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) _____

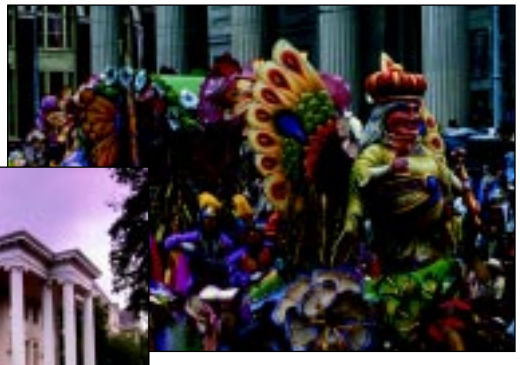
Signature: _____

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 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.

10:10 AM Break

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.

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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.