composite panels with woven fiber mats was developed. Part warpage (deformation), and residual stresses developed inside the panels are the damage types considered in this work. The effects of thermo-mechanical and thermo-chemical responses of the material on the evolution of damage during resin transfer molding of the panels are quantified. This numerical model in conjunction with an optimization module based on Simulated Annealing (SA) technique are used in conducting parametric design analysis for minimization of process-induced damage in composite panels. Experimental case studies utilizing epoxy resin with eight harness graphite fiber mats are used to verify the numerical model. Damage predictions from the present model are in agreement with the experimental measurements. The parametric analysis conducted on rectangle panels using the developed system indicates that high cure temperature, low heating rate and low rate of cooling after cure would minimize warpage and residual stresses.

Teaching and Learning Solid State Diffusion - Panel Discussion

Sponsored by: ASM International: Materials Science Critical Technology Sector, Atomic Transport Committee
Program Organizers: Richard D. Sisson, Worcester Polytechnic Institute, Materials Science & Engineering, Worcester, MA 01609 USA; Joe I. Goldstein, University of Massachusetts, Department of Engineering, Amherst, MA 01020 USA; John Morral, University of Connecticut, Department of Metallurgy, Storrs, CT 6260 USA

Wednesday PM Room: 202
February 14, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Richard D. Sisson, Worcester Polytechnic Institute, Mats. Sci. and Eng., Worcester, Massachusetts 01609 USA; John Morral, University of Connecticut, Dept. of Metall. and Mats. Eng., Storrs, CT USA

2:00 PM

Teaching and Learning Diffusion and Other Rate Processes: *Richard D. Sisson*[1]; M. M. Makhlouf[1]; [1]Worcester Polytechnic Institute, Mats. Sci. and Eng. Dept., 100 Institute Rd., Worcester, MA 01609 USA

Learning the fundamentals of diffusion in solids, conduction heat transfer and other dynamic processes is sometimes difficult for undergraduate and graduate students. The traditional teaching method of lectures emphasizing the solution to the Fick's or Fourier's Laws with a variety of boundary conditions does not address the learning styles of many students. In this presentation alternative teaching methods will be presented that may help students with a variety of learning styles learn the fundamentals of solid state diffusion. The incorporation of more active learning and team projects will also be presented and discussed.

2:25 PM Panel Discussion

Following the Opening Presentation, the AM Presenters will Form a Panel for an Open Ended Discussion on Teaching and Learning Diffusion.

NOTES

PLANT TOUR
NASA Michoud Assembly Facility
9:00 AM - 12:00 PM
Outside Ernest N. Morial Convention Center –
In Front of Hall A
★★★

Aluminum Joining-Emphasizing Laser and Friction Stir Welding:
Session 4 - Friction Stir Welding-Application and Performance

Sponsored by: Light Metals Division, Aluminum Association
Program Organizers: John A.S. Green, The Aluminum Association, Washington, DC 20006-2168 USA; J. Gilbert Kaufman, Columbus, OH 43220-4821 USA; Thomas J. Lienert, Edison Welding Institute, Columbus, OH 43221-3585 USA

Thursday AM Room: 214
February 15, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Thomas J. Lienert, Edison Welding Institute, Columbus, OH 43221-3585 USA

8:30 AM Keynote
A Comparison Between Microstructure, Properties and Toughness Behavior of Power Beam and Friction Stir Welds in Al-Alloys: *Jorge F. dos Santos*[1]; Gürel Çam[1]; Alexander von Strombeck[1]; Volker Ventzke[1]; Mustafa Koçak[1]; [1]GKSS Forschungszentrum, Instit. for Mats. Rsch., Join. Tech., Max-Planck-Str, Geesthacht D-21502 Germany

Power beam welding (LBW and EBW) and friction stir welding (FSW) processes can be successfully used to achieve defect-free joints in Al-alloys. However, a thorough characterisation of the joints is needed in order to satisfy the stringent requirements of advanced applications such us aerospace, automotive and shipbuilding. In this work, LBW, EBW and FSW were performed on four different aluminium alloys, namely 5005-H14, 2024-T351, 6061-T6, and 7020-T6 (plate thickness being 5 mm except alloy 5005, which is 3 mm thick). The main objective was to establish the local microstructure-property relationships and to determine the fracture toughness levels of welded plates with weld zone strength undermatching. Microstructural characterisation of the weld metals was performed by optical and scanning electron microscopy. A very low level of porosity was observed in all LBW and EBW welds. This has been attributed to careful surface cleaning prior to welding and the welding in vacuum as required by the EBW process. All FSW welds were void and crack free. Extensive microhardness measurements were conducted in the weld regions of the joints. Global and local (microflat specimens) tensile properties and fracture toughness properties (in terms of CTOD) of the power beam and FSW joints were determined at room temperature. The effects of strength mismatch and local microstructure on fracture toughness of the joints have been considered in the analysis of the results. Due to the reduction in yield strength in the FZ of power beam and FSW joints, higher fracture toughness values were obtained at this test location than in the respective base materials. The confined plasticity (increased constrain) within the fusion zone did not deteriorate toughness because apparently the inherent properties of the obtained microstructure is still higher than that of the base material. Hence, it can be concluded that the strength undermatching observed on both power beam and FSW joints did not generally lead to a degradation of toughness properties.

9:00 AM Invited
Mixed Mode Tearing Behavior of Aluminum FSW Joints: *Michael A. Sutton*[1]; [1]University of South Carolina, Dept. of Mech. Eng., 300 Main St., Rm. A129, Columbia, SC 29208 USA

Friction stir welded, 6.35 mm thick, 2024-T3 aluminum components, with initial fatigue cracks oriented along the FSW, are subjected to combined mixed mode I/II loading in an Arcan test fixture. Crack tip conditions ranging from pure tension to pure shear are applied to the specimen up to the initiation and stable growth of the flaw. Results from the studies including (a) crack paths, (b) load-crack extension data, (c) SEM photographs of fracture surface and (d) the microstructure in the FSW along the observed crack growth path prior to crack growth.

9:30 AM Invited
The Use of Friction Stir Welding Technology in Maritime Applications: *Rollin E. Collins, II*[1]; [1]High Tech Welding, 263 McLaws Circle, Ste. 203, Williamsburg, VA 23185 USA

Friction Stir Welding has been used successfully to produce large panels for the shipbuilding industry. The recent development of truly high speed vessels has driven the builders and ship owners to search for solutions to reduce overall weight while maintaining strength and at the same time optimize fabrication techniques to reduce construction time and lower production costs. This presentation will focus on experience to date with Friction Stir Welding showing examples of different panel solutions that have been produced and the many benefits derived from using such modular elements. Finally, the critical interaction between optomized designs, the proper material selection and the fabrication processes will be discussed.

10:00 AM Break

10:15 AM
FSW in the Automotive Industry: *Christopher B. Smith*[1]; [1]Tower Automotive, Adv. Techn., 3533 N. 27th St., Milwaukee, WI 53216 USA

One of the challenges with the implementation of aluminum into automotive structures is the relative difficulty of joining aluminum as compared to steel. Techniques for joining steel (e.g. GMAW, RSW, etc.) are also applicable to joining aluminum, but their controllability in a high production environment is much more difficult in the case of aluminum. Thus, there exists a need for alternative joining process for aluminum, for which friction stir welding (FSW) offers a possible opportunity to fill. With all of its advantages, FSW has an enormous potential for use in the automotive industry. However from its inception it had several limitations, which did not make the process justifiable. These limitations, their current status, and the research towards overcoming them will be discussed, as well as the automotive applications with the most potential for use of FSW.

10:45 AM
Joining Dissimilar Aluminum Alloys and Other Metals and Alloys by Friction-Stir Welding: *Lawrence E. Murr*[1]; R. D. Flores[1]; F. Contreras[1]; M. Guerra[1]; D. J. Shindo[1]; M. Siddiqua[1]; H. S. Kazi[1]; C. Schmidt[1]; J. C. McClure[1]; [1]University of Texas at El Paso, Metall. and Matls. Eng. M-201, 500 W. University Ave., El Paso, TX 79968-0520 USA

A wide range of aluminum alloys, particularly dissimilar alloy systems, have been friction-stir welded. These include Al 2024/Al 6061, Al 5052/Al 7075, Al 7075/Al 2017, Al 5052/Al 2017, Al 2195/Al 2024, and Al 2024/Ag, Al 2024/70/30 brass, etc. In all but the last two dissimilar weld systems the weld zone is uniformly dynamically recrystallized, and though there is a very complex and non-uniform mixing and flow of the components forming intercalated, lamellar-like microstructures. There is often a 40% loss of strength for these age-hardenable alloys even though the weld integrity is flawless. The mixing and weld zone microstructures are different for Al 2024/Ag and Al 2024/brass although dynamic recrystallization also plays a dominant role. Weld zone microstructures have been characterized extensively by optical metallography and transmission electron microscopy. The weld properties and microstructures are altered somewhat with tool rotation and traverse speeds

(actual weld speed) and these issues will be discussed. Some recent examples of dilute Sc(0.1%)-Al alloy joining by friction-stir welding will be illustrated. There appear to be very few aluminum alloys which cannot be successfully joined by friction-stir welding although complex, PM systems such as Al-Be alloys pose some particular challenges. Research supported by a General Services Administration (GSA) Grant PF-90-018 and NASA MURED Grant NAG8-1645.

11:15 AM

Corrosion-Fatigue Crack Propagation in Friction Stir Welded Al 7050: *Peter S. Pao*[1]; Steve J. Gill[1]; Jerry C.R. Feng[1]; K. K. Sankaran[2]; [1]Naval Research Laboratory, Code 6323, 4555 Overlook Ave. S.W., Washington, DC 20375-5320 USA; [2]The Boeing Company, St. Louis, MO 63166 USA

The corrosion-fatigue crack propagation of weld, HAZ, and base metal of friction stir welded Al 7050 were investigated. Fatigue crack growth rates in the weld in air and in 3.5% NaCl solution are slightly higher than those in the base metal. Fatigue crack growth rates in salt water are two to three times higher than those in air at high and intermediate stress intensities. Fatigue crack growth rates are significantly lower and fatigue crack growth thresholds are substantially higher in the HAZ than in the weld and base metal in both salt water and air. Post fatigue fractographic examinations revealed a variety of fracture modes such as intergranular separation in the recrystallized weld nugget in salt water and cleavage-like fracture in base metal in air. The observed corrosion-fatigue crack growth kinetics in the weld, HAZ, and base metal are discussed in terms of microstructural variations, environmental interactions, and crack closure.

Aluminum Reduction Technology: Soderberg and Prebake Cell Operation

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

Thursday AM
February 15, 2001
Room: 206-207
Location: Ernest N. Morial Convention Center

Session Chair: Knut Arne Paulsen, Hydro Aluminium Karmoy, Håvik, Karmøy N-4265 Norway

8:30 AM Invited

The Soderberg Cell Technology–Present Performance, Challenges and Possibilities: *Tor Bjarne Pedersen*[1]; [1]Elkem Aluminium Lista, P.O. Box 128, Farsund 4551 Norway

Søderberg Cell Technology has in general had inferior performance compared to modern PB-cells. The technology is also facing new and challenging environmental requirements. However, existing Søderberg cells may yield substantially better and satisfactory results with relatively small capital investments and alteration of operation procedures. Through systematic innovation Elkem Aluminium Lista has demonstrated that it is possible to meet coming new European environmental requirements, produce metal with high quality and at the same time increase productivity. For Lista, being a pure Søderberg smelter, this is far more economic than converting to PB-technology. The Søderberg technology has cost advantages since there is no need for rin furnace or rodding facility. Development work is going on at several locations and through gaining thorough process knowledge the technology will improve further in the coming years. This will make even small Søderberg plantscompetitive compared to modern PB-plants.

9:00 AM

Pilot Plant Operation of Russian VSS-Pots in Excess of 160 kA Following Magnetic Improvements, Installation of Pointfeeders and Process Control Systems: *T. K. Nasipuri*[1]; H. L. Stephen[1]; H. O. Bohner[1]; [1]HART, Singenbergstr. 18, St. Gallen CH-9003 Switzerland

On a total of 10 Test and 2 Buffer pots out of a line of 192 pots busbars were improved to various degrees on the basis of a model by Technology Center Chippis (TCC) of Alusuisse. Each of the pots was fitted with single-piston pointfeeders to add alumina and aluminium fluoride inside of the skirt. The main process parameters such as resistance, instability (noise), alumina and aluminium fluoride-content, skirt adjustment, beam raising, AE prediction and AE frequency as well as adaption of set resistance to stud pulling, tapping and AE voltage were process-controlled by hardware supplied by Hamilton Research & Technology (Hart), Calcutta and by software designed and developed for VSS pots by the the authors. Production per potday through higher current intensity and current efficiency, electric power and paste consumption, all improved to various degrees dependent upon the magnetic improvement of the busbar system as expressed as standard deviation of cathodic current distribution. Performance figures over a six-month test period, corrected for metal pad inventory changes are presented and discussed.

9:25 AM

Reduction of Anode Effect Frequency and Duration at VSS Line Operating with Heavy Power Modulation: *André Luiz Lopes Machado*[1]; Eliezer Duarte de Araújo[1]; João Bonfim Galvão Moraes[1]; [1]Alcan Aluminio do Brasil Ltda., Aratu Plant-Prim. Grp., Cx Postal 7391, Salvador, Bahia 41810-000 Brazil

Energy consumption is a representative factor in a Smelter operation cost, specially in Brazil. Because of high energy costs, big efforts have been carried out in order to reduce energy costs without affecting potline performance. The main operational change was the introduction of a very heavy power modulation 10 years ago, that resulted in high cost reduction, but caused a lot of fluctuation in the process. It's well known that anode effects are responsible for an important part of overall energy consumption, and also produce most of PFC's emissions in a potline. Therefore, an important tool to reduce energy consumption is to work on pot voltage control, with a special focus on anode effect detection and termination. In this paper we present our process development to reduce anode effect frequency and duration at Aratu's VSS Potline and some results of the implementation.

9:50 AM

3-D Mathematical Model for Studying Changes in Self-Baking Anode Power Regime: *S. A. Shcherbinin*[1]; V. V. Pingin[1]; V. K. Frizorger[1]; [1]PSC Krasnoyarsk Aluminum Plant, Krasnoyarsk 660111 Russia

The self-baking anodes of aluminum reduction cells undergo continuos changes of their power regime in operation. These variations of dynamic conditions are basically driven by varying anode studs positions and changed anode operating properties. A 3-D mathematical model was used in order to establish optimum operating parameters because it allowed to run evaluations of thermal and electrical fields for the whole cell. The uniqueness of the present software lays in a possibility to run prompt calculations for different sets of cell dimensional and process parameters. It was the uniqueness of the newly developed program which supported the performance of calculations and results evaluation for changing anode parameters. A developed in the package post-processor supports visualization of dynamic changes of anode power regime, in particular it allowed to run a comparison between different stud patterns and stud setting schedules. Dependencies were found based on the calculation results for changing cell thermal and electrical fields and optimization methods were established.

10:15 AM

Pot Life Prediction of Aluminium Soderberg Cells: M. A. Doheim[2]; S. M. El-Raghy[3]; *Mohamed A. Ali*[1]; [1]The Aluminum Company of Egypt, Nage-Hammadi Egypt; [2]Assiut University, Fac. of Eng.; [3]Cairo University, Fac. of Eng.

Factors affecting pot life has been identified as: thermal design, construction materials, construction practices, and cathode shell design, preheating, start-up and general operation of the cell. The

actual pot life statistics records at Aluminium Company of Egypt (Soderberg cells), for the period 1980 till 2000 analyzed. Though the average Soderberg pot life over this period was 57 months, yet, there were cells with few months' life. More than one cell over 100 months. An empirical correlation for pot life prediction was developed using the three-parameter Weibull cumulative distribution. The statistical model was tested for different carbon lining suppliers. Good agreement was found between the model and actual life after excluding the early and accidental failures. This paper indicates the relative weight of parameters to be considered for longer pot life.

10:40 AM Break

10:50 AM

Aluminum Fluoride Dissolution and Distribution-An Investigation of the Dynamic Mass Balance When Adding Large Quantities to a Prebake Cell: *Ketil Å. Rye*[1]; Ingar Solberg[1]; Trygve Eidet[2]; Sverre Rolseth[3]; [1]Elkem Aluminium ANS, P.O. Box 566, Mosjøen N-8650 Norway; [2]Elkem Research, P.O. Box 8040 Vågsbygs, Kristiansand N-4602 Norway; [3]SINTEF Materials Technology, Trondheim N-7465 Norway

The dynamic behavior of aluminium fluoride in Hall-Heroult cells is not readily understood, as the AlF3 mass balance sometimes gets out of control. This is manifested as "inactive"cells, where the AlF3 content stays nearly constant for days in spite of efforts to change the situation. So far there appears to be no comprehensive theory that can explain all unaccountable observations associated with such cells. In order to throw some light on this problem, investigations were carried out to check the AlF3 mass balance when adding large quantities of aluminium fluoride to normal cells. In spite of some uncertainty associated with the determination of the volume of molten bath in the cells, it was apparent that not all of the aluminium fluoride added dissolved rapidly. Frequent measurement of the side ledge thickness after the AlF3 addition revealed only minor changes, which could not account for the "missing" aluminium fluoride.

11:15 AM

Potroom Operations and Their Impact on Anode Spike Formation: *Neal Wai-Poi*[1]; Richard Jansen[2]; Bernd Rolofs[1]; [1]Corus Aluminium Voerde GmbH, SchleusenstraBe 11, Voerde D-46562 Germany; [2]Corus Research, Development and Technology, IJmuiden Tech. Ctr., P.O. Box 1000, IJmuiden 1970 CA, The Netherlands

Since 1992, the Voerde smelter has experienced a continuing problem with anode spikes, which culminated earlier this year with over 1100 spikes in one month. The anode spike problem is a result of persistent carbon dust problem in the cells. Last year, the results from a study investigating the influence of operational practices on anode spike formation and the negative impact anode spikes have on potroom performance were presented. Since then, further studies investigating the factors which influence anode spike formation and how operational practices can be modified to minimize their formation have been conducted. Changes to operational practices have included a new energy management strategy and removal of carbon dust from the anode cover. These changes have resulted in a reduction of over 60% in anode spikes. However, despite the dramatic reduction, the results show the only sustainable solution to permanently eradicate spikes at Voerde is to reduce the carbon dust levels, including an improvement in baked anode quality.

11:40 AM

Sludge in Operating Aluminium Smelting Cells: *Pierre Yves Geay*[1]; B. J. Welch[1]; P. Homsi[1]; [1]Aluminium Pechiney LRF, B.P. 114, St-Jean-de-Maurienne, Cedex 73303 France

There has been growing concern on cathode wear in modern aluminium smelting cells, with doubts existing on the mechanistic causes. The dissolution of carbide has been shown to be an important factor, but there have been suggestions that abrasive wear is also a major contributor. The main abrasive in contact with the cathodic blocks is the sludge but, to cause wear, it would have to have considerable mobility. Sludge also introduces a resistance to the current flow path since it has been shown that on some occasions it covers significant fractions of cathode surfaces. In this investigation, the first stage was to take representative sludge samples and accurately characterize them by XRF, XRD, SEM-EDX and other chemical analytical methods. New phases have been identified and the sludge eutectic temperature has been more precisely defined. The second stage of the project was carried out to ascertain the location of sludge and its general mobility. The sludge level in pots was found to be very erratic but statistically, its average profile showed a correlation with the cathode wear pattern. Separate laboratory experiments demonstrate that the electrical conductivity of sludge is typically half that of normal electrolyte.

12:05 PM

The Behaviour of Phosphorus Impurities in Aluminum Electrolysis Cells: *Elin Haugland*[1]; Geir Martin Haarberg[2]; Elke Thisted[2]; Jomar Thonstad[2]; [1]Hydro Aluminium a.s., Tech. Ctr. Ardal, P.O. Box 303, Ovre, Ardal N-6884 Norway; [2]Norwegian University of Science and Technology, Dept. of Mats. Tech. and Electrochem., Sem Saelandsvei 6, Trondheim N-7491 Norway

Phosphorus is an important impurity element in the Hall-Heroult process, where it affects the current efficiency and the quality of the aluminium produced. The chemical and electrochemical behaviour of phosphorus compounds in molten cryolite-based electrolytes was studied in controlled laboratory experiments. Measurements were also carried out in industrial cells by analysis of electrolyte and metal samples as a function of time after additions of phosphorus compounds to the electrolyte. Phosphorus has a relatively long residence time in the electrolyte due to cyclic oxidation and reduction reactions at the electrodes. Eventually, phosphorus escapes from the industrial cells through evaporation of elemental phosphorus or as a phosphorus compound together with small carbon particles (carbon dust). Mass balance studies show that the phosphorus concentration in the metal depends on both the phosphorus content in the raw materials and on the cell operation. A relatively small increase in the normal operating temperature was found to cause a decrease in the amount of phosphorus in aluminium.

Cast Shop Technology: Solidification & Foundry Technology

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

Thursday AM — Room: 208-210
February 15, 2001 — Location: Ernest N. Morial Convention Center

Session Chair: Peggy Jones, General Motors Corporation, Powertrain, Saginaw, MI 48605-5073 USA

8:30 AM

High Quality Magnesium Castings by the New Rheocasting (NRC) Approach: *Helmut Kaufmann*[1]; Peter J. Uggowitzer[2]; [1]Leichtmetall Kompetenzzentrum Ranshofen, Ranshofen, Upper Austria 5282 Austria; [2]ETH Zurich, Institute for Metallurgy, Sonneggstrasse 3, Zurich CH-8092 Switzerland

Metallurgical and processing aspects of New Rheocasting (NRC) are discussed in terms of microstructural analysis of the slurry and the NRC castings, and in terms of mechanical characterisation. The basic concept of NRC is explained and possible production equipment is introduced. The process is based on controlled cooling of a slightly superheated melt. This controlled cooling is performed in a carousel slurry maker. The semi-solid slugs are then cast to shape in a Vertical Squeeze Casting machine. Special focus is put on the requirements on Mg alloys for semi-solid forming, and it will be shown that proper alloy design can stabilize the process and improve reproducability of the casting properties. Magnesium casting and wrought alloys will be checked for semi-solid castability, and trial results will be presented. The results are compared with characteristic properties of High Pressure Die cast magnesium, Squeeze Castings and Thixomolding parts. An innovative recycling concept

for low cost re-use of globular semi-solid runners and scrap will be presented.

8:55 AM

Prediction of Mechanical Properties of As-Cast and Heat-Treated Automotive Al Alloys Using Artificial Neural Networks: *Daryoush Emadi*[1]; Mahi Sahoo[1]; Terri Castles[1]; Hekmat Alighanbari[2]; [1]CANMET, Mats. Techn. Lab., 568 Booth St., Ottawa, Ontario K1A 0G1 Canada; [2]Ryerson Polytechnic University, Mech./Aerosp. Eng., 350 Victoria St., Toronto, Ontario M5B 2K3 Canada

The desired mechanical properties of cast automotive aluminium alloys depend on the heat treatment conditions, alloy chemistry and casting parameters such as cooling rate, mould design and melt treatment. Despite extensive work in the literature, the large number of these controlling parameters have made it difficult to predict the mechanical properties and to model them using conventional techniques. In the present study, we used an alternative method, namely Artificial Neural Networks (ANN), to predict the mechanical properties. A database of mechanical properties (UTS, YS and EI%) as a function of chemical composition, heat treatment (solutionizing, quenching and ageing) and casting variables (mould type and melt treatment) was established based on published literature. Several standard multi-layer ANN models were then trained using data randomly selected from the database. The outputs of the ANN models were subsequently compared with the remaining data. The results indicate that ANN is a suitable modelling technique for prediction of mechanical properties and optimising the heat treatment process.

9:20 AM

Latent Heat Evolution During Solidification of Aluminum Based Alloys: *Carlos Enrique Schvezov*[1]; Alicia Ester Ares[1]; Sergio Fabian Gueijman[1]; [1]University of Misiones, Fac. of Sci., 1552 Azara, Posadas, Misiones 3300 Argentina

It has recently been presented a thermodynamic model for the calculation of the latent heat during solidification modeling with emphasis on its evolution in the mushy zone. The calculations were applied to Lead-Tin alloys and the results were in good agreement with experimental data. These calculations are now extended to binary aluminum alloys such as Al-Cu, Al-Mg and Al-Zn in a range of concentrations. The results show similar behavior of the latent heat as observed in Lead-Tin alloys that is, with a larger amount of latent heat being released at the beginning of the alloy solidification. In the present report these results are presented and discussed.

9:45 AM

Microstructure and Microsegregation in a Directionally Solidified Quaternary Al-Rich Al-Cu-Mg-Zn Alloy: *Fanyou Xie*[1]; Xinyan Yan[1]; Men Chu[2]; Y. Austin Chang[1]; [1]University of Wisconsin-Madison, Mats. Sci. and Eng., 1509 University Ave., Madison, WI 53706 USA; [2]Alcoa, Inc., Alcoa Technical Center, Alcoa Center, PA 15069 USA

Microstructure and microsegregation in a directionally solidified quaternary Al-3.86Cu-0.89Mg-0.99Zn alloy with cooling rates of 2, 0.78 and 0.23 K/s were studied. The solidification microstructures of the quaternary alloy were dendritic, similar to those of Al-rich Al-Cu-Mg alloys solidified at the same conditions in an earlier study. Both the fractions of solid formed and the solute concentration gradients in the dendrites were studied. While the volume fractions of solid formed were measured by image analysis, the solute concentration gradients within the dendrites were determined by an area scan technique. The fractions of solid formed were also obtained from the concentration profiles, which were in agreement with those fractions obtained by image analysis. The model-calculated fractions of solid and the concentration gradients were in reasonable accord with the measured data. The model used was a modified Scheil that includes back diffusion in the solid, dendrite arm coarsening and effect of undercooling.

10:10 AM Break

10:20 AM

Predicting Microstructure and Microsegregation in Multicomponent Aluminum Alloys: *Xinyan Yan*[1]; Ling Ding[1]; *Fanyou Xie*[1]; Y. Austin Chang[1]; [1]University of Wisconsin-Madison, Dept. of Mats. Sci. and Eng., 1509 University Ave., Madison, WI 53706 USA

Accurate predictions of microstructure and microsegregation in metallic alloys are highly important for applications such as alloy design and process optimization. Restricted assumptions concerning the phase diagram could easily lead to erroneous predictions. The best and most accurate method to treat the phase equilibrium at the interface is coupling the microsegregation calculation with phase diagram calculations according to the CALPHAD method. A newly developed numerical model for the prediction of microstructure and microsegregation in multicomponent alloys during dendritical solidification was introduced. The micromodel was directly coupled with phase diagram calculations using a user-friendly and robust phase diagram calculation engine-PANDAT. Solid back diffusion, undercooling and coarsening effects were included in this model, and the experimentally measured cooling curves were used as the inputs to carry out the microsegregation calculations. This model was used to predict the microstructure and microsegregation in some commercial aluminum alloys, such as 2024 and 7050. Microstructure and microsegregation in those multicomponent aluminum alloys were experimental investigated using directional solidification and electron probe microanalysis (EPMA). Calculated results using this model are in accord with the experimental data, while those results using Scheil model deviate significantly from the experimental data.

10:45 AM

Thermal Modeling of Ingot Chain Productivity: *Alain Chauvineau*[1]; Loic Maenner[1]; Serge Guy[1]; *Stephane Morency*[2]; Chantal Sztur[1]; [1]Pechiney, Aluminium Pechiney, 725 Aristide Berges BP n°7, Voreppe Cedex 38341 France; [2]University Laval, Dept. Min. and Metall., Ste-Foy, Québec G1K 7P4 Canada

Ingot chains casthouses are generally confronted with classical problems of productivity and reliability, sometimes difficult to analyze. Pechiney has developed a thermal model adapted to ingot chains in order to quantify the influence of the main process parameters. The PAMCAST /SIMULOR® software, dedicated to foundry casting, was used. The model utilizes industrial measurements and heat transfer coefficients determined in the laboratory. Among the many parameters explored with the model, the calculations show that the main parameters governing the productivity of the ingot chain are the water/mold heat transfer and the coating of the molds. The model is and will be used as a tool for optimizing present chains as well as for designing future generation ingot chains.

11:10 AM

The Response of Twin Roll Cast Aluminium Strips to Thermo-Mechanical Processing: *Yucel Birol*[1]; Murat Dundar[2]; Osman Cakir[1]; A. Soner Akkurt[2]; Shaun Hamer[3]; Chris Romanowski[3]; [1]MCTRI, Marmara Research Center, P.O. Box 21, Kocaeli, Gebze 41470 Turkey; [2]Assan Aluminum Works, E-5 Karayolu, 32 Km., Tuzla, Istanbul 81700 Turkey; [3]FATA Hunter, Inc., P.O. Box 5677, 6147 River Crest Dr., Riverside, CA 92507-0745 USA

The recent trend in Twin-Roll Casting has been to reduce the casting gauge to less than 3mm in order to reduce the processing costs, to take advantage of increasing rates of solidification and to increase productivity. To fully utilize the potential of their thin gauge and wide strip caster investment, ASSAN Aluminum, in cooperation with FATA Hunter and Marmara Research Center, has recently initiated an extensive R&D program to investigate the effect of casting parameters such as the casting gauge, the casting speed and tip setback on the metallurgical quality of the cast strip. 81 different samples covering a range of casting parameters for three common foil alloys, AA8011, AA8006 and AA1200, were cast on industrial scale with a 1725mm wide SpeedCaster. The effect of casting parameters on the macrostructural features of twin roll cast strips was investigated in the first phase of this program and was recently reported. This paper describes the response of the thin-gauge wide cast strips to thermo-mechanical processing with a particular emphasis on the effect of casting gauge on homogenization cycles.

Cast Shop Technology: Molten Metal Behaviour & Properties

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

Thursday AM Room: 224
February 15, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: M. M. Makhlouf, Worcester Polytechnic Institute, Mats. Sci. and Eng. Dept., Worcester, MA 01609 USA; George Ferguson, Auckland University, Auckland, New Zeland

8:30 AM
Effect of Ti and Zr on The Fluidity of High Strength Al-Alloys: *Young Dong Kwon*[1]; Zin Hyoung Lee[1]; [1]Korea Advanced Institute of Science and Technology, Mats. Sci. and Eng., Gusong-dong 373-1, Yusong-gu, Taejon 305-701 Korea

Ti and Zr are used as minor alloying elements and for grain refinement for high strength Al-alloys and they are known to have adverse effect on the fluidity of the alloy. The weight reduction is achieved by using high strength alloys and reducing the wall thickness. The fluidity is important to cast thin parts. The fluidity of a base alloy, Al-4.8%Cu-0.6%Mn, was measure by casting a multi-channel probe, which was proved to show a reproducible results. The results were compared with those of the well known casting alloy, A356. Initial content of Ti and Zr was varied from 0 to 0.3wt% and 0.05% respectively and Al-5Ti-B master alloy was added for grain refining. The average flow length varied linearly with the superheat of the pouring temperature. The Fluidity depended on the initial amount of Ti and Zr and the addition of the grain refiner.

8:55 AM
A New Technique to Dynamically Measure Surface Tension, Viscosity and Density of Molten Metals: *Steven John Roach*[1]; Craig Owens[1]; Hani Henein[1]; [1]University of Alberta, Adv. Mat. and Proc. Lab., 536 Chem./Min. Eng. Bldg., Alberta T6G 2G6 Canada

In many high temperature applications, knowledge of physical properties of a melt (e.g. surface tension, viscosity and density) is lacking in the literature. A new technique has been developed for measuring surface tension, viscosity and density based on flow of a melt draining from a crucible. Flowrate of a fluid stream through an orifice is not characterized by viscous losses alone; surface energy plays a significant role in fluid flow as well. A mathemeatical model based on an energy balance was developed to describe flow-rate of a melt stream through an orifice. By proper calibration of the crucible system, the surface tension, viscosity and density of the fluid can be measured as a function of processing variables such as temperature and atmosphere. The validation of the model will be discussed, and results with molten aluminum and magnesium will be presented.

9:20 AM
Measurements of Diffusion Coefficients in Molten Aluminum: *Ingeborg Birgitte Solheim*[1]; [1]SINTEF Materials Technology, Proc. Metall. and Ceram., Alfred Getz vei 2b, 7465 Trondheim, Norway

Diffusion-coefficients are important parameters in mathematical modeling of solidification and refining processes. The model's accuracy is naturally dependent on the input-data. Data found in the literature is often old and inconsistent -differing with each other. To improve foundry competence, a project in cooperation with industry-partners has been carried out. Diffusion coefficients in molten aluminum have successfully been measured by the capillary-reservoir-method. Three different alloys were tested; 99% aluminum with 1% Si, Mg and Mn, respectively. The measurements are compared with data from the literature, and a theoretical model for calculation of the diffusion coefficient. The diffusion-coefficients' dependence on temperature is also studied, along with possibility of numerically solving Fick's law. This work is still in progress.

9:45 AM
Capillary Phenomena During Filling of Fine Mold Cavities: *Jon L. Hilden*[1]; Kevin P. Trumble[1]; [1]Purdue University, Mats. Eng., 1289 Mats. and Elect. Eng. Bldg., West Lafayette, IN 47907-1289 USA

As higher demands are placed on metal casting technology, molten metal is often required to fill smaller, more intricate features in the mold. As the feature size becomes smaller, the effects of surface tension/capillary pressure become significant. The objectives of this work were to determine the capillary pressure of liquid filling cylindrical mold cavities defined by relatively large mold particles. The process was modeled by considering infiltration of a cylinder lined with 20 close-packed spheres around the circumference. Capillary pressures were determined for various liquid contact angles using Surface Evolver numerical software. Capillary pressures were compared to those obtained for liquid in a smooth-walled capillary. Comparisons show that substantially higher pressures are required to fill a rough-walled cylinder than a smooth-walled cylinder, and that the filling pressure is practically insensitive to contact angle above ~120°. This work is supported by Howmet Research Corp.

Chemistry and Electrochemistry of Corrosion and Stress Corrosion: A Symposium Honoring the Contributions of R.W. Staehle: Corrosion and Stress Corrosion of Lightweight Alloys

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee, Jt. Nuclear Materials Committee
Program Organizer: Russell H. Jones, Battelle Pacific Northwest National Laboratory, Richland, WA 99352 USA

Thursday AM Room: 221
February 15, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: David J. Duquette, Rensselaer Polytechnic Institute, Mats. Sci. and Eng. Dept., Troy, NY 12180-3590 USA; Robert P. Wei, Lehigh University, Mech. Eng. and Mech. Dept., Bethlehem, PA 18015 USA

8:30 AM
Rate-Limiting Processes in Environmental Fatigue Crack Propagation in 7000-Series Aluminum Alloys: Zuhair Gasem[1]; *Richard P. Gangloff*[1]; [1]University of Virginia, Dept. of Mats. Sci. and Eng., SEAS-Thornton Hall, Charlottesville, VA 22904 USA

The objective of this research is to understand the mass transport and reaction processes that govern the kinetics of hydrogen-environment enhanced fatigue crack propagation in 7000-series aluminum alloys. For pure chloride solution, crack growth rate (da/dN) is rate limited by hydrogen (H) diffusion in the crack tip process zone with rapid surface reaction kinetics. Quantitative modeling is hindered by the uncertain crack surface concentration of H, hydrogen diffusion path through the microstructure, effect of stress and plastic strain on H transport, and critical amount of H required for embrittlement. Chromate addition to the bulk NaCl solution results in reduced da/dN, presumably due to a reaction-rate limitation of H production on the crack surface. As crack tip strain rate increases, chromate inhibition is eliminated by destabilization of the passive film. Hydrogen diffusion can limit water vapor enhanced fatigue in 7000-series alloys, as suggested by an empirical correlation between da/dN and a frequency-based exposure parameter. Modeling of the transition from environment-mass transport to surface reaction to H diffusion control of da/dN is hindered by the uncertain amounts of H produced on the crack-tip surface for water vapor and electrolytes.

9:00 AM
Measurement and Modeling of Crack Conditions during the Environment-Assisted Cracking of an Al-Zn-Mg-Cu Alloy: *Kevin R. Cooper*[1]; Robert G. Kelly[1]; [1]University of Virginia, Mats.

Sci. and Eng. Dept., 116 Engineer's Way, Charlottesville, VA 22904 USA

Although it is well known that Al-Zn-Mg-(Cu) alloys are susceptible to intergranular environment-assisted cracking (EAC) in some temper conditions, the relative contribution of hydrogen embrittlement and anodic dissolution (AD) to the crack advance process is uncertain. Modeling of measured crack potential distributions and chemistry can elucidate the role of AD to the EAC process. Micro-reference electrodes were used to make in situ measurements of the crack potential during Stage II cracking of peak-aged AA 7050. The crack tip potential was -0.80 V_{SCE} and independent of the applied potential over the range -0.80 to -0.30 V_{SCE}. Near-tip potential gradients (1 V/cm) facilitated steady-state concentration gradients; the acidified tip solution was concentrated in bulk solution anions and alloy components. Modeled crack potential distributions were strongly dependent on the crack tip opening and the presumed presence or absence of a resistive salt-film at the crack tip.

9:30 AM

Effect of Stress on Penetration of Intergranular Corrosion in Aluminum Alloys; Transition of IGC to IGSCC: Xiaodong Liu[1]; Weilong Zhang[1]; *Gerald S. Frankel*[1]; [1]The Ohio State University, Fontana Corr. Cen., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA

Recent work has shown that the kinetics of localized corrosion in AA2024 are strongly anisotropic. The anisotropy of kinetics was associated with an intergranular growth morphology; the slower rate of penetration in the short transverse direction (through plate thickness) compared to the longitudinal or long transverse direction was a result of a longer path length around the pancake-shaped grains in the wrought microstructure. These experiments were performed using the foil penetration method, in which the time for penetration of a pit or other form of localized corrosion through a thin foil sample is determined by wetting of paper pressed against the sample back side. In this work, the influence of applied stress on the penetration kinetics was studied. ASTM G49 constant strain load frames were modified to permit the use of foil samples and attachment of one side of the samples to an electrochemical cell. Uniaxial elastic strains of varying magnitude were applied perpendicular to the nominal growth direction. This approach allows for the development of a detailed understanding of the transition from intergranular corrosion to intergranular stress corrosion cracking.

10:00 AM

Quantification of Crack Wake Hydrogen Concentrations on Small Length Scales: *John Robert Scully*[1]; George A. Young[1]; Lisa M. Young[1]; Richard P. Gangloff[1]; [1]University of Virginia, Dept. of Mats. Sci. and Eng., 116 Engineer's Way, P.O. Box 400745, Charlottesville, VA 22904-4745 USA

A damaging role of hydrogen is postulated in the mechanism for environment-assisted cracking (EAC) of many engineering alloys. One barrier to advancing an understanding of hydrogen-induced EAC has been the overall difficulty in quantifying exact hydrogen concentrations at crack tips. Recently, crack tip hydrogen concentrations have been quantified directly using newer experimental techniques. A variety of probes such as focused ion beam/secondary mass spectroscopy, thermal desorption spectroscopy, and nuclear reaction analysis offer combinations of lateral and depth resolution, as well as great sensitivity to hydrogen concentration. In our studies, these techniques are used to examine crack wake hydrogen concentrations in a classical environment-assisted cracking system; a peak aged versus an overaged Al-Zn-Mg-Cu alloy. Parallel studies with hot humid air and chromate-inhibited chloride solutions reveal high crack wake hydrogen concentrations and steep hydrogen concentration-depth gradients. Substantial hydrogen uptake occurs in both EAC-resistant overaged material and peak aged material in hot, humid air. High hydrogen concentrations are only observed in EAC-susceptible tempers exposed to chromate-inhibited chloride solution. The implications of these findings to HEAC mechanisms are discussed.

10:30 AM

Corrosion and Stress Corrosion Cracking of Al-Mg Alloys with Sc and Ag Additions: *Clyde L. Briant*[1]; Zhengfu Wang[1]; Ping Wang[1]; *Sharvan Kumar*[1]; [1]Brown University, Div. of Eng., Box D, Providence, RI 02912 USA

This paper will report a study of the microstructural development and corrosion and stress corrosion cracking properties of Al-5Mg alloys that contain additions of Sc and Ag. The results show that the main effect of Sc additions on the microstructure is to retard recrystallization. Silver additions appear to stimulate the precipitation of beta phase along the grain boundaries. The stress corrosion results for tests performed in 3.5% NaCl solution show that scandium additions have no detrimental effects on stress corrosion cracking resistance. In contrast, silver additions enhance the corrosion rate. This enhancement is greatest in samples with an equiaxed grain structure. Scatter observed in the tests can also be attributed to the morphology of the grains and its relation to the fracture path.

11:00 AM

Stress Corrosion Cracking Mechanisms for Crack-Tip/Particle Interactions with Electrochemically Active Particles: *Russell H. Jones*[1]; Donald R. Baer[1]; Michael J. Danielson[1]; John S. Vetrano[1]; Charles F. Windisch[1]; [1]Pacific Northwest National Laboratory, Mats. Scis. Dept., P.O. Box 999, MSIN P8-15, Richland, WA 99352 USA

Particles can have a mechanical, chemical or electrochemical interaction with an advancing stress corrosion crack. Particles with elastic modulii greater than the matrix will induce a repulsive force on the crack and particles with elastic modulii less than the matrix will induce an attractive force. Chemical interactions include particles that dissolve and alter crack-tip chemistry while electrochemical interactions include particles that are either anodic or cathodic to the advancing crack. Particles that are anodic to the crack, may impede crack advance until totally dissolved or until the particle/matrix interface dissolves. These particles may be highly polarized because of their contact with the matrix such as Al3Mg2 particles on grain boundaries of aged AA5083. Particles that are cathodic to the advancing crack may cause local hydrogen reduction that accelerates crack advance around or through the particle. Examples of these crack-tip/particle interactions for Al-Mg and Mg-Al alloys will be described.

11:30 AM

Corrosion of Aluminum Alloy 6061 and 6061/Al2O3 Composite Subjected to Equal-Channel Angular Pressing: Zofia Buczko[1]; *R. A. Buchanan*[1]; P. K. Liaw[1]; T. G. Langdon[2]; [1]University of Tennessee, Dept. of Mats. Sci. & Eng., Knoxville, TN 37996-2200 USA; [2]University of Southern California, Depts. of Mats. Sci. & Mech. Eng., Los Angeles, CA 90089-1453 USA

High interest is developing in a new materials processing method, equal-channel angular pressing (ECAP), because it has the capability to produce severe plastic deformation without changing the shape of an object. Under proper conditions, ECAP can produce an ultrafine-grained material, resulting in exceptional strength and ductility. The current study examined the effects of ECAP on the aqueous-corrosion properties of 6061 and a 6061/Al2O3 composite material. Two electrolytes were employed: (1) 0.5 M NaCl, and (2) a more aggressive 1.0 M NaCl+H2O2. Cyclic anodic polarization tests in solutions 1 and 2, and 6-hour chemical-immersion tests in solution 2, produced consistent results. In all cases, the dominant form of corrosion was pitting attack. In solution 1, the non-deformed alloy exhibited the lowest corrosion rate, and the ECAP increased the corrosion rates of the alloy and the composite to comparable values. In solution 2, these general trends again were evident, with the exception that the deformed alloy, as compared to the deformed composite, exhibited a much higher corrosion rate. An explanation may involve the ability of the Al2O3 dispersion in the composite to retard corrosion-pit propagation in the more aggressive electrolyte.

Chemistry and Electrochemistry of Corrosion and Stress Corrosion: A Symposium Honoring the Contributions of R.W. Staehle: Stress Corrosion of Waste Container Materials and Other Topics

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

Thursday AM Room: 222
February 15, 2001 Location: Ernest N. Morial Convention Center

Session Chairs: Gustavo A. Cragnolino, Southwest Research Institute, Center for Nuclear Waste Regulatory Analysis, San Antonio, TX, 78238-5166 USA; Gary S. Was, University of Michigan, Nucl. Eng. and Radiol. Scis., Ann Arbor, MI 48109-2104 USA

8:30 AM

Stress Corrosion Cracking in Supercritical Water Systems for Waste Destruction: *Ronald M. Latanision*[1]; D. B. Mitton[1]; [1]Massachusetts Institute of Technology, The H.H. Uhlig Corrosion Lab., Rm. 8-202, Cambridge, MA 02139 USA

There is a need to destroy both military and civilian hazardous wastes and an urgency, mandated by public concern over the traditional waste handling methodologies, to identify safe and efficient alternative technologies. One very effective process for the destruction of such wastes is supercritical water oxidation (SCWO). By capitalizing on the properties of water above its critical point, this technology provides rapid and complete destruction of a wide variety of wastes. A major limitation to full scale commercialization of SCWO is the corrosion-related failure of the materials of construction of these engineering systems. In this presentation, forensic analysis of failures in nickel-based alloy preheater tubes exposed to methylene chloride feed solutions will be shown to provide a basis for understanding the thermodynamic conditions under which SCWO reactors can be operated successfully.

9:00 AM

Stress Corrosion Cracking of a High Performance Nuclear Waste Containment Material: *R. Daniel McCright*[1]; Joseph C. Farmer[1]; [1]Lawrence Livermore National Laboratory, Livermore, CA 94550 USA

The prediction of the performance of materials for the very long-term containment of nuclear waste in a geological repository presents an unprecedented challenge in corrosion science and technology. The United States Department of Energy (DOE) is studying the suitability of the Yucca Mountain site in Nevada as a potential repository site. The current design for a waste package consists of a 2-cm thick layer of Alloy C-22 (UNS 0022) surrounding a thicker layer of austenitic stainless steel. This purpose of the outer Alloy C-22 layer is to provide the primary long-term containment of commercial spent nuclear fuel and reprocessed defense and commercial high-level waste. The stainless steel layer gives added bulk and strength to the waste package. Various corrosion-relevant scenarios for the waste package indicate that stress corrosion cracking (SCC) remains as the corrosion mode most likely to degrade seriously the waste package, particularly in and around the welded region. This paper focuses on the approaches taken to evaluate the SCC susceptibility of Alloy C-22 in the different environments expected to develop at the Yucca Mountain site over the next several thousands of years, as well as ways to mitigate against SCC susceptibility. The final closure weld, made after the waste is loaded inside the package, presents many constraints on ways to mitigate against residual stress in the weld region, and hence potential SCC susceptibility. The experimental SCC program closely couples with deterministic and probabilistic modeling work to project the long-term performance of the Alloy C-22 container from relatively short-term data.

9:30 AM

Initiation and Propagation of Stress-Corrosion Crack in Alpha-Titanium Alloys as Candidate Container Materials for the HLW Disposal: *Masatsune Akashi*[1]; Noriko Nakamura[1]; Yuichi Fukaya[1]; Guen Nakayama[1]; Hiroyoshi Ueda[2]; [1]Ishikawajima-Harima Heavy Industries Co., Ltd., Res. Lab., 3-1-15 Toyosu, Kotoku, Tokyo 135-8732 Japan; [2]The Tokyo Electric Power Company, Tokyo, Japan

Initiation and propagation behavior of stress-corrosion crack was investigated for alpha-phase titanium alloys in hydrogen-evolution electrode potential region. The following conclusions were made; (1) a definite correlation was found between the formation of Ti-hydride layer and the crack initiation based on microscopic observation, (2) a hydride layer started to grow from the surface to the inside of the specimen, (3) when the thickness of the layer reached a critical value, the hydride layer was fractured mechanically, (4) then, a hydride layer started to grow at the tip of the crack, (5) the newly formed hydride layer started cracking mechanically, and (6) the stress-corrosion crack propagated according to the sequence. Based on the experimental data on the critical thickness of hydride layer for cracking and on the time dependency of the hydrogen evolution rate, it was concluded that HLW containers of alpha-titanium alloys were unlikely to suffer the stress-corrosion cracking damage within the coming 1,000 years.

10:00 AM

Environmentally Assisted Cracking of 316 SS Exposed to Gallium and its Oxides: *David G. Kolman*[1]; [1]Los Alamos National Laboratory, Mail Stop E530, Los Alamos, NM 87545 USA

Stabilized plutonium-gallium compounds will be housed in 316 SS containers for up to 50 years prior to final disposition. The processing and storage of these compounds presents a variety of concerns with respect to furnace storage container integrity, respectively. One prominent concern is the unanticipated failure of containers following contact with gallium and its compounds. While gallium is known to embrittle many alloys, most prominently aluminum alloys, little is known about environmentally assisted cracking of stainless steels exposed to liquid Ga or Ga suboxide gas. The objective of this work is to document the susceptibility of storage container materials to environmentally assisted fatigue, crack initiation, and crack propagation using 316 SS compact tension specimens. The cracking behavior of 316 SS will be compared to other prominent engineering alloys.

10:30 AM

Hydrogen Damage of High Tensile Steel in Concrete: *R. Abdel-Karim*[1]; *S. M. El-Raghy*[3]; A. F. Waheed[2]; M. H. Sowelam[3]; [1]Cairo University, Dept. of Min., Petro., and Metall., Giza, Cairo, Egypt; [2]Nuclear Research Center, Anshas, Dept. of Metall., Anshas, Cairo, Egypt; [3]Cairo University, Fac. of Eng., Dept. of Struct. Eng., Giza, Cairo, Egypt

Tempered martensitic high tensile steel bars of different diameters used in prestressed concrete were tested for susceptibility for hydrogen damage. Bare or embedded bars in concrete were cathodically charged in chloride solutions of different pH. The as-received tensile strength was in the range of 2000 MPa. The drop of this strength due to hydrogen entry was dependent on many factors including bar diameter reflected on the microstructure, electrolyte and type of concrete. The same factors affected the drop in the ductility of the cathodically charged steel bars. Thus maximum drop in both tensile strength and in ductility was at pH 7.5, more than in acidic or basic solutions. High quality concrete with silica fume addition reduced the hydrogen damage caused by the same impressed current intensity. Bars with 4-mm diameters were more susceptible to hydrogen embrittlement than those of 5-mm or 6-mm diameter. Further heat treatment effects were investigated and proved the least affected microstructure was the tempered martensite. All the results are discussed and compared with the available literature.

11:00 AM

A Study on Corrosion Failure of a Weathering Steel Weldment with Various Applied Potentials in Acid-Chloride Solution: *Yoon Seok Choi*[1]; Jung Gu Kim[1]; [1]Sung Kyun Kwan University,

Adv. Mats. Eng. Dept., 300 Chunchun-dong, Jangan-gu, Suwon 440-746 Korea

The stress corrosion cracking(SCC) and hydrogen embrittlement cracking(HEC) characteristics of a weathering steel weldment were investigated in aerated acid-chloride solution. The electrochemical properties of weldment were investigated by polarization test and galvanic corrosion test. Weathering steel did not show passive behavior in the acid-chloride solution. Galvanic corrosion between the weld metal and the base metal was not observed because the base metal was anodic to the weld metal. The slow-strain-rate tests(SSRT) were conducted at a constant strain rate of 7.87e-7/s at corrosion potential, and at potentiostatically controlled anodic and cathodic potentials. The weldment of weathering steel was susceptible to both anodic dissolution SCC and hydrogen evolution HEC.

Computational Thermodynamics and Materials Design: Materials Design

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 Dr., Evanston, IL 60208 USA; Gary Shiflet, University of Virginia, Department of Materials Sci. & Eng., Charlottesville, VA 22903 USA; John Vitek, Oak Ridge National Laboratory, Oak Ridge, TN USA

Thursday AM Room: 201
February 15, 2001 Location: Ernest N. Morial Convention Center

Session Chair: John M. Vitek, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA

8:30 AM

Computational Systems Design of Materials: *G. B. Olson*[1]; [1]Northwestern University, Dept. MSE, 2225 N. Campus Dr., Evanston, IL 60208 USA

The computational thermodynamics pioneered by Kaufman has formed the foundation of a systems approach to multilevel materials design integrating materials science, applied mechanics and quantum physics. For martensitic transformations, a combination of quantum-mechanical total-energy calculations and nonlocal continuum methods predicts the evolution of Kaufman-Cohen embryos controlling Ms temperatures. Quantitative nucleation theory permits optimization of dispersed austenite stability for transformation toughening in UHS martensitic steels efficiently strengthened by nanoscale alloy carbide precipitation. Total energy calculations further predict grain boundary thermodynamics for hydrogen embrittlement resistance. New modeling initiatives address the acceleration of the full materials development and qualification cycle, revolutionizing the materials profession.

8:55 AM

Using Computational Thermodynamics to Design CuInSe2 Thin Film Solar Cells: *Tim Anderson*[1]; Weidong Zhuang[2]; Mushin Ider[1]; Alex Chang[3]; B. J. Stanbery[1]; Jianyun Shen[2]; [1]University of Florida, Chem. Eng. Dept., Gainesville, FL 32611 USA; [2]General Research Institute for Nonferrous Metals, Beijing 100088 China; [3]Oregon State University, Chem. Eng. Dept., Corvallis, OR USA

Significant advances have been made in Cu(In,Ga)Se2(CIGS)-based solar cell performance during the past several years, while the challenge of developing low cost manufacturing processes remains. Selection of low temperature reaction pathways leading to formation of the absorber layer will permit use of lower cost substrates. Processes that convert precursor films to efficient absorber layers, however, are not optimized or fully understood, and it is likely that other pathways exist. A description of phase equilibria in the Cu-In-Ga-Se system is used to suggest alternative low temperature routes to synthesizing Cu(In,Ga)Se2. EMF measurements are coupled with literature data to provide assessed phase diagrams for selected subsystems. The associated solution model is used to describe the liquid phase with different associates. The sublattice model is used to describe the non-stoichiometric ordered intermediate phases. In addition, structure models and a point defects chemistries for ternary compounds are explored using EXAFS.

9:20 AM

Alloy Design and Life-Time Assessments of Cr Steels for Power Generation: *John Ågren*[1]; Åsa Gustafson[1]; [1]Royal Institute of Technology, Mats. Sci. and Eng., KTH, SE-100 44 Stockholm, Sweden

Tempered martensitic steels, based on 9-12%Cr, 0.1-0.2%C and W, Mo, V, Nb, B, are being developed with the objective of allowing temperatures above 650ºC in boilers and turbines of fossile-fuel power plants. The high temperature strength is goverened by the martensitic lath structure, which thus has to be stabilized at high T, and strengthening by a fine dispersion of carbo-nitrides, MX. In a long-term collaboration with the physics department at the Chalmers Institute of Technology and several industrial partners we have applied thermodynamic and kinetic calcuilations supported by experimental work using SEM, TEM, EFTEM and APFIM, to predict the microstructural changes during heat treatment and usage of the steels. In particular coarsening of MX has been considered.

9:45 AM

Optimization of the Heat-Treatment Schedule for Ni-Base Superalloys: *C. E. Campbell*[1]; W. J. Boettinger[1]; U. R. Kattner[1]; [1]National Institute of Standards and Technology, Metall. Div., 100 Bureau Dr. Stop 8555, Gaithersburg, MD 20899-8555

Reduction of costs and processing times can be achieved through thermodynamic and diffusion modeling that predicts the optimal time-temperature schedule to reach the solution temperature of the alloy. The heat treatment process is optimized to avoid incipient melting and to determine either the fastest or the minimum power usage time-temperature schedule. For an as-cast Ni-Al-Ta alloy, prediction of the optimal time-temperature schedule requires modeling first the solidification process and then optimizing a modeled heat treatment process. The microsegregation resulting from the casting process is predicted using Scheil solidification calculations and numerical simulations of the solidification process (DICTRA). The resulting composition profiles are then used as input files for the numerical diffusion simulations of the heat treatment process, considering both linear and non-linear heating rates. Initial modeling demonstrates that the predictions are strongly dependent on the accuracy of the thermodynamic and diffusion mobility assessments.

10:10 AM Break

10:30 AM

Phase Diagrams and the Aluminum Industry: *Joanne L. Murray*[1]; Douglas A. Weirauch[1]; [1]Alcoa Technical Center, 100 Technical Dr., Alcoa Center, PA 15069 USA

Phase equilibria in both metallic and ceramic systems are key to maintaining existing technologies "in-control and capable" and developing technologies for aluminum production. We organize our process into the segments: Refining-mining the bauxite and refining the ore to alumina-chemicals; Smelting-extracting metallic aluminum from alumina, traditionally via the Hall-Heroult process; Fabricating-alloying, casting, and mechanical; Recycling-cleaning, sorting, environmentally responsible disposal of process byproducts. Applications of metallic phase diagrams always address the problem of producing a tailored set of product properties, e.g. electrical properties, mechanical properties, corrosion-resistance, surface appearance. Applications of mixed-phase equilibrium include: developing refractories for highly reactive alloys, optimizing compatibility of metal and refractories and of metal matrix composites, controlling

aluminum electrolysis using salt-ceramic equilibria, improving recovery in molten-metal processing, and finding optimal waste disposal strategies for bauxite residue. Existing technologies will be reviewed, and needs will be identified for emerging technologies.

10:55 AM

Computational Thermochemistry–From Its Early CALPHAD Days to a Cost-Effective Role in Materials Development and Processing: *Philip John Spencer*[1]; [1]The Spencer Group, P.O. Box 393, Trumansburg, NY 14886 USA

Computational thermochemistry has made giant strides in a relatively short time period. This rapid development is due not only to the influence of the com-puter, both on the ease of computation and on the assessment and storage of data, but also the scientific stimulation and close collaboration maintained over nearly three decades by the relatively small number of scientists participating in CALPHAD meetings and using CALPHAD calculation methods. The use of the commercial computer packages now available enables rapid and inexpensive information to be obtained on process conditions necessary to achieve a product of the required purity with minimum wastage of energy and materials. In this paper, some examples of computer-assisted materials development will be presented, illustrating how early CALPHAD discussions relating to the calculation of ternary phase equilibria from assessed binary alloy data have now been extended to enable reliable prediction of multicomponent phase equilibria in complex materials systems. Thermodynamic predictions of optimum composition ranges for the production of metastable hard-metal and oxynitride coatings by PVD methods will be described.

11:20 AM

Design of Improved Heat Resistant Materials by Use of Computational Thermodynamics: *Bernhard C. Schaffernak*[1]; Horst H. Cerjak[1]; [1]TU Graz, Mats. Sci., Weld. and Form., Koperni-kusgasse 24, Graz A-8010 Austria

Ferritic-martensitic steels are widely used for high temperature applications. Compared with austenitic materials they own a better thermal conductivity, lower thermal expansion coefficient, lower costs and a better resistance against stress corrosion cracking. In this contribution the modelling of the microstructure based on thermodynamic calculations has been used for the description and further alloy design for this steel group. The results are phase diagrams showing the precipitates to be expected as well as driving forces, phase compositions and diffusion coefficients as base for further modelling activities. A calculation model based on the determination of the (calculated) transformation temperatures Ae1 and Ae3 and the diffusion coefficients is proposed, which can help to predict the microstructural stability and the creep strength from the chemical composition. Based on this model test melts for some new alloys has been produced. First creep tests of these alloys confirm the presented approach.

11:45 AM

From Thermodynamics and Phase Diagrams to Computer Design of Stable Many-Player Many-Functional Materials: *A. L. Udovsky*[1]; [1]Russian Acadademy of Science, Inst. of Metall. and Matls. Sci., Leninsky Prospect 49, Moscow 117334 Russia

The main principles of computer design of stable many-player many-functional materials will be consider. This report will be present our new results in the following fields: 1) development of general algorithms for calculations of phase diagrams and thermodynamic properties for binary and ternary systems; 2) creation of autonomic computer programs for calculation of phase diagrams and thermodynamic properties for binary systems with up to 10 solution phases and 10 stechiometric chemical compounds; 3) carrying assessment and out optimizing calculations of phase diagrams and thermodynamic properties for the Ni-Al and Ni-Cr; 4) testing for calculated thermodynamic properties of solid alloys for the Ni-Cr system, which we have not using for optimizing procedure as input data; 5) predictions of thermodynamic properties as functions of compositions and temperature; 6) computer design of stable high-temperature two-player materials-the substrate chemically compatible with heat-resistant coating, 7) determination of compositions of substrate and coating and temperature ranges using as an examples two-layered materials from the Ni-Cr and Ni-Al-Cr alloys; 8) experimental investigations on creation of chemically compatible substrate and coating, including study of thermal stability and chemical stability of coatings on as the Ni-Cr and for as two-phase the Ni-Al-Cr substrates.

Cyanide: Social, Industrial, and Economic Aspects: Alternatives

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

Thursday AM — Room: 225
February 15, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Corby Anderson, Montana Tech, CAMP and Metallurgical and Materials Engineering, Butte, MT 59701 USA; Kenneth Han, South Dakota School of Mines and Technology, Dept. of Mats. and Metall. Eng., Rapid City, SD 57701-3995 USA

8:30 AM Plenary

Are There Any Realistic Alternatives to Cyanide as a Gold Lixivant at the Present Time?: M. J. Nicol[1]; D. M. Muir[1]; *I. M. Ritchie*[1]; W. P. Staunton[1]; [1]A J Parker Cooperative Research Centre for Hydrometallurgy, Perth, Western Australia 6150, Australia

Cyanide owes its great success as a lixivant for gold to the great strength of the Au-CN- bond. A replacement for cyanide must similarly be a strong complexant for gold (I) or gold (III). The various alternatives are briefly considered and it is concluded that only thiosulfate has a reasonable chance of replacing cyanide as a relatively non-toxic lixivant at the present time. However, there are considerable technical problems to be overcome before thiosulfate can be considered to be a realistic alternative to cyanide. Firstly, the leaching of gold using aerated ammoniacal thiosulfate with copper as a catalyst is a complex process which needs to be controlled carefully in order to achieve maximum recoveries with minimum reagent losses. Secondly, there needs to be something similar to the CIP process for concentrating the dilute gold thiosulfate solutions resulting from processing low grade gold ores. Finally, the gold must be recovered from the solution. The prospects for overcoming these technical challenges are discussed.

9:10 AM Invited

How Rapidly Do Alternative Lixiviants Leach Gold? M. I. Jeffrey[1]; P. L. Breuer[1]; W. L. Choo[1]; Monash University, Dept. of Chem. Eng., Clayton, Vic. 3168 Australia

Due to the increasing environmental and public concerns over cyanidation, there has been a large amount of research into viable alternative lixiviants. Despite these studies, there is little published data directly comparing the kinetics of gold leaching for each of these alternatives; most of the literature is concerned with gold extraction from ores. This paper will present a detailed kinetic study of gold leaching in cyanide, ammonia/thiosulfate, chloride/hypochlorite, and ammonia solutions. The gold leach rates were measured using a rotating electrochemical quartz crystal microbalance (REQCM). This instrument allows the mass of a gold sample to measured in-situ, with a sensitivity of less than 10 ng. From the results of these studies, it is possible to select experimental conditions where the leach rate of the alternative lixiviants is similar to that for the traditional cyanide system.

9:35 AM Invited
Fundamental Aspects of the Gold Thiosulfate Leaching Process: *Paul Breuer*[1]; Matthew Jeffrey[1]; Wei Lit Choo[1]; [1]Monash University, Chem. Eng. Dept., Clayton, Vic 3800, Australia

The majority of studies conducted on the leaching of gold in solutions containing thiosulfate, ammonia and copper during the past 3 decades have failed to acknowledge the undesirable side reaction between copper(II) and thiosulfate, let alone account for its effect. The kinetics of this side reaction have been studied in detail and found to be highly dependent on solution conditions. Since the solution chemistry is constantly changing it is very difficult to perform a fundamental study of gold leaching. With the advent of the rotating electrochemical quartz crystal microbalance, such fundamental studies are possible, as the leach rate can be measured in a matter of minutes and hence at essentially constant solution conditions. The effect of various parameters on the long term gold leach rate have been investigated. Studies have also commenced into the cementation and electrowinning of gold from thiosulfate solutions over a wide range of experimental conditions.

10:00 AM Break

10:15 AM Invited
Ion Exchange Resins for the Recovery of Gold from Thiosulfate Leach Pulps: *Glen O'Malley*[1]; Michael J. Nicol[1]; [1]Murdoch University, Min. Sci. Dept., South St., Murdoch, Perth, Western Australia 6150, Australia

Growing environmental concerns about the use of cyanide in gold processing has increased the interest for more acceptable alternatives that most likely to involve thiosulfate. However, as activated carbon is ineffective in recovery of the gold thiosulfate complex, the thiosulfate process lacks a proven in-pulp method of recovering the dissolved gold. Anion exchange resins do offer a possible route for in-pulp recovery. This paper describes some work undertaken at the AJ Parker Centre for Hydrometallurgy to evaluate the effectiveness of commercially available anion exchange resins for recovering gold from thiosulfate leach liquors and pulps. The effect of competing anions on the equilibrium and the kinetics of gold loading were investigated and will be discussed. Evaluation of the effectiveness of resins for gold recovery from thiosulfate pulps by counter-current adsorption will also be presented. An assessment of a proprietary elution process for the recovery of gold from the resin and an electrowinning study will also be covered within the paper.

10:40 AM Invited
Electrochemical Behavior of the Dissolution of Gold in Ammoniacal Solutions: *Kenneth N. Han*[1]; [1]South Dakota School of Mines and Technology, Dept. of Matls. and Metall. Eng., Rapid City, SD 57701-3995 USA

Ammonia has been found to be an effective leaching reagent for gold, silver and platinum group metals in the presence of appropriate oxidants. Electrochemical and kinetic aspects of the dissolution of elemental gold in ammoniacal solutions have been investigated. The cathodic reaction of aurous di-ammine on the gold cathode has been investigated first. From this investigation, the standard electropotential of the cathodic reaction and hence the Gibbs free energy of formation of the aurous di-ammine were established. The solubility of gold in ammoniacal solutions was examined in relation to ammonia concentration, temperature and concentration of various oxidants. The anodic reaction of the gold disk in ammonia in the presence of various oxidants including oxygen, ozon, hypochlorite, hydrogen peroxide, cupric and cobaltic ammines, and iodine was also examined and the results have been discussed. Excellent dissolution yielding of gold and many other metals, typically better than 95% recovery in 1-2 hours of leaching at a temperature ranging 80°C to 190°C has been observed. Test results of the dissolution of gold from elemental state as well as other sources including sulfidic ores and scrap are presented.

11:05 AM Invited
Halides as Alternative Lixivants for Processing Gold and SIlver? An Update: *Tam Tran*[1]; Ken Lee[1]; Kapila Fernando[1]; [1]The University of New South Wales, Sch. of Chem. Eng. & Indust. Chem., Sudney, New South Wales 2052, Australia

Over the last decade, halide (chloride, bromide and iodide) systems have been suggested as alternative lixiviants for the processing of gold and silver to replace cyanidation which has been widely used in commercial practice. This paper first reviews the development of the processes promoted by Kaljas Ltd (Australia) and the Great Lakes Corp (USA) using bromide/bromine systems. Several research groups subsequently evaluated the use of iodide/iodine and chloride/chlorine for gold and silver extraction, showing similar characteristics of gold/silver dissolution and recovery. The processing of silver and gold has also been dealt with by Intec Copper Ltd., in the development of their Intec copper process using chloride. The paper provides a critique on the advantages and the shortfalls of halide systems as alternative lixiviants. The stability of gold/silver halide complexes is discussed, based on practical experience and thermodynamic modelling of their speciation. The iodide system provides the most stable gold/silver complex for easier handling. However, the cost of iodide inhibits its application for ore processing. The cheapest system which involves the use of chloride for leaching (in conjunction with an appropriate oxidant) however has a major drawback related to the unstable nature of gold/silver choride in a low Eh environment. No doubt the halide systems can be used for gold/silver processing. However, their potential might be limited to applications outside mineral processing areas.

11:25 AM Invited
The Industrial Non-Cyanide Hydrometallurgical Recovery of Silver and Gold Utilizing Nitrogen Species Catalyzed Pressure Oxidation: *Corby G. Anderson*[1]; [1]Montana Tech, The Center for Adv. Min. & Metall. Proc., 1300 West Park St., Rm. 221 ELC Building, Butte, MT 59701

In the majority of industrial operations that recover gold and silver by hydrometallurgical means, cyanide is the predominant lixiviant. However, as an alternative, production of massive quantities of silver chloride was done on an industrial scale in the nitrogen species catalyzed pressure leaching of a refractory precious metals concentrate. This proven process required no cyanide. Moreover, gold bearing concentrates can also be trested by nitrogen species catalyzed pressure leaching followed by a novel non-cyanide leaching and reocvery method. In the paper an in-depth description of the proven pressure leaching system is included along with the silver and gold recovery methodologies.

11:50 AM Invited
Closing Remarks: Courtney A. Young; Montana Tech, Metallurgical and Materials Engineering, 2154 ELC Building, 1300 W. Park Street, Montana Tech, Butte, MT 59701 USA

Defect Properties and Mechanical Behavior of HCP Metals and Alloys: Alloy Design, Processing, and Applications

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Electronic, Magnetic & Photonic Materials Division, Chemistry & Physics of Materials Committee, Jt. Nuclear Materials Committee, Titanium Committee
Program Organizers: Man H. Yoo, Oak Ridge National Laboratory, Metals & Ceramic Division, Oak Ridge, TN 37831-6115 USA; James R. Morris, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; Carlos N. Tome, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Thursday AM	Room: 211
February 15, 2001	Location: Ernest N. Morial Convention Center

Session Chairs: James C. Williams, Ohio State University, Mats. Sci. and Eng., Columbus, OH 43210-1178 USA; K. Linga Murty, North Calorina State University, Raleigh, NC 27695-7907 USA

8:30 AM Invited
Magnesium Alloys for Structural Application: *Karl Ulrich Kainer*[1]; [1]GKSS Research Center Geesthacht, Inst. for Mats. Res., Max-Planck-Str., Geesthacht D-21502 Germany

Increasing demand for lightweight structural material in transportation industry leads to a renaissance for magnesium. Magnesium alloys fulfill the requirements of low density, good and economic processability by using cast technology and high recycling potential. The driving force for the constant growth in magnesium production recently has been the increasing demand for die cast parts in the automotive industry. Die-casting is the dominating production techniques for economical magnesium parts. The range of applications in automobile goes from use in powertrains, as body parts, in interior and chassis. There is also a growing interest in the use of magnesium materials in areas in which the properties of introduced magnesium alloys are not matching the requirements. Results are raising R&D-activities in developing new alloys and processes. This paper gives an overview on commonly used magnesium alloys and their application. An outlook on trends in alloy development and on potential applications is given.

9:00 AM

Twinning, Dynamic Recovery and Recrystallization in Hot Worked Mg-Al-Zn Alloy: Mikhail M. Myshlyaev[1]; *Hugh J. McQueen*[2]; A. Mwembela[2]; E. V. Konopleva[2]; [1]Baikov Institute of Metallurgy RAS, Moscow 117911 Russia; [2]Concordia University, Mech. Eng. Dept., 1455 Maisonneuve W., Montreal, Quebec H3G 1M8 Canada

The AZ31 Mg alloy was subjected to hot torsion testing over the range of 180 to 450°C and 0.01 to 1.0 s-1. The flow curves showed a peak and a decline towards a steady state regime which were lower as temperature rose and strain rate declined; however, the fracture strain increased to about 1.9 at 0.1s-1. In transmission electron microscopy, twins were observed from 180 to 360°C (in declining numbers). At low temperature, they had sharp walls and contrasting transverse bands; while the matrix showed indistinct linear streaks. As T rose, the bands became cells with tangled walls and finally subgrains (~360°C), while the twin walls became tangles of dislocations and finally serrated boundaries. The matrix developed elongated dislocation walls and subgrains at higher T. The twin intersections at 180 and 240°C consisted of diamond-shaped cells with a duplex set of orientations but at 300 and 360°C, these had developed into polygonal cells with high misorientations in dark field. The first dynamically recrystallized grains were observed at these intersections, slightly larger than the cells but free of dislocations. At 420 and 450°C, large dynamically recrystallized grains were observed, apparently in the matrix and probably at the original grain boundaries, as was observed by optical microscopy.

9:20 AM

Directional Mechanical Performance of Wrought AZ80 Magnesium: *Matthias Hilpert*[1]; H. J. Rack[2]; L. Wagner[1]; [1]Technical University of Brandenburg at Cottbus, Chair of Phys. Metall. and Mats. Techn., P.O. Box 101344, 03013 Cottbus, Brandenburg, Germany; [2]Clemson University, Dept. of Ceram. and Mats. Eng., 110 Olin Hall, Clemson, SC 29634-0907 USA

This presention will examine the effects of thermomechanical processing (rolling, pressing and swaging) on the mechanical behavior of extruded AZ80 (8%Al, 0.5%Zn, 0.2%Mn) magnesium. It will be shown that the monotonic stress-strain response strongly depends upon loading direction. For example, differences between the compressive and tensile yield strengths in the as-extruded condition can exceed 50%. These differences are also reflected in fully reversed, R=-1, fatigue loading, where a marked asymmetry in the cyclic stress-strain response is observed. These observations will be shown to result from the interrelationship between crystallographic texture development and the stresses necessary for twin deformation during compressive/tensile loading.

9:40 AM

Equal Channel Angular Extrusion (ECAE) of Beryllium: *R. D. Field*[1]; K. T. Hartwig[2]; C. T. Necker[1]; J. F. Bingert[1]; S. R. Agnew[3]; [1]Los Alamos National Laboratory, MST-6, Mail Stop G770, Los Alamos, NM 87545 USA; [2]Texas A&M University, Dept. of Mech. Eng., College Station, TX 77843-3123 USA; [3]Oak Ridge National Laboratory, Mets. and Ceram. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831-6115 USA

P/M source beryllium was processed by Equal Channel Angular Extrusion (ECAE). Billets were canned in Ni and extruded at 400°C. After two passes (using two different processing routes for the second pass) the billets display significant grain refinement with no cracking. Deformation structures were examined using transmission electron microscopy (TEM) and textures measured by x-ray diffraction (XRD) and orientation imaging microscopy (OIM). The billets develop crystallographic texture, which is dependent upon the orientation of the second pass. Anneals up to 800°C yield recrystallized microstructures, retaining the texture and much of the grain refinement developed during extrusion. The texture data were compared to simulated textures calculated using a self consistent deformation model. Deformation microstructures and texture development will be discussed in terms of the deformation modes of Be and the geometry of the ECAE process. This work was supported under DOE Contract W-7405-ENG-36.

10:00 AM

Pressure Effects on Flow and Fracture Behavior of Al-Be Composites: *Joël Larose*[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Mats. Sci. and Eng., The Case School of Eng., Cleveland, OH 44106 USA

The flow and fracture behavior of Al-Be composites are being determined under a variety of test conditions. The flow behavior is being determined in tension with different levels of superimposed pressure. Significant effects of pressure on both the flow and ductility have been observed. The effects of test conditions on the flow and fracture behavior will be summarized in addition to both optical and SEM examination of the fracture surfaces. Separate other studies on the composite constituents (e.g. Al, Be) will be also reported.

10:20 AM Break

10:40 AM Invited

Processing and Anisotropic Properties of Zirconium Alloys for Nuclear Applications: *R. A. Holt*[1]; [1]Atomic Energy of Canada Limited, Chalk River Labs., Chalk River, Ontario K0J 1J0 Canada

Dilute zirconium alloys are used extensively for structural components in the cores of nuclear reactors. These alloys are highly anisotropic because of their HCP crystal structure and the pronounced crystallographic textures developed during processing. A unique feature of the reactor core environment is the continual displacement of the atoms from their crystal lattice positions by neutrons. This causes changes in the microstructure and mechanical properties of in-core materials during service and radiation induced dimensional changes including irradiation growth, irradiation creep and swelling (which does not occur in zirconium alloys at normal reactor operating temperatures). This paper reviews the development of crystallographic texture in zirconium alloys during manufacturing, the anisotropic properties pertinent to their application in nuclear reactors and the changes induced by fast neutron irradiation under normal reactor operating conditions. The microscopic mechanisms controlling the development of texture and the anisotropic properties, including irradiation creep and growth are discussed. Two outstanding issues in this area are the mechanism of texture development during alpha-beta processing and the contribution of second phase (i.e., meta-stable beta-zirconium) to the anisotropy of textured polycrystalline material.

11:10 AM

Plastic Response of Chemical Vapor Deposited Rhenium: *Ghatu Subhash*[1]; Phil Lukens[1]; Pletka J. Bruce[2]; [1]Michigan Technological University, ME-EM Dept., 1400 Townsend Dr., Houghton, MI 49931 USA; [2]Michigan Technological University, Mats. Sci. and Eng., Houghton, MI 49931 USA

Plastic response of rhenium (Re) produced by chemical vapor deposition (CVD) was investigated under uniaxial tensile loading. The initial microstrctural analysis of CVD Re revealed layers of columnar grains and a strong basal texture perpendicular to the tensile axis. The plastic response exhibited a two stage hardening behavior with considerably higher hardening rate than other refractory metals including rhenium produced by powder metallurgy. The fracture surfaces of the specimens revealed typical ductile fracture. Microscopic observations of the lateral surfaces of the deformed

tensile specimens revealed cracking at 45 degrees to the tensile axis. Extensive twinning was also observed within the deformed zone. Currently transmission electron microscopic observations are being conducted on specimen obtained from various regions of the tensile specimens. The experimental results and microscopic investigations of the micromechanisms of deformation responsible for the observed behavior of CVD Re will be discussed during the presentation.

11:30 AM

Delayed Hydrogen Cracking in CANDU Pressure Tubes as a Result of Local Reorientation of the Matrix within Zones of Stress Concentration by Tensile Loading: *Yuriy Perlovich*[1]; Margarita Isaenkova[1]; Young Suk Kim[2]; [1]Moscow State Engineering Physics Institute (Technical University), Met. Sci. Dept., Kashirskoe shosse 31, Moscow 115409 Russia; [2]Korean Atomic Energy Research Institute, Yusong, Taejon 305-600 Korea

The anisotropy of DHC in CANDU pressure tubes becomes understandable by taking into account the local reorientation of the alpha-Zr matrix under tensile loading within zones of stress concentration near tips of notches, cracks and precipitates. Because of the tangential orientation of basal planes in CANDU tubes, the local plastic deformation within these zones under transverse tensile loading realizes at first by means of twinning with the resulting reorientation of basal axes by ~85°. Due to accompanying redistribution of shear stresses, the following activation of the prismatic slip within the twinned reoriented crystallites proves to be possible, so that the plastic deformation zone with an increased lattice distortion is surrounded by a sharp boundary, separating it from the undeformed matrix. The high gradient of lattice distortion promotes intensifying of the hydrogen diffusion to the plastic deformation zone, where new stress-oriented hydrides precipitate according to the orientation relationship with the twinned crystallites of alpha-Zr. When determining the habit planes of hydrides relative to the reoriented matrix, contradictory data on habit planes can be corrected. The difference in DHC by axial and radial directions of the notch under transverse tensile loading is conditioned by features of the compressive strain along the notch direction in front of the crack tip.

Defect Properties and Mechanical Behavior of HCP Metals and Alloys: *Poster Session*

Computer Simulation of Interfacial Structure and Defect Properties in the HCP Metals: *Anna Serra*[1]; David J. Bacon[2]; [1]Universitat Politecnica de Catalunya (UPC), Dept. Matematica Aplicada III, Jordi Girona,1-3 Modul C-2, Barcelona 08034 Spain; [2]The University of Liverpool, Dept. of Eng. Brownlow Hill, Liverpool L69 3GH UK

It has been stablished by computer simulation that clusters of self-interstitial atoms (SIAs) form in the displacement cascade process that gives rise to radiation damage in irradiated metals. These clusters (or small dislocation loops) can be highly mobile and this has implications for the evolution of damage microstructure and changes in material properties. The interaction of clusters with other defects can result in hardening by the formation of atmospheres and/or resistance to glide as dislocations move through distributed obstacles. In some cases, plastic deformation is accompanied by the formation of cleared channels. In the hcp metals the interaction of clusters and dislocation loops with twin boundaries has been suggested as a possible mechanism for cleaning channels. The present research work is the first stage of a study of this by computer simulation. It is focused on the interaction of these clusters with twin boundaries that we have modelled previously. Results will be presented for the interaction of a SIA cluster with; (a) a planar twin interface; (b) a boundary with a stationary twinning dislocation and; (c) a boundary containing moving twinning dislocations under twinning shear stress.

Composition Effects on the Crystallininty of Beryllium-Rich Coatings: *Alan F. Jankowski*[1]; [1]Lawrence Livermore National Laboratory, Chem. and Mats. Sci., P.O. Box 808, L-352, Livermore, CA 94551-9900 USA

The material properties of sputter deposited coatings are sensitive to the growth morphology and microstructure of the deposit. For example, the application of an applied bias to the substrate can densify the columnar microstructure of a crystalline coating through ion bombardment thereby minimizing porosity and increasing the mechanical strength of the coating. We are developing a process to sputter deposit Be-rich, thick coatings that must be homogeneous, isotropic, mechanically strong, smooth and serve as a membrane across which gas permeation is controllable. The refinement of grain size to the nanoscale is preferable for each of these attributes. It is widely known from prior studies of evaporation and sputter deposition that the grain size of nominally pure beryllium, that is beryllium with greater than 99.8 atomic percent purity, can be dramatically refined through the incorporation of specific metal impurities like iron. Additionally, the use of boron doping may serve as a potential glassy phase former in the composition range greater than at the depressed eutectic seen in the binary alloy phase diagram. We report on the changes in crystallinity and growth morphology found in these Be-rich coatings. The effects of dopant additions are characterized with electron diffraction and microscopies. This work was performed under the auspices of the U.S. Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

Dynamic Recrystallization in a Magnesium Alloy at Intermediate Temperatures: Arthur Galiyev[1]; *Rustam Kaibyshev*[1]; [1]Institute for Metals Superplasticity Problems, Khalturin 39, Ufa 450001 Russia

Effect of temperature on deformation mechanisms and microstructure formed was studied in a Mg-5.8%Zn-0.65%Zr alloy in the temperature range from 423K to 573K (0.46-0.62 Tm, where Tm is the absolute melting temperature). Microstructural evolution and mechanisms of plastic deformation were found to be in strong dependence on temperature. At T=423K the low temperature dynamic recrystallization (LTDRX) occurred. The deformation twinning is operative at initial stage of plastic deformation. Evidences for operation of basal slip and <a+c> dislocation slip were found. At temperatures ranging from 473K to 573K the continuos DRX (CDRX) took place. In this temperature interval an extensive cross-slip was developed. A mechanism for the CDRX occurring in the magnesium alloy is presented. A relationship between the operating deformation mechanisms and mechanisms of grain formation is discussed. A role of dislocation cross-slip in DRX in the magnesium alloy was considered.

Diffusion in HCP Ti and Zr at Low Temperatures: *Rodolfo Ariel Perez*[1]; Fanny Dyment[1]; [1]CNEA, Materials, Av. General Paz 1499, San Martin, Pcia de Buenos Aires 1650 Argentina

Diffusion studies of Zr, Hf, Au, Co, Pb, Sn, In and Ta in hcp Ti matrix and Pb, Hf and Au in hcp Zr were performed. The combination of Rutherford Backscattering Spectrometry (RBS) Heavy Ions RBS (HIRBS) and Direct Sectioning techniques were used in superposed ranges, in order to cover a wide temperature range, from the phase transition (1156 for Ti and 1138 for Zr) to low temperatures (between 773K and 620K according to the diffusing impurity). All the diffusing impurities in a-Ti follows the Arrhenius law whereas in the a-Zr matrix all the elements studied shows a downward curvature that follow the self-diffusion behaviour. A discussion between the fast impurities influence on the diffusion in such matrix versus intrinsic behaviour is proposed.

Heredity of Structure Inhomogeneity by Phase Transformations Beta->Omega and Beta->Alpha in the Rolled Zr-20%Nb Alloy: *Yuriy Perlovich*[1]; Margarita Isaenkova[1]; Hans-Joachim Bunge[2]; Vladimir Fesenko[1]; [1]Moscow State Engineering Physics Institute (Technical University), Met. Sci. Dept., Kashirskoe shosse 31, Moscow 115409 Russia; [2]TU Clausthal, Clausthal-Zellerfeld 38678 Germany

Development of phase transformations (PT) β->ω and β->α in the rolled quenched alloy Zr-20%Nb by its annealings at 400º and 500°C was studied by the X-ray method, combining texture measurements and X-ray line profile analysis. Both ω- and α-phases

prove to be textured and inherit the substructure inhomogeneity of initial β-Zr, i.e. in texture minima their grains are most disperse and/or have the most distorted crystalline lattice, while in texture maxima crystallites of derivative phases are coarser by lower lattice distortions. However, though grains of the original β-phase differ significantly in lattice condition, grains of the athermal ω-phase have very close substructure parameters. The opposite tendency takes place by isothermal PT β->α: since α-grains form by decomposition of quenched β-Zr into two equilibrium phases, the final structure of the alloy is characterized by increased dispersity of components. Elastic microstrains in the ω-phase are higher than in the initial β-phase, though main features of the anisotropic microstrain distribution proves to be inherited. The dependence of both PT on elastic compression and extension was analyzed in details.

Mechanical Anisotropy of Textured Zr-Alloys as a Result of Differences in Plastic Deformation at the Crack Tip: *Yuriy Perlovich*[1]; Margarita Isaenkova[1]; Vladimir Goltcev[1]; [1]Moscow State Engineering Physics Institute (Technical University), Met. Sci. Dept., Kashirskoe shosse 31, Moscow 115409 Russia

By mechanical testing a wave of local plastic deformation moves ahead of any crack and is responsible for strain hardening and rotation of the crystalline lattice within the layer adjacent to the fracture surface. By means of X-ray diffractometric study of fracture surfaces as applied to sheets of Zr-alloys it was shown, that in alpha-Zr near this surface the specific texture of tension forms with the axis perpendicular to the crack plane. Structure features of the plastic deformation zone at the crack tip controls the further behavior of the fracture process. Depending on the direction of tensile loading, shear stresses in systems of possible slip and twinning vary, so that the local plastic deformation of the notched sample realizes in different manners: either by the prismatic slip resulting only in minor texture changes within a rather extended zone in the case of RD-tension or by the twinning, associated with a significant jump-like reorientation of crystallites and the following prismatic slip within a smaller zone with distinct boundaries in the case of TD-tension. The sharp texture inhomogeneity, arising near the crack tip by TD-tension, causes an additional stress concentration and therefore quickens the further movement of the crack as well as fracture of the sample.

Mechanisms of Structure Development in Hf and Hf-Zr Alloys: *Yuriy Perlovich*[1]; Margarita Isaenkova[1]; Oleg Bocharov[2]; Vladimir Fesenko[1]; Alexander Shikov[2]; Sergey Bochenkov[1]; [1]Moscow State Engineering Physics Institute (Technical University), Met. Sci. Dept., Kashirskoe shosse 31, Moscow 115409 Russia; [2]All-Russia Institute of Inorganic Materials, Rogova, 5, Moscow, Russia

The process of texture development by hot and cold deformation of Hf and Hf-Zr alloys with the Zr content up to 20% was studied using methods of X-ray diffractometry. The participation of different micromechanisms in the plastic deformation at its successive stages was evaluated by means of texture analysis. Among these mechanisms there are twinning by different systems as well as basal, pyramidal and prismatic slip. The dependence of acting mechanisms on the grain orientation, the deformation temperature and the content of Zr was considered. Substructural features of grains with different orientations were compared owing to the selective character of X-ray data. Changes of the texture of samples by the recrystallization at different temperatures were studied also. The correlation of the recrystallization textures with the nonuniform strain hardening of samples was ascertained. The effect was discovered, consisting in the formation of the omega-phase in the surface layer of rolled Hf-Zr alloys by their annealing. The volume fraction of the omega-phase depends on the content of Zr.

Substructure Inhomogeneity and Distribution of Residual Micro- and Macrostresses in Products of Zr-Alloys: New Approaches on the Basis of X-ray Diffractometry and Computer Data Treatment: *Yuriy Perlovich*[1]; Margarita Isaenkova[1]; [1]Moscow State Engineering Physics Institute (Technical University), Met. Sci. Dept., Kashirskoe shosse 31, Moscow 115409 Russia

The recent development of X-ray diffractometry resulted in the conclusion, that real textured metal materials are extremely inhomogeneous and include a very wide spectrum of substructure conditions. As applied to products of Zr-alloys, a new X-ray method of the fullest description of their structure features was elaborated, combining measurements of X-ray line profiles and texture pole figures. Distributions of physical line broadening and peak position as well as derivative distributions of coherent block size, lattice distortion and elastic microstrains are constructed depending on the grain orientation (so-called generalized pole figures). A procedure to construct the polar distribution of macrostresses on the basis of peak position pole figures was elaborated also. Rich possibilities of the method are demonstrated by an example of the comparative study of Zr-1%Nb and Zr-2.5%Nb sheets, experienced both straight and transverse rolling as well as following heat treatment and hydrogen charging. For the first time an actual scale of the inevitable substructure inhomogeneity and its connection with the texture were revealed for products of commercial Zr-alloys, modes of the equilibrium of elastic microstresses and the anisotropy of residual macrostresses were studied, effects of plastic deformation and annealing on the shape and the volume of the elementary cell in alpha-Zr grains with different orientations were analyzed in details.

Size Effects in the Hardness of Titanium Interlayers Roll Bonded With Aluminum: *Michael E. Stevenson*[1]; Jian-Guo Luo[1]; Viola L. Acoff[1]; *Richard C. Bradt*[1]; [1]University of Alabama, Metall. and Mats. Eng., Box 870202, Tuscaloosa, AL 35487-0202 USA

Laminates of titanium and aluminum sheet were roll bonded to reductions from 40 to 80% thickness. Knoop hardnesses were measured for the titanium layers for loads from 10-500 g at each layer thickness. Results were analyzed in terms of the indentation size effect. The load independent hardnesses were determined as a function of the percent reduction. These results were subsequently applied to address the effects of layer thickness in a Hall-Petch relationship.

Correlation Between Substructure and Mechanical Properties of Alpha-Ti at Varying Deformation Temperatures 4.2-373K: A. R. Smirnov[1]; V. A. Moskalenko[1]; [1]B. Verkin Institute for Low Temperature Physics & Engineering, Nat. Acad. of Sci. of Ukraine, 47, Lenin Ave., Kharkov 61164 Ukraine

The correlation between the substructure of alpha-Ti predeformed at the temperature T1 and its mechanical properties (stress of onset of plastic deformation, true rupture strength, residual relative ductility) on subsequent deformation at the temperature T2 varying within 4.2-373K have been studied. It is shown that the changes in the mechanical properties at T1>T2 and T1<T2 are fundamentally different. In contrast to the isothermal deformation at T2, in the T>T2 case the increment in the deforming stress is smaller and the total ductility does not exceed the highest isothermal value; conversely, at T1<T2 the deforming stress and the total ductility surpass the isothermal level. The different predeformation effects are connected with the stability of the the developed substructure against subsequent deformation: it is poor if T1>T2 and high if T1<T2.

Lightweight Alloys for Aerospace Applications: Deformation, Fatigue and Environmental Fracture-II

Sponsored by: Structural Materials Division, Non-Ferrous Metals Committee

Program Organizers: Kumar Jata, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA; Nack J. Kim, Center for 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

Thursday AM Room: 213
February 15, 2001 Location: Ernest N. Morial Convention Center

Session Chair: Nack J. Kim, POSTECH, Pohang, Korea

8:30 AM Invited
Application of 3D Digital Image Processing to Quantify Frac-

ture Mechanisms in Al 7050 Alloy: Manish D. Dighe[1]; Sunit S. Mukherjee[1]; *Arun M. Gokhale*[1]; [1]Georgia Institute of Technology, Mats. Sci. and Eng. Dept., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Aluminum 7050 is an important alloy that is widely used for applications such as aircraft wing skin structures, aircraft landing gear parts, and fuselage frame structure. Commercial 7050 alloy plates have been observed to have a partially recrystallized microstructure. Thus, it is of interest to study the effect of this microstructure on the fracture properties and fracture morphology. Consequently, an attempt has been made to develop a digital image processing based methodology to reconstruct the three-dimensional (3D) fracture surface and the exact three-dimensional microstructure beneath the fracture surface using serial sectioning. This is to facilitate correlation and quantification by simultaneously observing the 3D morphology of the fracture surface and the microstructure just beneath it. This correlation has also enabled the quantification of the contributions of different types of high angle grain boundaries to inter-granular fracture.

9:00 AM

An Overview on the Fracture Behavior of Discontinuously-Reinforced Aluminum: *A. B. Pandey*[1]; [1]Pratt & Whitney, Liquid Space Prop., P.O. Box 109600, West Palm Beach, FL 33410

Discontinuously-reinforced aluminum (DRA) possesses superior specific stiffness, specific strength, wear resistance and thermal resistance as compared to the monolithic alloys. The use of DRA in aerospace structures such as ventral fin and fan exit guide vane has generated considerable interest in the materials community to widen the applications. One of the major constraints in using DRA for load bearing applications is the lower ductility and fracture toughness of DRA compared to the monolithic alloys. Considerable effort has been made in the past to understand the fracture mechanism in the DRA materials. This paper provides an overview on the microstructural effects on the ductility and fracture toughness of DRA. The influence of matrix heat treatment, particle size, volume fraction and distribution of the reinforcement on the fracture of DRA are discussed. The results indicated that the finer particle size improves the strength, ductility, and toughness of the composite. The heat treatment has significant influence on the fracture behavior of DRA. The reinforcement fracture and matrix/reinforcement interface debonding were dominant damage modes. The effects of specimen size and lamination on the fracture mechanism of DRA are also discussed.

9:30 AM

Microstructure and Mechanical Properties of Metallic Foams: *J. Zhou*[1]; Chris Mercer[1]; Seyed Allameh[1]; B. S.H. Royce[1]; A. G. Evans[1]; W. O. Soboyejo[1]; [1]Princeton University, The Princeton Mats. Instit., Dept. of Mech. and Aero. Eng., Princeton, NJ 08544 USA

The results of ongoing studies of the microstructure and mechanical properties of metallic foams are presented in this paper. These include aluminum, steel and titanium-based steel foams with a range of cell geometries and volume fractions. The microstructure of the cell walls is characterized with scanning and transmission electron microscopy. Quantitative image analysis techniques are then used to characterize the variations in pore geometry at the micro- and meso-scales. The deformation behavior of the cell walls, metallic foams and sandwich geometries, is discussed within the context of experimental observations/measurement and micromechanical models. Preliminary measurements of fatigue and fracture properties are also presented.

9:50 AM

Surface Intrusions in Permanent Mold Cast Aluminum Alloys: J. Shenefelt[1]; R. Thavarajah[1]; R. Luck[1]; *John T. Berry*[1]; [1]Mississippi State University, Mississippi State, MS 39762

Aluminum castings play important roles in a wide variety of civilian and military structures. Since many such applications involve dynamic loading, increased attention to crack initiation and propagation has been paid of late. Unfortunately, much of the current work has involved the evaluation of samples excised from laboratory-scale test castings. The present investigators have purposely conserved the the as-cast surface in determining its effect on fracture in four-pont bend tests on alloys 319-T6 and A356-T6. The samples concerned were cut from actual commercial castings drawn from particular heats in such a manner that the as-cast surface experienced tensional loading. Surface features, particularly intrusions in the case of the 319 alloy are thought to have played an important part in the fracture process.

10:10 AM

On the Effect of Thermomechanical Processing and Thermal Exposure to the Mechanical Properties of 2297 Plates: E. Acosta[1]; O. Garcia[1]; A. Dakessian[1]; K. Aung Ra[1]; J. Torroledo[1]; A. Tsang[1]; M. Hahn[2]; J. Foyos[1]; J. Ogren[1]; *O. S. Es-Said*[1]; [1]Loyola Marymount University, Res. Exp. for Undergrad. Prog., Los Angeles, CA 90045 USA; [2]Northrop Grumman, Mats. and Proc. Techn., Dept 9L26, Zone W5, El Segundo, CA 90245 USA

The objective of this study is to assess the effect of thermal exposure on the tensile properties of 2297 and to assess the feasibility of forging; compression instead of stretch. 24 blocks of 3.8 cm x 3.8 cm x 5.4 cm (1.5 in x 1.5 in x 2.125 in), were solution heat treated at 521ºC, water quenched, and naturally aged for 24 hours. After natural aging the blocks were compressed 0-15% and artificially aged. The artificial aging schedule was: 160ºC for 36 hours, and 177ºC for 36 hours. The hardness values varied in the short-transverse direction showing maximum values in the middle of the surface compared to the edges. The hardness values of the aged samples increased with longer aging times (24 versus 36 hours) and higher temperatures 160ºC versus 177ºC. A multi-step aging process was then incorporated for the compression study in order to optimize the heat treatment. Six samples were solution treated, compressed at 0, 4, and 10%, and aged at the same temperatures as the single step aging study. Three samples were then artificially aged at an initial temperature of 121ºC for 24 hours and then increased to a second temperature of 177ºC for 24 hours. The remaining 3 samples were aged at an initial temperature of 149ºC for 18 hours and then increased to a second temperature of 177ºC for 24 hours.

10:30 AM

Retrogression and Reaging of 7249 Plates: *P. Fleck*[1]; K. Koziar[1]; E. Fromer[1]; P. Herbe[1]; G. Davila[1]; M. Leal[1]; E. W. Lee[2]; Omar Es Said[1]; [1]Loyola Marymount University, Nat. Sci. Found. Res. Exper. for Undergrad. Prog., Los Angeles, CA 90045 USA; [2]Naval Air Systems Command, Code 4.3.4.2, Aircraft Div., Patuxent River, MD 20670 USA

The objective of this study was to investigate the feasibility of performing retrogression and reaging (RRA) heat treatments on 7249-T7651 aluminum alloy in muffle furnaces. The T6 temper was optimized after 39 heat treatments to be 474°C for 1 hour solution treatment followed by water quenching, 24 hours natural aging and artificial aging at 121°C for 24 hours. The retrogression temperatures and times were: 170°, 180°, 190°, and 210°C for 20, 40, 60, 90, and 120 minutes. Reaging was performed at 121°C for 24 hours. Tensile testing, hardness and electrical resistivity measurements were determined.

10:50 AM

Fracture and Fatigue Studies on Al-Be Composites: *Joël Larose*[1]; John J. Lewandowski[1]; [1]Case Western Reserve University, Mat. Sci. and Eng., The Case Sch. of Eng., Cleveland, OH 44106 USA

The fracture and fatigue behavior of Al-Be composites are being determined under a variety of test conditions. Fracture toughness is being determined on both notched and fatigue precracked specimens, while fatigue crack growth behavior is being measured at different R-ratios. The effects of test conditions (e.g. R-ratio, test temperature) on the fracture behavior will be summarized in addition to both optical and SEM examination of the fracture path and fracture surfaces.

11:10 AM

Intergranular Corrosion of RRA-Treated 7075 Aluminum Forgings: Ana Leticia Campuzano-Contreras[1]; *Harold Kelly*[1]; Roy Arrowood[1]; Lawrence E. Murr[1]; Elizabeth A. Trillo[1]; [1]University of Texas at El Paso, Metallur. and Mats. Eng., M201 Eng. Scis., El

Paso, TX 79968-0520 USA; [1]University of Texas at El Paso, Metallur. and Mats. Eng., M201 Eng. Sci., El Paso, TX 79968-0520 USA

7075-T6 stringer ties were collected after long-term service in KC135 fuel tanker aircraft. Samples cut from the forgings were used in a study of retrogression and reaging (RRA) heat treatments. Hardness and conductivity varied with heat treatment as expected from the published literature. Potential-scanning polarization tests in 3.5% NaCl solution revealed that T6 (as received) samples were at or near a localized film-breakdown condition at their free corrosion potential. Samples given a T73-like post-aging were passive. After 5- to 60-minute retrogression, followed by 120°C reaging, samples showed a fragile passivity, with the free corrosion potential being only a few millivolts below localized film breakdown. Post-corrosion microscopy revealed that the breakdown potentials correspond to the onset of grain-boundary etching and intermetallic particle dissolution. These results provide new insights into the effects of heat treatments on corrosion and stress-corrosion resistance.

11:30 AM

Stress Corrosion Cracking of a-Ti in a Methanol Solution: *W. Y. Chu*[1]; H. Lu[1]; L. J. Qiao[1]; [1]University of Science and Technology, Dept. of Mat. Phys., Beijing 100083 China

Stress corrosion cracking of a-Ti in a methanol solution containing 0.6mol/L KCl was very high and an intergranular SCC fracture was obtained. As adding 10% H2O into the methanol solution, however, no SCC occurred. In situ TEM observation showed that corrosion process itself could enhance dislocation emission and motion, and microcrack of SCC initiated when the corrosion-enhanced dislocation emission and motion reached a certain condition. Why can the corrosion process itself facilitate dislocation and motion? a-Ti foil with a protective layer formed on one side was deflected during corrosion in the methanol solution, and then a tensile stress was generated at or near the metal/passive film interface. Adding 10% H2O into the solution, decreased the passive film-induced stress from 320 MPa to zero. Maybe the film-induced tensile stress is necessary condition for corrosion-enhanced localized plasticity, and then SCC.

Magnesium Technology 2001: Corrosion and Future Trends

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

Thursday AM
February 15, 2001

Room: 203-205
Location: Ernest N. Morial Convention Center

Session Chair: Gerald S. Cole, Ford Motor Company, Manuf. Sys. Dept., Dearborn, MI 48121 USA

8:30 AM

Measurement of the Corrosion Performance of Magnesium Alloys: *Guangling Song*[1]; David H StJohn[1]; Andrejs Atrens[1]; Li Zheng[1]; [1]University of Queensland, CRC for Cast Mets. Munuf. (CAST), Dept. of Min., Mins. and Mats. Eng., Brisbane, Queensland 4072 Australia

Due to a special electrochemical phenomenon for magnesium alloys called the negative difference effect, significant errors can be introduced into corrosion rate measurement when using traditional electrochemical techniques. Also, the classical weight-loss method only provides final corrosion information, and some experimental and theoretical errors are easily introduced into the final result during the removal of corrosion products and the corrosion rate calculation. This paper experimentally and theoretically demonstrates the use of a hydrogen evolution method of evaluating the corrosion performance of magnesium alloys. The advantages of this method include: 1) the amount of the collected hydrogen is equal to the amount of the dissolved magnesium through Faraday conversion; 2) the collection of evolved hydrogen can reveal the instantaneous corrosion rate as well as the final total amount of corroded magnesium alloy; 3) less theoretical and experimental errors are introduced by the hydrogen evolution collection method.

8:55 AM

Corrosion Behavior of Several Advanced Magnesium Alloys: *Michael J. Danielson*[1]; Russell H. Jones[1]; Eric A. Nyberg[1]; [1]Pacific Northwest National Laboratory, Mats. Res./Matls. Devel., P.O. Box 999; MSIN P8-15, Richland, WA 99337 USA

The corrosion behavior of Mg alloys is highly dependent on their impurities, inclusions and microchemistry and microstructure of the primary phases. New alloys are being developed to meet specific automotive needs that have compositions and microstructures very different from the well established AZ91D, AM60, etc. alloys. These modifications may greatly affect the corrosion behavior of these alloys. Therefore, the corrosion behavior of several die cast and semi-solid molded alloys have been measured and compared to their microchemistry and microstructure. The alloys evaluated were die cast AZ91D, AE42 and ZAC8506 and semi-solid molded AZ91D and ZAC8506. A JEOL 2010F high-resolution analytical/transmission electron microscope was used for the microchemistry and microstructure analysis. Electrochemical corrosion studies were performed to determine the anodic and cathodic polarization response and pitting potentials. Tests were conducted in neutral NaCl solutions at ambient temperature.

9:20 AM

Surface and Environmental Effects on the Fatigue Behavior of Wrought and Cast Magnesium Alloys: *Jens Wendt*[1]; Matthias Hilpert[1]; Jürgen Kiese[1]; Lothar Wagner[1]; [1]Technical University of Brandenburg at Cottbus, Chair of Phys. Metall. and Mats. Techn., P.O. Box 101344, Cottbus, Brandenburg 03013 Germany

The fatigue behavior of the extruded magnesium alloys AZ 31 and AZ 80 as well cast magnesium alloys AM 50 and AZ 91 was investigated. Fatigue tests were performed on hour-glass shaped specimens under fully reversed loading conditions (R = -1) in ambient air and in an aggressive NaCl solution. To study the effect of mechanical surface treatment on the fatigue life, shot peening and roller-burnishing were carried out and the results compared to an electropolished reference. In ambient air, shot peening and roller-burnishing led to a pronounced improvement in fatigue life. In the aggressive environment, only roller-burnishing markedly improved the fatigue life. The change in fatigue performance will be explained by the different surface layer properties such as surface topography, dislocation density and residual stresses as affected by shot peening and roller-burnishing results will be interpreted by the influence of there surface layer on fatigue crack nucleation and microcrack growth.

9:45 AM

Corrosion Fatigue of High Pressure Die Cast Magnesium Alloys: *W. George Ferguson*[1]; [1]University of Auckland, Chem. & Mats. Eng., Private Bag 92019, Auckland, New Zealand

Magnesium is the lightest of all the commercial metallic construction materials. With the increased emphasis on weight reduction in automobiles, magnesium is receiving much attention as a material for use on the next-generation automobiles, especially using die-cast components. Fatigue performance is one of the most important

mechanical properties of engineering materials. Magnesium alloys, which are used in the automotive industry, suffer from dynamic loading when in service. The purpose of determining the fatigue resistance of AZ91D and AM50 high pressure die casting alloys, which are the most popular magnesium alloys, is to obtain a reliable database for design, research and application of these materials. Standard Charpy sized test specimens (50´10´10) with smooth surfaces were used for fatigue testing using the three point bend method. S-N curves were determined for die cast specimens of Magnesium alloys AZ91D and AM50. The environments adopted were natural seawater, tap water and air. A difference in corrosion fatigue performance has been found, between AZ91D and AM50. AZ91D has better corrosion fatigue resistance in tap water than in seawater; conversely, AM50 has better corrosion fatigue resistance in seawater than tap water. The influence of frequency was also studied. A non-metallic coating produced electrolytically on magnesium alloys by the 'Anomag' process was used as a coating to improve corrosion protection and its effect on fatigue performance was investigated. In seawater the coating had little effect on the performance of AM50 but improved the fatigue performance of AZ91D. Key words: Magnesium alloys, AM50, AZ91D, high pressure die castings, fatigue and corrosion fatigue, anodized coatings.

10:10 AM Break

10:20 AM
Magnesium's Potential for Powertrain Components: *Naiyi Li*[1]; Jim E. Kearns[1]; Gerald S. Cole[1]; [1]Ford Motor Company, Manuf. Sys. Dept., 2101 Villiage Rd., SRL, MS 3011, Dearborn, MI 48121 USA

Magnesium alloys are being considered the most attractive lightweight metal as the auto industry searches for effective and low cost ways to reduce vehicle mass. In this paper, we will address the technical challenges, and functional and manufacturing issues of using magnesium die castings in powertrain applications. An example of converting a current production aluminum automatic transmission housing to magnesium will be discussed. We will also present the FEA results of filling and solidification and their impact on shape optimization.

10:45 AM
Round Table Discussion:
Magnesium Corrosion Issues That Concern Automotive Engineers: Gerald S. Cole, Ford Motor Company, Manufacturing Systems Dept., 2101 Village Rd., Dearborn, MI 48121 USA

Materials Issues in Microelectronics - II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Packaging and Interconnection Materials Committee

Program Organizers: Michael R. Notis, Lehigh University, Department of Materials Science, Bethlehem, PA 18015 USA; Sailesh Merchant, Lucent Technologies, Orlando, FL 32819 USA; Martin Weiser, Honeywell Electronic Materials, Spokane, WA 99216 USA; Jin Yu, KAIST, Department of Materials Science, Seoul, Korea

Thursday AM — Room: 226
February 15, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Martin W. Weiser, Honeywell Electronic Materials, Spokane, WA 99216 USA; Michael R. Notis, Lehigh University, Dept .of Mats. Sci. & Eng., Bethlehem, PA USA

8:30 AM Invited
Alpha Particle Issues in Microelectronics: *Martin W. Weiser*[1]; [1]Honeywell Electronic Materials, 15128 E. Euclid Ave., Spokane, WA 99216

Alpha particles and other radiation can cause soft errors in microelectronic circuitry. These soft errors do not cause permanent physical damage, but can cause incorrect calculations up to crashing the computer system. The problem has been recognized for many years, but has come to the forefront as the device sizes and operating voltages shrink and flip chip packaging takes off. This paper will describe how soft errors occur and what can be done to prevent or correct for them. The drive for more performance from microelectronic systems is forcing circuit designers to minimize software correction and push the problem to the materials engineers, particularly those involved in flip chip packaging. As a consequence the packaging materials community must now be concerned with radiation emitted by the materials in addition to the standard processing and reliability concerns.

9:00 AM Invited
Optical Packaging Using Improved Au-Based Solders: *Hareesh Mavoori*[1]; Ainissa G. Ramirez[1]; Sungho Jin[1]; [1]Bell Laboratories, Lucent Technologies, Appl. Mats. and Metall. Res., 700 Mountain Ave., Murray Hill, NJ 07974 USA

Eutectic Au-Sn and other Au-based solders are highly creep-resistant and hence desirable for optical packaging where dimensional stability is crucial. In optical device packages, even small, micron-scale misalignments between optical components such as semiconductor lasers/detectors and optical fibers/waveguides are sufficient to cause complete loss of communication signal transmission. We have developed new Au-based solders with enhanced mechanical, chemical and thermal properties for reliable bonding of optical components and devices. Additions of ternary alloying elements to eutectic and near-eutectic Au-Sn have been employed to achieve specific, desirable combinations of properties. Addition of Ga lowers the melting point and enhances the temperature-sensitivity of creep-resistance, yielding a solder that shows promise for forming creep-resistant, low-stress bonds for packaging stress-sensitive components. Alternatively, reactive elements can be added to Au-Sn solder to improve wettability of telecommunication optical fibers without the need for metallization such as electroless Ni or sputtered Ti/Pt/Au. The metallurgy, microstructure, mechanical properties and phase equlibria of these new solders will be presented and their technical implications discussed.

9:30 AM Invited
Microstructure Development in Ag-Cu-Sn Ternary Eutectic Solder Alloys: *Michael R. Notis*[1]; Daniel J. Lewis[1]; Sarah Allen[2]; [1]Lehigh University, Mats. Sci. & Eng., 442 Whitaker Lab., Bethlehem, PA 18015 USA

The morphology & microstructural stability of binary and higher-order eutectic alloys have been the subject of continued interest for many years. Models have been developed in the past to predict lamellar versus fibrous (rod-like) morphology in binary eutectics, based solely on interfacial energy arguments, and have been successful in correlating morphology-type with phase fraction in the eutectic (rod-like structures being favored at low phase fraction). We have extended this model to ternary eutectic systems, and we will demonstrate the applicability of this model to a study of microstructural stability during processing of alloys in the Ag-Cu-Sn solder system, as well as other lead-free and lead-containing ternary solders.

10:00 AM
Model Development of Base Metal Dissolution by Molten Solder: *Kenneth Erickson*[1]; Polly Hopkins[2]; Cynthia Hernandez[3]; Paul Vianco[3]; [1]Sandia National Laboratories, Dept. 9112, MS0834, P.O. Box 5800, Albuquerque, NM 87185 USA; [2]Sandia National Laboratories, Dept. 9117, MS0834, P.O Box 5800, Albuquerque, NM 87185 USA; [3]Sandia National Laboratories, Dept. 1835, MS1411, P.O. Box 5800, Albuquerque, NM 87185 USA

An understanding of base metal dissolution by molten solder is important towards the development of soldering processes. Intermetallic compound layer formation at the solder/metal interface as well as diffusion processes within the molten solder are critical mechanisms in the dissolution phenomenon. In order to develop an analytical model describing solder dissolution processes, a series of experiments were performed that documented the such processes between 100Au and 76Au-21Pt-3Pd (wt.%) base materials and 63Sn-37Pb and 50In-50Pb solders. The extent of base material loss into the solder as well as the growth kinetics of the intermetallic com-

pound layer were measured as a function of molten solder temperature and time of contact. The development of a computational model, and the use of the above data in that model, will be described. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US DoE under DE-AC04-94AL85000.

10:20 AM Break

10:40 AM

Kinetic Study of Solid State Reaction of Eutectic SnPb and Pb-Free Solders on Electroplated Cu UBM: *J. W. Jang*[1]; T. Y. Lee[2]; J. K. Lin[1]; D. R. Frear[1]; K.-N. Tu[2]; [1]Motorola, Semiconductor Products Sector, MD EL 725, 2100 East Elliot Rd., Tempe, AZ 85284 USA; [2]UCLA, Dept. of Mats. Sci. & Eng., Los Angeles, CA 90095-1595 USA

The kinetics of solid-state reaction of eutectic SnPb and a variety of Pb-free solders (Sn-3.5Ag, Sn-3.8Ag-0.7Cu, and Sn-0.7Cu) on electroplated Cu were investigated using optical microscopy. From the growth thickness of Cu3Sn and Cu6Sn5 intermetallics, the kinetic data such as the consumption thickness of Cu UBM, the activation energy, and diffusion coefficient for intermetallic growth were calculated. It was found that the solid state kinetics of Pb-free solder resulted in faster interfacial intermetallic growth than those of Pb-free solders. The difference is attributed to the presence of Pb. The diffusion mechanisms in Cu6Sn5 and Cu3Sn are also discussed in terms of microstructure of the intermetallics.

11:00 AM

Phase Coarsening During Static Annealing of Eutectic Pb-Sn Solder Joints: *Kang Jung*[1]; Hans Conrad[1]; [1]North Carolina State University, Dept. of Mats. Sci. and Eng., Raleigh, NC 27695-7907 USA

The coarsening of the Pb and Sn phases during the static annealing of 60Sn40Pb solder joints was investigated. The average phase sizes D and their distributions were determined at 50°C to 150°C for t=1 to 70 hr. Employing the time law $D^n-D^n_o=At$, it was found that n decreased with temperature from 5.5 at 50°C to 2 at 150°C without change in phase size distribution. The constant $A=_o\exp(-Q/RT)$ with Q=50±10 kJ/mole. Possible mechanisms are discussed.

11:20 AM

Microstructures and Joint Reliabilities of the Sn-3.5wt%Ag-X (X=In, Cu, Ni) Solder Alloys: *W. K. Choi*[1]; H. M. Lee[1]; [1]Korea Advanced Institute of Science and Technology, Cen. for Electr. Pack. Mats., 373-1 Kusong-dong Yusong-gu, Taejon, Korea

The Sn-3.5wt%Ag-X(X=In, Cu, Ni) solder alloys were designed through thermodynamic calculations. Indium was selected to lower the melting temperature of Sn-3.5Ag solder. Copper and Nickel were alloyed for improving the interfacial characteristics of Sn-3.5Ag solder. The microstructures and the thermodynamic behaviors of bulk solder alloys were investigated. To examine joint characteristics, the soldering was performed on various substrates at 250°C for 60 s followed by aging at 130°C for 100 and 400 hrs. The interfacial phenomena in solder joints were observed by XRD, SEM and EDX during soldering and aging, and after temperature cycles (-65°C~150°C). The solder joint strength was tested using the ball shear test for the jointed specimens after soldering and after temperature cycles. It was found that the interfacial characteristic has a major effect on the joint reliability. And the optimum solder composition could be presented.

11:40 AM

Metal/Polymer Interface and Solder Joint Reliability of a Wafer Level CSP: *H. Han*[1]; Jin Yu[1]; [1]KAIST, Center for Electro. Packag. Mats., 373-1 Kusong-dong Yusong-gu, Taejon, Korea

Wafer level chip scale package(WLCSP) has the highest potential compared with flip chip package(FCP), because they provide the benefits of real chip size package with low cost and high reliability. We use low modulus polymers(stress buffer layer; SBL) which were designed to relax the thermal strain generated at the solder joint. However, reliability of metal/polymer interface and solder joint are still critical problems to the WLCSP. We studied the reliabilities of metal/SBL interface and solder joint according to variation of the polymer surface treatments and the metal structure. To measurement of adhesion property on metal/SBL, peel strength was evaluated. In addition, the effects of under bump metallurgy(UBM) on the reliability solder joint at the WLCSP will be discussed.

Second Global Symposium on Innovations in Materials Process & Manufacturing: Sheet Materials: Numerical Modeling and Optimal Design

Sponsored by: Materials Processing and Manufacturing Division, Powder Materials Committee, Shaping and Forming Committee, Solidification Committee

Program Organizers: Mahmoud Y. Demeri, Ford Motor Company, Manufacturing System Department, Northville, MI 48167 USA; Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA; David L. Bourell, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA; Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; John Papazian, Northrup Grumman, Bethpage, NY 11714 USA; Klaus Siegert, University of Stuttgart, Institute for Metal Forming Technology, Stuttgart D-70174 Germany

Thursday AM — Room: 228
February 15, 2001 — Location: Ernest N. Morial Convention Center

Session Chairs: Michael Saran, Case Western Reserve University, Cleveland, OH 44106 USA; A. Sherif El-Gizawy, University of Missouri, Columbia, MI USA

8:30 AM

Impact of Advances in Computer Technology on Sheet Metal Forming Simulation: *Perry R. MacNeille*[1]; [1]Ford Motor Company, 2101 Village Dr., Dearborn, MI 48121 USA

Advances in computational technology such as mass-production, miniaturization and transmission technology are making computers faster, cheaper, smaller and more connected. Advances in computer hardware drive the advances in computer software that make it possible to harness the power of the new hardware. These advances lead to both qualitative and quantitative changes in computing that will impact most aspects of industry including the processing and manufacturing of sheet materials. This paper discusses some of these advances in computer technology and their consequences for the production of stamped sheet metal parts.

8:50 AM

Optimal Design of Materials Processing Based on Mechanics Fundamentals–Next Step Beyond Numerical Simulation: *Michael J. Saran*[1]; [1]Case Western Reserve University, Dept. of Mats. Sci. and Eng., 516 White Bldg., Cleveland, OH 44106-7204 USA

Currently, most often, design of the sheet metal forming process is performed by a trial & error procedure, where the trials are performed using stamping presses and/or numerical simulation. A current design is usually improved by evaluating the latest results and making design changes based on experience or intuition. The outcome is an acceptable but usually not optimal design. There is a number of drawbacks of such approach including non-optimal final design, long times required, and incorrect results when variations of multiple process parameters are considered. In this paper, an overview of pertinent optimal design algorithms will be presented. Then, the application of the selected approaches to optimal design of the sheet forming process will be introduced. Such algorithms will calculate the optimal values of process, material, and geometrical parameters and do so in a shorter time compared to the design by multiple simulations. The theory of optimal design will be illustrated by specific examples of sheet metal forming processes.

9:10 AM

A Finite Element Simulation System for Sheet Metal Forming Using Reconfigurable Tooling: *Elias L. Anagnostou*[1]; John M. Papazian[1]; [1]Northrop Grumman Corporation, Tech. Dev., South Oyster Bay Rd., M/S A01-26, Bethpage, NY 11714 USA

A simplified front end for a finite element simulation system for prediction of a springback correct die shape has been developed for the sheet metal stretch forming process. This system has been designed for use in conjunction with a "discrete-die" reconfigurable tool. The simulation system takes into account the deformation of the unique compliant layer that is required for use with a discrete die. The system is highly automated, and is designed to be used by a tool design engineer with little finite element modeling experience. The system performs initial forming checks to determine if the part can be formed with a particular reconfigurable tool, it then prepares and submits a finite element model of the process, and retrieves and analyzes the output of the calculation. A die design algorithm is incorporated for prediction of the springback corrected die shape. The predicted die shape and the desired forming trajectory are then downloaded directly to a web-server database.

9:30 AM

Effect of Global and Local Stiffness on Blankholder Pressure in Draw-Die Forming: *Matthew Dingle*[1]; John Duncan[2]; Michael Cardew-Hall[3]; Peter Hodgson[4]; [1]Ford Motor Company of Australia/STAMP Research Group, CAE, Prod. Dev., P.O. Box 14, Geelong, Victoria 3220 Australia; [2]University of Auckland; [3]Australian National University; [4]DeakiN University

Elastic deformation in mechanical presses will influence the blankholder pressure during the operating stroke. Quantitative information is sparse in the literature and in most cases of die design, the effect is neglected. In this paper, the global effects are analysed by reducing a typical double-acting press to relatively few kinematic, rigid or elastic elements. Data from a particular press, including measurement of force in the outer slide and main ram, are used to calibrate this model, which is found to explain the observed blankholder force signature. Variation of pressure at any instant around the binders is also an important die design consideration. Simple analytical models show that the pressure distribution can be controlled by local variation of stiffness of the structure supporting the binder surface. These calculations are in agreement with more detailed finite element analysis of actual die structures. The results of this work support the view that, if incorporated in the regular die design process, quite simple stiffness analysis could significantly improve stamping performance and reduce die try-out time.

9:50 AM Break

10:10 AM

Computer Prediction of Dent Resistance of Automotive Steel Panels: *Naji Arwashan*[1]; Maurice M. Lou[2]; [1]LTV Steel Company, Auto. Dev. Grp., 2000 Town Ctr., Ste. 540, Southfield, MI 48009 USA; [2]Ford Motor Company, FEA and Die Stnds., Mail Drop 268 Cubicale GB-N78, 20910 Oakwood Blvd., Dearborn, MI 48121 USA

Dent Resistance of exterior steel body panels is an increasingly important issue in light of the current trend toward downgaging for achieving weight reduction. Predicting dent behavior of panels at the early stage of tools and die design can help save a lot of time and money. Dent resistance depends among other things on the geometry of the panel and on the strains caused by the forming operation. Hence, it is almost impossible for a simple formula to predict dent resistance for automotive panels. It is only by finite element analysis that the complexity in geometry and in forming process can be analyzed. The behavior of steel panels in resisting denting is significantly improved by the work hardening of the steel during the forming operation. A key to a successful prediction of denting is to know the distribution of the strains in the panel after forming. Therefore, simulation of dent behavior cannot be accurately performed without first simulating the forming of the panel. Explicit finite element codes are widely used for forming simulation. However, due to the dynamic nature of these codes, they are not suited to simulate quasi-static dent resistance. Only implicit finite element codes can simulate accurately the quasi-static denting problem. LS-DYNA, with its explicit and implicit solvers was used to perform seamless simulation of forming and denting. The computer simulation of dent resistance of an automotive door panel was compared with experimental results. The good agreement between simulation and experimental results supports the applicability of the approach used in this investigation.

10:30 AM

Material Variations and Springback: A Stochastic Approach: *Cedric Xia*[1]; [1]Ford Motor Company, Scien. Res. Lab., MD3135/ SRL, P.O.Box 2053, Dearborn, MI 48121 USA

A robust stamping process has to have the cabability to tolerate inherent variations of both material properties and process parameters. This study attempts to examine the effect of material property variations on the springback behavior from a stochastic point of view. A plane strain flanging problem is employed to facilitate the study. Deterministic solution of the springback is first derived analytically under pure bending assumption. Random distribution of such material properties as the yield strength, hardening coefficient, and blank thickness is then introduced. A Monte Carlo simulation is performed to evaluate its springback response. Emphasis is placed on the response variance and their correlations. The level of quality control required for a robust stamping is discussed.

10:50 AM

Improvements in Numerical Prediction of Springback Aided by The Split Ring Benchmark Test: *Michael J. Saran*[1]; Mahmoud Y. Demeri[2]; Nana Andoh[1]; [1]Case Western Reserve University, Dept. of Mats. Sci. and Eng., 516 White Bldg., Cleveland, OH 44106-7204 USA; [2]Ford Motor Company, MM, 2101 Village Rd., P.O. Box 2053, MD 3135, SRL, Dearborn, MI 48121-2053 USA

Springback is one of the key factors influencing quality of the sheet metal forming process. Therefore, an accurate and reliable prediction of springback would be of great value for the part & process designer to arrive at ever-tighter targets of dimensional and surface quality, and stamping robustness. Recently, a new Split Ring Benchmark Test for springback simulation was developed to aid in validation of numerical predictions. The test encompasses complex forming modes, but avoids problems with measurement fixtures, and provides considerably improved measurement accuracy and reduced experimental error. The preliminary simulations using the "general purpose" numerical and materials settings showed a good qualitative agreement with the test results and a reasonable prediction of trends for steel and aluminum. It also indicated that a further study to obtain an improved quantitative agreement with the test results is needed. Aided by the test results, this paper investigates sources of approximation in the numerical models and attempts to identify which of the problem description parameters are important for a good springback prediction. Selected numerical, material, and process parameters are considered.

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Thermo-Mechanical Coupling Finite Element Analysis of Non-isothermal Sheet Extrusion Process: *Zhanghua Chen*[1]; T. C. Lee[1]; C.Y. Tang[1]; [1]The Hong Kong Polytechnic University, Dept. of Manuf. Eng., Kowloon, Hong Kong, China

In sheet metal forming process, the forming limit and strain distribution are governed by plastic instability and fracture following strain localization. It has been proved that the temperature gradient caused by plastic deformation as well as friction is one of crucial factors to induce the strain localization in high-speed metal forming processes. In this paper, a numerical simulation of the sheet metal extrusion process has been conducted by using thermal-mechanical coupling finite element method. An improved mixed finite element method has been used to solve the elasto-plastic problem. In thermal phase, the transient heat transfer finite element method together with the Crank-Nicholson algorithm has been employed to determine the temperature field. A comparison with the experimental result revealed that the temperature gradient plays an important role in inducing the strain localization which led to fracture failure in the sheet metal extrusion process.

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