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 131st Annual Meeting & Exhibition at the non-member fee will automatically receive a one-year complimentary introductory membership for 2002. 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 castello@tms.org or via phone to Margie Castello (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

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

TMS2002
131st Annual Meeting & Exhibition

Advance Registration Deadline: January 28, 2002

AM02-PDF

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. Title: _____

_____
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 1/28/02)	On-Site Fees (after 1/28/02)
☐ Member	$395 M	$495 ML
☐ Non-Member Author	$395 NMA	$495 NMAL
☐ Non-Member *	$520 NM	$620 NML
☐ Student Member ##	$0 STU	$0 STUL
☐ Student Non-Member ## *	$25 STUN	$25 STUNL
☐ TMS Retired Member	$220 RM	$220 RML
☐ Exhibit Booth Personnel	$0 E	$0 EL
☐ Exhibit Only	$35 EO	$35 EOL

* Includes TMS membership for 2002
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.

Qty.	Order Number and Title	Book Price	Shipping*	Total
___	#514X CALPHAD and Alloy Thermodynamics	$72	+_____	=_____
___	#5158 Creep Deformation: Fundamentals and Applications	$105	+_____	=_____
___	#5166 EPD Congress 2002	$96	+_____	=_____
___	#5182 High-Cycle Fatigue, the David L. Davidson symposium	$110	+_____	=_____
___	#5190 Light Metals 2002 (Book and CD-ROM set)	$158	+_____	=_____
___	#5247 Magnesium Technology 2002 (Book and CD-ROM set)	$111	+_____	=_____
___	#5204 Science and Technology of Interfaces, International Symposium in Honor of Dr. Bhakta Rath (PDF)	$122	+_____	=_____
___	#5255 Sulfide Smelting 2002	$70	+_____	=_____
___	#5212 Surface Engineering: Science and Technology II (PDF)	$111	+_____	=_____
___	#5220 High-Performance Metallic Mat'ls for Cost-Sensitive Applications (PDF)	$117	+_____	=_____
___	#5239 Ultrafine Grained Materials II	$125	+_____	=_____
___	#5131 Computational Modeling of Materials Minerals, and Metals Processing	$129	+_____	=_____

* Shipping: in the United States–$10 per item • all other countries–$20 per item

Tutorial Luncheon Lecture Tickets

Optional Box Lunches	Fee	No.	Total
Monday 2/18/02 (sponsored by Young Leaders)			
Young Leaders Tutorial Box Lunch	$25	_____	$_____

Refund policy: Written requests must be mailed to TMS, post-marked no later than January 28, 2002. A $50 processing fee will be charged for all meeting registration cancellations in addition to all short course registrations cancellations.

Continuing Education Short Courses:

	Advance Fees (until 1/28/02)	On-Site Fees (after 1/28/02)
Aluminum Smelter Cell Dynamics		
☐ Member	$645	$695
☐ Non-Member	$735	$785
Texture and Anistrophy for the Materials Professional		
☐ Member	$395	$475
☐ Non-Member	$445	$525
Computational Modeling Techniques and Applications for Materials Processing		
☐ Member	$395	$475
☐ Non-Member	$445	$525

Social Function Tickets:

	Fee	No.	Total
Monday 2/18/02			
Bhakta Rath Honorary Dinner	$60	_____	$_____ RD
Charles McMahon Honorary Dinner	$60	_____	$_____ MD
David L. Davidson Honorary Dinner	$35*	_____	$_____ DD
* A portion of each ticket is being subsidized by the organizing committee			
Tuesday 2/19/02			
TMS Banquet	$60	_____	$_____ AD
Tables of 8	$480	_____	$_____ AD8
Table Sign to Read _____			
Extraction & Processing Division Luncheon	$30	_____	$_____ EP
Tables of 8	$240	_____	$_____ EP8
Table Sign to Read _____			
Wednesday 2/20/02			
Light Metals Division Luncheon	$30	_____	$_____ LM
Tables of 8	$240	_____	$_____ LM8
Table Sign to Read _____			

Plant Tour:

Thursday 2/21/02	Fee	No.	Total
Boeing 747/767/777	$39	_____	$_____BT

2002 Membership Dues—for current TMS members only:

☐ 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: _____

TMS2002

131st Annual Meeting & Exhibition

February 17-21, 2002 • Seattle, Washington, USA

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

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

Reservations must be received at Travel Planners by: January 18, 2002

Arrival Date _____ Departure Date _____

Last Name _____ First Name _____ MI _____

Company _____

Street Address _____

City _____ State/Country _____ Zip/Postal Code _____

Daytime Phone _____ Fax _____

Additional Room Occupants _____

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

Non-Smoking Room Requested _____ Special Needs _____

 # after hotel correspondes with map below.

HEADQUARTERS
Sheraton Hotel • 1
$175/single • $175/double

W Hotel Seattle • 7
$179/single • $199/double

Renaissance Madison • 5
$149/single • $149/double

West Coast/Grand • 8
$135/single • $135/double

Hilton Seattle • 3
$140/single • $140/double

Mayflower Park Hotel • 4
$140/single • $140/double

West Coast/Roosevelt • 9
$132/single • $132/double

Westin Seattle • 11
$165/single • $165/double

Crowne Plaza Seattle • 2
$134/single • $134/double

Summerfield Suites Wyndham • 6
$119/single • $129/double

West Coast/Vance • 10
$125/single • $125/double

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

1. _____
2. _____
3. _____

Type of Accomodations: (check one)
☐ Single 1 person/1bed ☐ Double 2 people/1bed ☐ Twin 2 people/2 beds
☐ Triple 3 people/2 beds ☐ Quad 4 people/2 beds

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

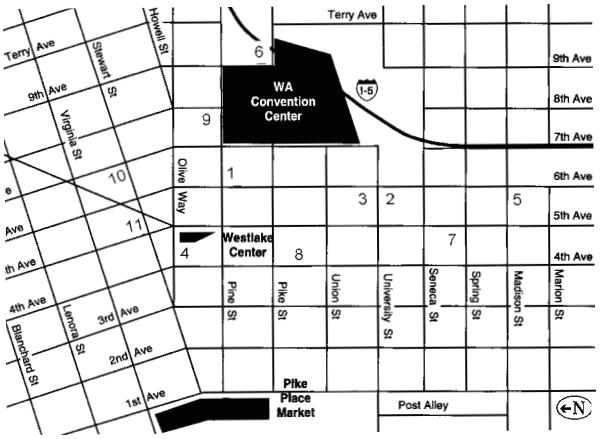
TMS has contracted a block of rooms at the headquarters hotel, Sheraton Seattle Hotel & Towers, along with each of the hotels, and therefore has assumed a financial liability for any and all rooms in that block that are not reserved. You are strongly encouraged to reserve your room(s) at the hotels listed to limit our financial liability. Please help TMS achieve overall success with the 131st TMS Annual Meeting & Exhibition by making your reservation at one of the listed hotels prior to the advance housing deadline. Thank you.

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

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

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

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



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

Account Number _____ Expiration Date _____

Card Holder Name _____ Authorized Signature _____

ACCOMPANYING PERSONS TOUR REGISTRATION FORM

TMS2002
131st Annual Meeting & Exhibition

February 17-21, 2002 • Seattle, Washington, USA

Convention Services Northwest has arranged tours for attendees/guests.

Please make your reservation by noting choice of tour. Pre-sold tickets will be held at the tour desk located in Hall 4C of the Washington State Convention and Trade Center. Please arrive 15 minutes prior to departure time.

Registration:

To register for the tours, please complete and return the registration form along with full payment to:
Convention Services Northwest, Attn: Becky Haiduck
Tower Building/Suite 1414,1809 Seventh Avenue, Seattle, WA 98101, USA
PHONE: (206) 292-9198 • FAX: (206) 292-0559
*** PLEASE CHOOSE ONLY ONE OPTION FOR SENDING FORM ***

Description	Date/Time	Price	No.	Amount
Sample Seattle – A Deluxe City Tour	Monday, February 18, 2002 – 9:00am-12:30pm	$28	_____	$_____
Historic Port Townsend Day Trip	Tuesday, February 19, 2002 – 9:00am-4:30pm	$59	_____	$_____
Wine Tasting Tour	Wednesday, February 20, 2002 – 1:00pm-5:00pm	$39	_____	$_____

Total $_____

Last Name _____ First Name _____ MI _____

Street Address _____

City _____ State/Country _____ Zip/Postal Code _____

Daytime Phone _____ Fax _____

E-mail _____

☐ I have special needs, disabilities or dietary concerns. Please have someone from Convention Services Northwest contact me.

Payment options:

☐ Check or Money Order Enclosed (remit in US funds)

☐ American Express ☐ MasterCard ☐ VISA ☐ Discover ☐ Diners

Account Number _____ Expiration Date _____

Card Holder Name _____ Authorized Signature _____

Reservations:

Reservations must be received by January 16, 2002. Tickets may be purchased on-site on a space-available basis only and tickets will include an additional $2.00 charge. You may pick up your tickets at the Washington State Convention and Trade Center in Hall 4C staring at 11:00am on Sunday, February 17, 2002.

Refunds:

Refunds for cancellations will be made if written request is received at the Convention Services Northwest office by January 16, 2002, less a $5.00 handling fee per ticket. After January 16, 2002, no refunds or exchanges can be made. If minimum registration on a tour is not met, Convention Services Northwest will refund the cancelled tour or apply the refund towards another tour.

For Office Use Only:

Deposit No. _____ Amount $ _____

Check No. _____ Inn. _____

CONFERENCE PROCEEDINGS

THE FOLLOWING TITLES WILL BE AVAILABLE AT THE MEETING. RESERVE YOUR COPY USING THE TMS ANNUAL MEETING REGISTRATION FORM.

CALPHAD and Alloy Ther modynamics

Patrice E.A. Turchi, Antonios Gonis, and Robert D. Shull, editors

These proceedings emphasize all theoretical aspects of computational thermodynamics and kinetics and their impact on the science of alloys and materials design. The book will provide an assessment of the CALPHAD (Calculation of Phase Diagrams) approach pioneered by 2002 Hume-Rothery Award recipient Larry Kaufman, a review of the current status of the software applications based on the CALPHAD approach, the impact of CALPHAD on alloy thermodynamics and design, and future prospects. The book will provide a detailed picture of the development of CALPHAD and point to further research.

ISBN 0-87339-514-X

Approx. 390 pp., illus., index, hardcover

Order No. 514X

Member price: $72

Computational Modeling of Materials, Minerals, and Metals Processing

M. Cross, J.W. Evans, and C. Bailey, editors

This set contains the proceedings, in both print and CD-ROM formats, of the Computational Modeling of Materials, Minerals, and Metals Processing Symposium to be held at the 2002 TMS Annual Meeting in Seattle, Washington.

Computational models offer an effective way to design, analyze, and optimize materials, metals, and minerals processing. Computational modeling is increasingly becoming the means by which every factor in the operation of a process can be analyzed in a rational manner. Such models also represent a framework for much of our understanding of process operations.

This international symposium brings together participants from all aspects of computational modeling of materials, minerals, and metals processing, including the engineers involved primarily in specific applications, developers of computational modeling tools, and the developers of models. This allows all involved to understand the most recent advances in this rapidly developing enabling technology.

ISBN 0-87339-513-1

Approx. 700 pp., illus., index, hardcover & CD-ROM

Order No. 5131

Price: $129

Creep Defor mation: Fundamentals and Applications

Rajiv S. Mishra, James C. Earthman, and Sai V. Raj, editors

These proceedings focus on the application of fundamental creep research to the design and development of high temperature materials for engineering applications. The book deals with all aspects of creep deformation and high temperature materials development, specifically the influence of microstructures on various aspects of creep and the application of this information in the design of highly creep resistant materials. Emphasis will be placed on advanced ceramic and metal matrix composites and advanced intermetallics.

This volume brings together researchers working on fundamental issues relating to the development and characterization of high temperature materials and design engineers involved in high temperature applications.

ISBN 0-87339-515-8

Approx. 415 pp., illus., index, hardcover

Order No. 5158

Member price: $105

Light Metals 2002

Wolfgang Schneider, editor

Held at the TMS Annual Meeting & Exhibition each year, the Light Metals series has become the definitive annual reference source in the field of aluminum production and related light metals technologies. Each volume contains complete coverage of advancements and current work in cast shop technology, alumina and bauxite, carbon technology, aluminum reduction technology, and recycling. In addition, Light Metals 2002 includes coverage of reactive metals and advances in molten salt processing technology. Light Metals 2002 is sold as a package that includes the proceedings in both hardcover and text-searchable CD-ROM formats.

ISBN 0-87339-519-0

Approx. 1200 pp., illus., index, hardcover & CD-ROM

Order No. 5190

Member price: $158

EPD Congr ess 2002

Patrick R. Taylor, editor

The Extraction & Processing Division Congress, held at the TMS Annual Meeting & Exhibition each year, has become the definitive annual forum for new technological developments in the process metallurgy community. This volume will also include the proceedings of the Fundamentals of Advanced Materials for Energy Conversion symposium.

In addition to general abstracts, this year's edition will include papers on:

- Modeling of high temperature alloy processing
- General pyrometallurgy
- Generation, treatment, metal recovery, and disposal of flyash
- Imaging of dynamic processes
- Materials processing fundamentals
- General recycling of materials

ISBN 0-87339-516-6

Approx. 812 pp., index, hardcover

Order No. 5166

Member price: $96

High-Cycle Fatigue, the David L. Davidson Symposium

Kwai S. Chan and Peter K. Liaw, editors

These proceedings from the David L. Davidson Symposium on High-Cycle Fatigue review the current research activities and assess the state-of-the-art in high-cycle fatigue, which has become a major concern in the design of engineering components and structures. This volume will include papers on a basic understanding of failure mechanisms, experimental methods and studies, modeling and simulation, and life-prediction methodology. This volume will also contain papers from the Fatigue of High Temperature Alloys Symposium, which deals with fatigue behavior of high temperature alloys, including crack initiation and propagation modes.

ISBN 0-87339-518-2

Approx. 350 pp., index, hardcover

Order No. 5182

Member price: $110

High Per formance Metallic Materials for Cost-Sensitive Applications

F.H. (Sam) Froes and Lu Li, editors

These proceedings will examine the most recent advances and best practices in structural materials selection, design, and manufacturing for producing affordable components, with a focus on titanium, aluminum, and other advanced metallic materials. This volume will discuss melting, casting, powder metallurgy, forging, forming, extrusion, and machining, as well as processing advances, innovative processing techniques, process modeling and materials by design, new alloys, and related processing-microstructure-properties-performance-cost studies.

ISBN 0-87339-522-0

Approx. 295 pp., index, PDF publication

Order No. 5220

Member price: $117

Sulfide Smelting 2002

Robert L. Stephens, and H.Y. Sohn, editors

These proceedings focus on all aspects of the pyrometallurgical production of primary metals from sulfide concentrates. Papers discuss industrial operations producing copper and nickel and the direct production of lead and zinc. Key topic areas include smelting and converting processes, recently completed capital projects, current operating practices, the predicted future of sulfide smelting operations, furnace integrity and refractory design, gas handling processes and equipment, issues related to the treatment of high-strength sulfur dioxide-containing off-gases, the production of alternative sulfur products, the treatment of acid plant blowdown streams and sulfated smelter dusts, and the capture and treatment of fugitive emissions.

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Advances in Metallic Glasses: Glass Crystallization - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, *Program Organizers:* K. F. Kelton, Washington University, Department of Physics, St. Louis, MO 63130 USA; A. L. Greer, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3QZ UK; Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

Tuesday AM Room: 212
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: K. F. Kelton, Washington University, Dept. of Phys., Campus Box 1105, One Brookings Dr., St. Louis, MO 63130-4899 USA; Daniel Sordelet, Ames Lab./ Iowa State University, Metall. & Cer./Dept. of Matls. Sci. & Eng., 107 Metals Dvlp., Ames, IA 50014 USA

8:30 AM Invited
Time-Resolved Studies of Ti-Zr-Cu-Ni-(Si) Devitrification using High Temperature X-Ray Powder Diffraction: *Matthew J. Kramer*[1]; Dan J. Sordelet[1]; Matthew Besser[1]; Elena Rozhkova[1]; [1]Iowa State University, Ames Lab., 37 Wilhelm Hall, Ames, IA 50011 USA

Time-resolved devitrification studies of $Ti_{34-x}Cu_{47}Zr_{11}Ni_8Si_x$ metallic glasses were performed using a high temperature XRD furnace. Samples included powders produced by high pressure gas atomization and surface coatings deposited by air plasma spraying. Using synchrotron radiation at the Advanced Photon Source at Argonne National Laboratory and image plates, spectrum covering wave momentum (Q) > 6 can be taken < 2 s. This technique was used to follow the devitrification of samples during heating at 40 K min^{-1} between 623 and 1073 K. The crystallization behavior observed with structural diffraction data compare well with results from thermal analysis using differential scanning calorimetery. At 1073 K, these amorphous alloys evolve to a four phase microstructure which includes phases that are closely related to $Cu_{51}Zr_{14}$, CuTi and Cu_2TiZr. The work at Ames Laboratory was supported by USDOE-BES under W-7405-ENG-82, at the MUCAT, APS/ ANL under contract N. W-31-109-Eng-38.

9:00 AM
Primary Crystallization in (Fe, Ni)-Based Metallic Glasses: *A. L. Greer*[1]; I. T. Walker[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

A wide variety of Fe-Ni-B and Fe-Ni-Zr-B compositions have been melt-spun into fully glassy ribbons. The glasses which show clear primary crystallization have been more closely studied. It is known from studies of droplet solidification that the bcc phase nucleates more easily than the ccp phase, with strong effects on phase selection. The preference for bcc is confirmed in devitrification and is analysed in detail. These studies are also complicated by phase transformations within the primary phase. The addition of Zr greatly refines the devitrification microstructure and permits studies of the effects of crystallite size on mechanical properties. Hardening mechanisms are discussed.

9:20 AM
Crystallization Behavior of Nd-Fe-B-Based Alloys: *Branden B. Kappes*[1]; Jeffrey E. Shield[2]; [1]University of Utah, Matls. Sci. & Eng., 122 S. Central Campus Dr., Rm. 304, Salt Lake City, UT 84112 USA; [2]University of Nebraska-Lincoln, Mechl. Eng., N104 Walter Scott Engineering Ctr., PO Box 880656, Lincoln, NE 68588-0656 USA

Devitrification of Nd-Fe-B alloys is a popular route to the nanocrystalline grain structure required for high performance permanent magnets. Alloying additions are utilized to enhance the scale and uniformity of the grain structure in both the as-solidified and crystallized material. However, the alloying additions also significantly alter the crystallization kinetics. Crystallization of stoichiometric $Nd_2Fe_{14}B$ alloys displayed conventional behavior, with an Avrami exponent, n, close to 4. TiC additions stabilized the glassy phase and contributed to a more uniform crystallized grain structure. The time to complete crystallization was extended, implying a change in crystallization kinetics. However, the behavior remained consistent with n close to 4. Anomalous behavior with overtly large n was observed for multicomponent alloys with substitution additions. For example, a nine-component alloy containing Dy, Pr and Co substitution additions displayed markedly different kinetics. EDS results, which showed partitioning of Ti and C, interfacial enrichment of the rare-earths, and interfacial depletion of Fe, supported the crystallization of two immiscible phases. The crystallization kinetics, results investigating partitioning during crystallization, and the effects of alloy composition on the developing nanostructure will be discussed.

9:40 AM
The Nucleation of Al Nanocrystals during the Devitrification of Amorphous Al Alloys: *Jonathan E. Spowart*[2]; Daniel B. Miracle[1]; Herbert M. Mullens[3]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA; [3]Southern Ohio Council of Higher Education, 3171 Research Blvd., Ste. 141, Dayton, OH 45420 USA

The first step in the crystallization of many amorphous Al alloys consists of the formation of an exceptionally high volume density (~10E+21/m3) of nanocrystalline Al precipitates. In this study, the nucleation process has been modeled by considering a random distribution of solute atoms at a representative bulk atomic concentration. The volume density of potential nucleation sites is then estimated by determining the number of locations in the modeled volume within which a critical nucleus can be placed, without overlapping any solute atoms. The size of the critical nucleus is varied in the model from 1-1000 atoms. A statistical approach is adopted, since a very large system size is required for a reasonable number of nucleation events. The results show that the measured volume density of Al precipitates is predicted for a critical nucleus of 30-60 atoms, which is in reasonable agreement with suggested values in the literature of ~100 atoms. The influence of non-random distributions of solute atoms, motivated by the preference for formation of the stable intermetallic phases, will be discussed.

10:00 AM Break

10:20 AM Invited
Primary Crystallization in Amorphous Al-Base Alloys: *John H. Perepezko*[1]; Rainer J. Hebert[1]; Robert I. Wu[3]; Gerhard Wilde[2]; [1]University of Wisconsin, Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706 USA; [2]Forschungszentrum Karlsruhe, INT, PO Box 3640, Karlsruhe D-76021 Germany; [3]Intel Corporation, Portland, OR USA

An important characteristic of the new marginal glass forming alloys, that include amorphous Al-Base alloys, is the development of a high number density of Al nanocrystals during initial devitrification. The observed nanocrystal densities can range from 10E21 to 10E23 m-3 and display a remarkable thermal stability that is reflected in a wide separation of 75°C or more between the primary and final crystallization reactions. Isothermal crystallization studies based upon nanocrystal size distribution and microcalorimetry heat flow measurements confirm that the nanocrystal dispersions develop by a heterogeneous mechanism that appears to be related to a site density retained from melt quenching. Annealing experiments at temperatures spanning the glass transition as well as the use of incorporated nucleation catalysts and deformation treatments have revealed new aspects of the crystallization kinetics and strategies for the control of the nanocrystal density during microstructure evolution. The support of the ARO (DAAD19-01-1-0486) and the DFG (GW, WI 1899/1-1) is most gratefully acknowledged.

10:50 AM
Mechanisms for Nano-Crystal Formation in Metallic Glasses: *K. F. Kelton*[1]; T. K. Croat[1]; A. K. Gangopadhyay[1]; A. L. Greer[2]; M. Weyland[2]; X. Li[3]; K. Rajan[3]; [1]Washington University, Dept. of Phys., CB 1105, One Brookings Dr., St. Louis, MO 63130 USA; [2]University of Cambridge, Dept. of Matls. Sci. & Metall., Cambridge CB2 3QZ UK; [3]Rensselaer Polytechnic Institute, Dept. of Matls. Sci. & Eng., Troy, NY 12180 USA

Possible mechanisms for nano-crystal formation in metallic glasses are discussed, focusing primarily on the Al-transition metal-rare earth glasses. Transmission electron microscopy (TEM) data are presented that provide the first demonstration of nano-scale phase separation prior to crystallization in $Al_{88}RE_8Ni_4$ glasses. Although phase separation is commonly observed in bulk metallic glasses, the high Al concentration make it unexpected in the AlRETM glasses. TEM observations, and modeling studies of the devitrification kinetic data for some of these glasses, are presented that demonstrate preferential nucleation of nano-crystal α-Al grains near the boundaries of the phase separated regions. Preliminary studies show no evidence for phase separation in Al-RE-Fe glasses, which also crystallize to a nanoscale

microstructure. A new model for homogeneous nucleation, coupling the interfacial and the long-range diffusion fluxes, is advanced to explain this.

11:10 AM
Microstructural Implications of Non-Random Nucleation Protocols in Nanocrystallized Metallic Glasses: *Eloi Pineda*[1]; Daniel Crespo[2]; [1]Universitat Politecnica de Catalunya, ESAB, Urgell 187, Barcelona 08036 Spain; [2]Universitat Politecnica de Catalunya, Fisica Aplicada, Escola Politecnica Superior de Castelldefels, Av. del Canal Olimpic s/n, Castelldefels, Barcelona 08860 Spain

Macroscopic properties of nanocrystallized metallic glasses are determined by its nanostructure. The knowledge of the crystallization kinetics and its effect on the nanostructure are then essential in designing production and annealing protocols. Deviations of the Avrami kinetics in many of such systems are interpreted by means of either nonrandom nucleation or soft impingement due to overlapping concentration gradients. In this work, simple simulations of nonrandom nucleation processes allow us to evaluate the main features of both their kinetics and nanostructure. It is shown that nonrandom nucleation highly affects the nanostructure while has a reduced effect on the transformed fraction evolution, and that the decreasing Avrami exponents reported in many systems have to be associated to a time dependent growth rate. Moreover, as similar kinetic behaviors are observed for different kinds of nucleation and growth protocols the comprehension of the kinetics-nanostructure relationship became fundamental in studying such systems.

11:30 AM
Mechanism of Ultrafine Nanostructure Formation in a Bulk Amorphous Zr-Based Alloy: *Helmut Hermann*[1]; [1]Institute of Solid State & Materials Research Dresden, PF 27 01 16, Dresden D-01171 Germany

The bulk amorphous Zr54.5Ti7.5Al10Cu20Ni8 alloy transforms into an ultrafine nanostructured state during annealing just below the glass transition temperature. Differential scanning calorimetry, X-ray diffraction, high resolution electron microscopy, small-angle neutron scattering, and thermomechanical analysis are used to study this process. The experimental results are consistent with the following model. Structural fluctuations on a length scale below 1nm characterize the initial state. Domains of comparatively well developed local order grow during annealing by local cooperative re-ordering. Impinging domains form boundary regions where enhanced diffusion becomes possible. At high packing fraction of partially ordered domains the boundaries generate an interconnected system and a percolation thresholds occurs initiating diffusion. The final diameter of the crystallites is about 2.5nm.

Alumina and Bauxite: Hydrate and Alumina

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Jacques M. Mordini, Aluminium Pechiney, Gardanne, Cedex 13541 France; Steve Rosenberg, Worsley Alumina Pty, Ltd., Process Chemistry Group, Collie, West Australia 6225 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday AM — Room: 609
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: TBA

8:30 AM
Nearly 30 Years of Experience with Lurgi Calciners and Influence Concerning Particle Breakage: *Eberhard Guhl*[1]; Rolf Arpe[1]; [1]Aluminium Oxid Stade GmbH, Johann-Rathje-Köser-Strasse, Stade 21683 Germany

Following the development of the circulating fluid bed calciners (CFB) at the Nabwerk and Lünen VAW plants, in 1973 AOS became the first alumina plant in the world to 100% employ this technique. AOS installed three calciners with a designed capacity of 650 t/d each. Since AOS was built as floury type alumina plant, the demand of the smelters needed several modifications of the alumina quality. The goals, a coarse material and lower particle breakage, were met by several modifications. This paper will present the modification of the calciners and the influence on the hydrate quality regarding particle breakage.

8:55 AM
Evaluation of the Precipitation Circuit Operation Parameters to Control the Alumina Attrition Index: *Enio Beltran Rodriguez*[1]; *Roco José Mendoza*[1]; [1]C.V.G. Bauxilum, Control de Calidad y Proceso, Zona Industrial Matanzas, Puerto Ordaz, Bolívar 80154 Venezuela

The alumina granulometry is of vital importance in the quality characteristics required by the client. The attrition index in the hydrate particles obtained during the Bayer process has marked influence on the results of final product granulometry. At C.V.G. Bauxilum has been possible to reduce by 52% the alumina attrition index in the last decade, reducing from 23% to 11%, by means of controlling the precipitation circuit operation parameters. These parameters are directly related with the saturation of the mother liquor, where the mother liquor temperature and molar ratio stand out, combined with the quantity and grain size of the fine seed to feed to the agglomeration phase.

9:20 AM
Effect of Precipitating Condition and Additives on the Attrition-Resistance Property of Hydroxide Alumina: *Xie Yanli*[1]; Zhao Qun[1]; Bi Shiwen[1]; [1]Northeastern University, Sch. of Matl. & Met., No.11, Ln. 3 Wenhua Rd., Heping Dist., PO Box 117, Shenyang, Liaoning 110004 China

Improving the attrition-resistance property of gibbsite is the critical question to produce sandy alumina in alumina refinery. The effects of some precipitation condition such as temperature system, seed content and particle size of seeds were researched in this paper. It was demonstrated that moderate preliminary temperature, seed content and suitable percentage of fine particles in seed were propitious to get high attrition-resistance property hydroxide alumina. In addition, the effect of a certain additive and its adding method on the intensity of gibbsite was discussed in detail. All results were explained by analyzing the data obtained and comparing the SEM photographs of products which showed that gibbsite with inlaid structure had high attrition-resistance.

9:45 AM Break

10:05 AM
A Method for Evaluating Operating Parameters of Alumina Refinery Seed Classifiers using a Mathematical Model for Solids Settling: *Walter Mason Bounds*[1]; [1]Kaiser Aluminum, Gramercy Bus. Unit, PO Box 3370, Gramercy, LA 70052 USA

Information from a previously reported model for solids settling is incorporated into a mass balance in order to determine critical operating characteristics of seed classifiers. In particular, values for underflow and overflow rates as a function of solids concentration, along with underflow fine fraction and overflow coarse fraction, are extracted from the settling model in the form of curve fit equations. The settling model and classifier material balance equations are then solved simultaneously to provide information useful for interpreting and predicting classifier performance.

10:30 AM
Research on the Application and Mechanism of Crystal Growth Modifier on the Precipitation Process in Sodium Aluminate Liquors: *Xie Yanli*[1]; Zhao Qun[1]; Bi Shiwen[1]; [1]Northeastern University, Sch. of Matl. & Met., No.11, Ln. 3 Wenhua Rd., Heping Dist., PO Box 117, Shenyang, Liaoning 110004 China

Sandy alumina has become the dominant product in alumina industry since 1970s in order to meet the need of environmental protection and energy saving in modern aluminum smelter. Doping Crystal growth modifier is one of the most efficient methods to produce coarse and attrition-resistance hydroxide alumina but always decrease the precipitation ratio so that reduce the solution productivity. The effect and mechanism of different quantity of modifier on the precipitation ratio, the particle size and intensity of hydroxide alumina is studied in detail. As a result, it is found that when the quantity charged is increased to 180mg/l, the use of modifier can not only coarsen the particle and improve the strength of product, but enhance the precipitation process. As to other quantity added, the method to compensate for its shortcoming is also discussed minutely in this paper.

10:55 AM
Study on Alumina Hydrate Precipitation under Ultrasound: *Zhao Ji Hua*[1]; [1]Lanzhou University, Col. of Chem. & Cheml. Eng., Tianshui Rd., No. 298, Lanzhou, Gansu 730000 China

It is studied the effect of 20kHz and 33kHz ultrasound on alumina hydrate precipitation by Bayer Process. Compared with alumina hydrate precipitation without treatment of ultrasound, the precipitation times are reduced from 30h to 15h when the precipitation ratio is 45% under 20kHz and 33kHz ultrasound. The enhancement effects of two frequencies are not the same. Induction time is prolonged from 20min

to 40min under 20 kHz ultrasound, but it is reduced by 20min under 33 kHz ultrasound. From the comparison of grain size distribution and SEM photographs of alumina hydrate, it is found that secondary nucleation and agglomeration could be enhanced under 33kHz ultrasound. There are more little crystals in the product, though the average size is similar to that of without ultrasound. On the other hand, agglomeration is enhanced and the growth of alumina hydrate is increased under 20kHz ultrasound. As a result, the average diameter of alumina hydrate product is increased by 3.7 μm.

11:20 AM
Special Requirements to Aluminium Hydroxide of Non-Metallurgical Application: Vadim A. Lipin[1]; *Viktor I. Danilov*[2]; Andrey A. Kuznetzov[3]; [1]Russian National Aluminium-Magnesium Institute (VAMI), 86, Sredny pr., St. Petersburg 199106 Russia; [2]JSC "Volkhov aluminium", 20, Kirovsky pr., Volkhov, Leningrad Region 187400 Russia; [3]JSC PC "Glinozyom", 1, Spryamlyonnoye shosse, Pikalyovo, Leningrad Region 187600 Russia

For non-metallurgical application the special properties of Aluminium Hydroxide must be foreseen. These properties include the higher requirements to physical structure, chemical properties, particle size, etc. The special properties, as a rule, are reached by processing of usual Hydroxide precipitated in a cycle of alumina production as a result of decomposition or carbonization. The Sintering method used in Russia for alumina production has advantages before the Bayer process in purity of Aluminium Hydroxide. This method is most convenient for production of Aluminium Hydroxides with the low contents of iron and organic impurity, and also opportunities of reception of diverse physical structure of a products. The precipitation of Aluminium Hydroxides by carbonization allows to produce a gelatinous boehmite directly from aluminate liquors. Use of Sintering method for reception of non-metallurgical grades of Aluminium Hydroxides is most expedient with relation to both economy and environmental aspects of production.

Aluminum Reduction Technology: Söderberg and Prebake Cell Operations

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Martin Segatz, VAW Aluminum AG, D-53117, Bonn Germany; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday AM — Room: 6B
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Xiaoling Liu, Comalco

8:30 AM Invited
The Söderberg Cell Technology–Future Challenges and Possibilities: *Adolf Karstein Syrdal*[1]; [1]Elkem Aluminium Lista, PO Box 128, Farsund 4551 Norway

In the sixties the Söderberg cell technology came under intense pressure due to high emissions of tar fumes and poor working environment. Technology development during the past 40 years has therefore given preference to prebake rather than Söderberg cells. A strong paradigm has been that all Söderberg smelters would be phased out before the new millennium. It is therefore a paradox that in 2002 Söderberg plants still count for a substantial part of the global annual production capacity of primary aluminium. The present paper gives a short history of the Söderberg cell development and the present distribution of Söderberg lines around the world. Söderberg cell advantages and paradigms will also be discussed. In Western Europe new requirements for emissions will come into effect in 2007 and 2010. The capability of Söderberg plants to meet these new requirements and secure an acceptable working environment and thereby sustain future operation will be discussed.

8:55 AM
Experience of Vertical Stud Söderberg Operation Improvements at the Largest Aluminium Smelters of Russia: *V. Y. Buzunov*[1]; V. I. Savinov[1]; A. G. Barantsev[1]; V. K. Frizorger[1]; S. A. Shcherbinin[1]; V. H. Mann[2]; [1]PSC Bratsk Aluminum Plant, Bratsk 665716 Russia; [2]PSC Krasnoyarsk Aluminum Plant, Krasnoyarsk 660111 Russia

Largest world aluminum producers with VSS technology Krasnoyarsk and Bratsk aluminum smelters (KrAZ & BrAZ) are located in the center of East Siberia. One electrolysis line has been modernized with conversion to "dry" anode technology in the Krasnoyarsk plant in cooperation with Kaiser Aluminum company at 1990-1998, and "semidry" anode technology has been independently developed and implemented smelterwide. During the realization of these projects new methods of cathode and anode operation management have been maintained on the basis of the regular analysis and correction of process parameters using a technical database and advanced three-dimensional mathematical modeling. Also a new, for Russian aluminum industry, bath composition and anode technology has been introduced. As a result of the work carried out, the current efficiently was increased from 86 to 88%, the energy consumption was reduced from 16150 to 15500 kWh/t Al during 1996-1999. The experience gained at KrAZ has been successfully and quickly distributed at Bratsk in 2000. For Bratsk smelter current efficiency has increased from 85 to 87.5% and the energy consumption has reduced from 16150 to 15500 kWh/t Al during second half of 2000.

9:20 AM
Restart of 100 kA VSS Potlines after Long Shutdown: *Umakant Agrawal*[1]; G. D. Upadhyay[1]; C. W. Deoras[1]; [1]Bharat Aluminium Company, Ltd., Smelter, Balco Korba, Chhattisgrah 495684 India

Bharat Aluminium Company, Ltd. (BALCO) has an integrated aluminium complex at Korba, CG, India. The reduction plant has an installed capacity of 100,000 MT of metal per anum and employes 100 kA VSS technology of sixties supplied by VAMI, erstwhile USSR. It was incorporated as Government of India owned enterprise. With market liberalisation and opening of economy, Government of India disinvested its stake in BALCO by selling 51% of the equity and handing over the management control to M/S Sterlite Industries, India on March 2, 2001. Opposing the disinvestment the workers went on strike from March 3, 2001. The potlines were dipped in metal and power was switched off on March 8. The strike continued for 67 days and the work resumed on May 9. After restoration of normalcy, the power was switched on May 14. The pots were restarted in phased manner and within 30 days 332 pots (out of 400 pots) representing 83% of installed capacity were restarted. The total restart operation was over within 48 days. The pots have been restarted by gradually heating the potlines by pushing the power. Thus the cathode cavity was not cleaned, instead the solid metal and bath in the cells was melted gradually by heating only. This not only saved time but also the cost of restart. Initially few pots were dry restarted by melting the cryolite by forced arcing between anode and cathode. Later on pots were restarted by pouring liquid bath from these dry restarted pots or mother pots. The paper discusses the strategies adopted within the constraints of power station capacity, the expenses incurred and the technological parameter normalisation during restart operation. The paper also discusses the pot failures during restart.

9:45 AM
230KA Cell Pilot Plant—Guizhou Aluminium Complex of PRC: *Hongpeng Li*[1]; Chaohong Yang[2]; Shihuan Yao[2]; [1]Guizhou Aluminium Complex, Guiyang, Guizhou China; [2]Guiyang Aluminium Magnesium Design & Research Institute, Guiyang, Guizhou China

Guizhou Aluminium Complex is the largest primary aluminium producer in China. It has 560 prebake anode pots with capacity of 240,000t/y at present. To reach the target capacity of 400,000t/y, it is proposed to build a potline No. 4 which is to apply 230-250KA prebake pots. A pilot plant of six 230KA prebake pots (GY-230) were commissioned in Sept. 2000. In order to reach the stability of magnetic hydrodynamics of pots, optimal option is selected from the three simulated busbar arrangements. Advanced design and new materials are applied for the pot shell and lining to get better heat balance. These prototype pots can automatically measure and log the current distribution of each cathode group and anode. With nine months test production, the current efficiency is up to 94-95%, average voltage 4.25V and DC power consumption 13300-13500KWh/t.

10:10 AM Break

10:20 AM
Norðurál, the Icelandic Saga Continues: *Detlef Vogelsang*[1]; Joe Lombard[1]; Friedhelm Waldmann[1]; [1]VAW Aluminium-Technologie GmbH, PO Box 2468, D-53014 Bonn Germany

On June 17, 2001, the last pot of the Norðurál Phase II project was taken into operation. This expansion consisted of 54 CA180 cells as well as integrated into the system, the debut of 6 of the VAW CA210 cells. These cells are the result of VAW-ATG's modelling and engineering team developed over the last few years. The pots commissioned during the Norðurál Phase I project are operating well achieving world-class results. Since the Norðurál smelter was from the beginning designed as a modular smelter, the owners, CVC, have decided to expand in early 2000. This paper will deal with the Phase II project, highlighting the mechanism of concurrent engineering as it was practiced with

the introduction of the new technology. It will be concluded with a review and comparison of the theoretical and actual results predicted and achieved.

10:45 AM
Thermal Bake-Out of Aluminium Reduction Cells, A Technology for the Future: *Bernd Rolofs*[1]; David Eisma[1]; Gordon Dickinson[2]; Denis Hunzinger[2]; [1]CORUS Aluminium Voerde; [2]Zedtec Combustion Systems, Inc., 3801 Washington Rd., McMurray, PA 15330 USA

Thermal bake is a well-established method of preheating aluminium reduction cells as it provides better control of the preheating cycle, compared to resistor bake. It enables aluminium producers to significantly improve heat distribution by using convection and radiation to preheat the cells. This ensures that the cell lining is thoroughly baked, making cell start-up smoother and faster, and increasing the potential to extend cell life. Many of the recent advances in reduction cell technology point towards the use of thermal baking as the optimum preheating method. CORUS Voerde are obliged to use thermal bake for cells with coated cathodes but use it as well for normal cells. This paper presents extensive data gathered by CORUS Voerde since first using thermal bake in December 1999 and by Zedtec who have supplied the fully automatic gas preheating system.

11:10 AM
Investigating Thermoelectric Fields and Cathode Bottom Integrity during Cell Preheating, Start Up and Initial Operating Period: *G. Arkhipov*[1]; V. Pingin[2]; [1]Krasnoyarsk Academy of Non-Ferrous Metals and Gold, Krasnoyarsk 660111 Russia; [2]Krasnoyarsk Aluminum Plant, Krasnoyarsk 660111 Russia

This paper presents the results of calculations for temperature gradients and electric fields of the aluminum electrolysis cell, stress-strained state of cathode by oil preheating, start up and during initial operating period. Analysis of temperature gradients of the cathode bottom, their influence on joints and peripheral seams baking, stress-strained state and potential failure of the cathode bottom integrity was performed for different preheating regimes. The temperature gradients after bath pouring and flash heating were calculated based on the temperature gradients and bath conductivity by start up. The cathode preheating conditions effected the heating rate and non-uniformity of current distribution. Stress-strained state of the cathode structure and bottom integrity during start up and initial operating period were evaluated as time function taking into account cathode bottom thermal and sodium expansion, the size depending upon time, molten metal penetration rate, cathode bottom temperature and non-uniformity of current density.

11:35 AM
Coke Powder Bake Preheat Start-Up of 280kA Alumina Electrolysis Cells in China: *Zhao Qun*[1]; Xie Yanli[1]; Zhao Wuwei[2]; Qiu Zhuxian[1]; [1]Northeastern University, PO 117, Sch. of Matl. & Met., No.11, Ln. 3 Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China; [2]Zhenzhou Light Metal Research Institute, No.76, Jiyuan Rd., Shangjie Dist., Zhenzhou, Henan 450041 China

In this paper, the author introduced the first successful trials on 280kA cells using coke powder bake preheat start-up in China. From stuffing coke powder to pouring electrolyte, the general start-up procedure was illustrated step-by-step, the problems occurred in each stage were also discussed. The variety of current, potential and distributary current during start-up were recorded and the temperature field of the cell was described in detail, especially the temperature of lining and cathode steel bar. By this way, we have got shorter start-up period and less energy spend. The thermal impact and pre-mature failure caused by liquid aluminum during start-up were avoided and the cell service life will be extended.

Automotive Alloys 2002 - II

Sponsored by: Light Metals Division, Aluminum Association, *Program Organizer:* Subodh K. Das, Secat, Inc., Lexington, KY 40511 USA

Tuesday AM Room: 611
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chair: Subodh K. Das, Secat Inc., 1505 Bull Lea Blvd., Lexington, KY 40511 USA

8:30 AM
Formability of Aluminum Alloy Extrusions during Tube Hydroforming: *Rich W. Davies*[1]; Glenn J. Grant[1]; Kirit N. Shah[2]; Edmund W. Chu[2]; Robert P. Evert[2]; Mark T. Smith[1]; [1]Pacific Northwest National Laboratory, Matls. Proc. & Perfor., 902 Battelle Blvd., PO Box 999, Richland, WA 99352 USA; [2]Alcoa, Inc., Alcoa Techl. Ctr., 100 Technical Dr., Alcoa Center, PA 15069-0001 USA

The automotive industry is finding an ever-increasing number of applications for products manufactured using the tubular hydroforming process. Most of the current hydroforming applications use steel tubes. However, with the mounting regulatory pressure to reduce vehicle emissions, aluminum alloys appear attractive as an alternative material to reduce vehicle weight. The introduction of aluminum alloys to tubular hydroforming requires knowledge of their forming limits and modes of failure. The current work investigates the forming limits of various aluminum alloy extrusions in both the T4 and T6 tempers under laboratory conditions. These laboratory experiments consist of formability investigations under both proportional and non-proportional loading.

9:00 AM
Optimizing the Aging Heat Treatment of Cast Aluminum Alloys: *Joseph W. Newkirk*[1]; Qingcai Liu[1]; Alidad Mohammadi[1]; [1]University of Missouri-Rolla, Dept. of Metlgcl. Eng., 1870 Miner Cir., Rolla, MO 65409 USA

Cast aluminum alloys are used for many engine parts in today's cars. Many of the alloys used are age hardenable to get higher strengths. The optimum heat treatment used for producing the best combination of properties changes as the cast part gets larger, due to changes in quench rate. In addition, parts are sometimes aged after casting (T5) without a separate solution step. In order to sort out all of the parameters that effect the resulting properties Design of Experiments has been used to identify the most important parameters. In addition the quench sensitivity of A356 and B319 have been studied to determine the effect of quench rate and heat treatment on the optimization of properties. Results will be presented on the effect of these parameters on the strength of aluminum cast alloys.

9:25 AM
The Influence of Precipitation on the Work Hardening Behaviour of AA6111 and AA7030 Aluminum Alloys: *Leon M. Cheng*[1]; Warren J. Poole[1]; David Embury[2]; David J. Lloyd[3]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]McMaster University, Dept. of Matls. Sci. & Eng., 1280 Main St. W., Hamilton, ON L8S 1L7 Canada; [3]Alcan International, Ltd., Kingston R&D Ctr., PO Box 8400, 945 Princess St., Kingston, ON K7L 5L9 Canada

The influence of precipitation states on the yield stress and work hardening behaviour of AA6111 and AA7030 aluminum alloys after various aging treatments were examined. The presence of either a supersaturated solid solution or GP zones resulted in particularly high work hardening characteristics. During artificial aging, drastic changes in the work hardening rate were observed when precipitation occurred, which can be related to the different types of dislocations-obstacles interactions. These significant changes in work hardening behaviour as the materials were aged suggest that a work hardening analysis is a useful tool for studying the nature of dislocation accumulation processes and the interaction between dislocation hardening and precipitation phenomena. The change in work hardening behaviour has been interpreted by a consideration of addition laws for different sets of obstacles to dislocation motion. These changes were attributed to the depletion of solute and to the transition of the precipitates from shearable to non-shearable.

9:50 AM
A Process Model for Age Hardening of AA6111: *Shahrzad Esmaeili*[1]; Warren J. Poole[1]; David J. Lloyd[2]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada; [2]Alcan International, Kingston R&D, PO Box 8400, 945 Princess St., Kingston, ON K7L 5L9 Canada

A process model has been developed to predict the age hardening response of AA6111 as a function of thermal processing history. The aging treatments of the alloy usually lead to microstructures consisting of spherical GP zones, needle-shape β" and lath-shape Q phases. Both β" needles and Q laths are oriented along <100> direction of the matrix. The kinetics of formation of these phases is strongly dependent on the previous thermal path. Obtaining quantitative information on the kinetics of precipitation as a function of thermal history is a key component of the yield strength model. In this work we have used isothermal calorimetry to estimate the evolution of volume fraction of the precipitates during aging. The model takes into account the shape and orientation relationship of the precipitates with the slip plane. The present approach reduces the number of variables required for modeling and minimizes the need for experimental characterization of the microstructure for yield strength predictions.

10:15 AM Break

10:30 AM
Development of Strain Induced Surface Topography of 6XXX Series Aluminum Sheet under Balanced Biaxial Tension: *Stephen W. Banovic*[1]; T. Foecke[1]; [1]National Institute of Standards & Technology, Metall. Div., 100 Bureau Dr., MS 8553, Gaithersburg, MD 20899-8553 USA

With gas mileage goals set down in the Partnership of a New Generation of Vehicles as a driving force, the investigation into reducing overall vehicle weight by replacing conventional steel sheet metal with lightweight materials has been initiated. However, implementation of these lightweight alloys, specifically those that are Al-base, has been slow due to both a limited knowledge of material behavior and a lack of experience in the sheet forming process. By obtaining a better understanding of the material characteristics (e.g., surface roughening, friction, biaxial strain limits, and springback) during biaxial stretching, these materials can be more readily utilized for complex stamped components. Along these lines, the free surface roughening of aluminum alloys in the 6xxx series was studied as a function of biaxial straining using the modified Marciniak in-plane stretching test. Through the use of x-ray diffraction techniques, the change in crystallographic texture of the sheet was determined as a function of the strain level. Modification of the surface topography was monitored through electron microscopy and surface roughness measurements (via mechanical stylus profilometer and laser scanning confocal microscopy). From these results, relationships between the effective strain level, relative change in crystallographic orientation, and free surface roughening were developed.

10:55 AM
Effect of Cu and Si on the Microstructural Properties in the 3xx.x Alloy Series: *W. Kierkus*[1]; R. Mackay[1]; Q. Ren[1]; J. H. Sokolowski[1]; R. Hasenbusch[2]; [1]NSERC/Ford-Nemak/University of Windsor Industrial Research Chair in Light Metals Casting Technology, Mechl., Autom. & Matls. Eng., Rm. 203, Essex Hall, 401 Sunset Ave., Windsor, Ontario N9B 3P4 Canada; [2]Nemak Corporation of Canada, Casting Proc. Dvlp. Ctr. (CPDC), 4655 G. N. Booth Dr., Windsor, Ontario N9C 4G5 Canada

This work investigates the effect of Si and Cu levels on the as-cast and heat treated condition of 3xx.x microstructures. The compositions studied were 7 and 9 wt.% Si alloys each having 1, 2 & 4 wt.% Cu. The batch melts were cast in wedge sand moulds that facilitated cooling conditions ranging from 40 seconds up to 42 minutes. The effect that chemistry and cooling rate had on segregation, secondary phase size, shape, and porosity were studied for the test compositions. Finally thermal analysis was performed on all compositions to identify their relative characteristics of the mushy zone which yields the final cast structure. The results indicate that fast cooling rates facilitated the best refinement of all secondary phases present in the test alloys studied, but that the Al-9 wt. % Si-1 wt. % Cu test alloy had the lowest porosity of all test alloys.

11:20 AM
Microstructural Study of the Mechanism of Rapid Aging in Al-Mg-Cu Alloys: *Libor Kovarik*[1]; Perena I. Gouma[3]; Stephen A. Court[2]; Michael J. Mills[1]; [1]The Ohio State University, Matls. Sci. & Eng., Watts Hall #477, 2041 College Rd., Columbus, OH 43210 USA; [2]Alcan International, Ltd., Banbury Lab., Southam Rd., Banbury, Oxfordshire OX16 7SP UK; [3]State University of New York at Stony Brook, Matls. Sci. & Eng., Old Engineering 201, Stony Brook, NY 11794-2275 USA

The cause of the initial stage of rapid hardening in ternary Al-Mg-Cu alloys, which proceeds after several minutes of aging at temperatures around 180°C, is still disputed in the literature. The aim of our current research is to address the rapid hardening issue by high resolution transmission electron microscopy (HRTEM) coupled with image processing and image simulation. The slip behavior analysis reveals a weak tendency toward planar slip, suggesting the presence of short range order (SRO) or short range clustering (SRC). The image processing of HRTEM images and complementary image simulation also reveals the presence of SRO. Based on our present findings, we are inclined towards the theory that the initial stage of hardening is caused by formation of SRO or SRC of Mg atoms in the aluminum matrix.

11:40 AM
Effect of Stress State and Strain on the Particle Cracking Damage Evolution in 5086 (O) Al-Alloy: *A. Balasundaram*[1]; Arun M. Gokhale[1]; [1]Georgia Institute of Technology, Sch. of Matls. Sci. & Eng., Atlanta, GA 30332 USA

A series of experiments have been performed to quantitatively characterize the particle cracking damage evolution in 5086 (O) Al-alloy as a function of strain under compressive, torsional, and tensile loading conditions. Various microstructural parameters such as volume fraction, two-dimensional number density, geometric attributes, and orientation distribution of cracked and bulk Fe-rich intermetallic particles have been estimated to quantify the microstructural damage in 5086 (O) Al-alloy. In addition, three-dimensional microstructural parameters such as number density and average volume of cracked and bulk Fe-rich intermetallic particles have been estimated using the Large Area disector (LAD) technique, which is an unbiased technique and makes no assumptions on the shape and size of the particles. The analysis of these quantitative microstructural and mechanical properties data, and their comparison with the earlier data on 6061(T6) Al-alloy, leads to the following important observations and conclusions. In the 5086 (O) alloy, Fe-rich particles rotate during deformation of the alloy under compression and torsion and align themselves along the direction of applied/induced tensile stress. However, the extent of particle rotations is significantly lower than that in the 6061 (T6) alloy for deformation under uniaxial compression. For torsional deformation, the particle rotation tendencies are comparable in the two alloys. These differences are attributed to dynamic strain aging. It is observed that the anisotropy of crack orientations in 5086 (O) and 6061 (T6) alloys strongly depend on the loading condition and stress state: under uniaxial tension, the cracks are mostly perpendicular to direction of applied load, whereas they are mostly parallel to the loading direction when a compressive load is applied. Progression of damage evolution under compressive loading involves cracking of more larger particles in 5086 (O) alloy as compared to those in 6061 (T6) alloy. However, the progression of damage nucleation under tensile loading due to particle cracking is predominantly due to progressive cracking of large unfavorably oriented particles, whereas that in the 6061 (T6) alloy is due to progressive cracking of smaller particles. At stress values beyond the yield stress of 6061 (T6) alloy, the number fraction of cracked particles in 5086 (O) alloy is significantly lower than that in the 6061 (T6) alloy. This is due to a variety of factors such as finer microstructure, differences in flaw density, fracture strength of the particles, lower particle rotation tendencies and dynamic strain aging (DSA) in 5086 (O) alloy as compared to 6061 (T6) alloy.

Carbon Technology: Anode Production and Performance Improvements

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Don T. Walton, Aluminum Company of America, Wenatchee Works, Malaga, WA 98828-9728 USA; Les Edwards, CII Carbon, Chalmette, LA 70044 USA; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday AM — Room: 602-603
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Michael Schneider, Corus Aluminium, SchleusenstraBe 11, Voerde D-46562 Germany

8:30 AM
Reduction of PAH Emission in Alcan Quebec's H.S. Söderberg Smelters by Evaluation and Conversion to Low PAH Pitch: Amir A. Mirchi[1]; Andre L. Proulx[1]; Gaby Savard[1]; Emile Simard[1]; *Herman Vermette*[1]; Michel Hamel[2]; [1]Alcan International, Ltd., Reduction Tech., 1955 Mellon Blvd., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada; [2]Alcan Primary Metal Group, Shawinigan Smelter, 1100 St-Sacrement Blvd., Shawinigan, Quebec G9N 6W4 Canada

Low PAH pitch binder implementation in Alcan's H.S. Söderberg plants marks an important step in the Company's continuing efforts to reduce the Polycyclic Aromatic Hydrocarbons (PAH's) emission of the Söderberg technology. Improvements to H.S. Söderberg anode formulation and implementation of the low PAH pitch binder has provided a significant reduction in PAH's emission in Alcan's H.S. plants over the last twenty years. A demonstration test was carried out with a low PAH pitch in the Söderberg plant before Alcan's H.S. plants conversion to this new generation of pitch. The impact of this new generation of pitch on the paste plant operation, anode performance and PAH's emission during cell operation was evaluated. The plant performance of the low PAH pitch in terms of emission was compared with the laboratory characterization of the pitch.

8:55 AM
Featuring the New AP-FCBA Paste Plant Technology: *Christian Dreyer*[2]; Pinoncely Andre[1]; Nigel Backhouse[2] [1]FCB Aluminium, 32

Rue Fleury Neuvesel, Givors 69702 France; [2]Aluminium Pechiney LRF, BP 114, Saint Jean de Maurienne 733000 France

Aluminium Pechiney and FCB Aluminium jointly developed a new process for dry mix preparation, dedicated to the production of an optimized grain size distribution at the lowest investment and operating cost. An industrial scale 35 tph pilot plant, which incorporates the use of a Rhodax® vibrating crusher, is currently featured in the latest paste plant technology. This paper discloses the proposed process route, explains the operating philosophy of the plant, and summarizes the main expectations of such new technology which will be available as early as 2002.

9:20 AM

Installation of an Anode Paste Cooling System at Slovalco: *Berthold Hohl*[1]; Lubomir Gocnik[2]; [1]Maschinenfabrik Gustav Eirich, Prod. Mgr. Carbon, D- 74736, Hardheim Germany; [2]SLOVALCO a.s, Head of Carbon Plant, 96 563 Ziar nad, Hronom Slovakia

As a part of an expansion program of the SLOVALCO smelter, EIRICH has successfully commissioned a paste cooler/mixer together with a new pitch fume and vapor treatment system. In the second half of the year 2000, after a start-up period of only 4 weeks, the extended paste mixing line came into operation. Being originally equipped with only one continuous kneader, the plant now shows a significantly higher performance, especially as far as paste quality is concerned. The main purpose of this paper is to present the technical solution, the operational results before and after the start-up of the new machine as well as the environmental situation.

9:45 AM

Recent Developments in Process Control for Green Anodes Production: *André Pinoncely*[1]; [1]FCB Aluminium, 32 rue Fleury Neuvesel, Givors 69702 France

The construction of new smelters using the latest technology gives the opportunity to implement original control systems and to further improve the process for production of green anodes. The present paper highlights most of the PLC driven control loops that were developed and describes how much operation of the plant was smoothed on a continuous basis. Moreover, anode quality reached new records in steadiness while green rejects almost disappeared. The new generation of "gently controlled" paste plants went recently on stream, and key figures extracted from over 18 months industrial operation illustrate the benefits resulting from such improvements, even under very different project environments.

10:10 AM Break

10:20 AM

Converting Ball Mill System from Steel to Rubber Liners-Case Study: *Ned Malcolm*[1]; Donna Marvel[1]; James Roberts[2]; [1]Alcoa, Warrick Ops., Newburgh, IN 47630 USA; [2]Alcoa, Rockdale Ops.

The Warrick and Rockdale Smelters have undertaken a conversion of the ball mill liners from the traditional steel to rubber. This procedure was undertaken primarily for Safety reasons, but the economic factors are not insignificant. The end result was one successful and one unsuccessful conversion. This paper will explore the reasons driving the conversion, the grinding condition before and after the conversion, and a discussion on why one conversion was successful and the other not.

10:45 AM Cancelled

Exhaustion, Pneumatic Conveyor and Storage of Carbonaceous Waste Materials: *Paulo Douglas Santos Vasconcelos*

11:10 AM

The Aluminum Company of Egypt (Egyptalum)–A New Anode Producer: *Mohiy Eldin Zakiy*[1]; Vincent Giroud[2]; Giovanni Magarotto[3]; Harald Onder[4]; [1]The Aluminum Company of Egypt, Plants Sector, Managing Dir., 48/50 Abd El Khalik St., Nag Hammady, Cairo Egypt; [2]FCB Aluminium, Givors France; [3]Tomorrow Technology, Due Carrare Italy; [4]Venco, Techl. Consultant, Houston, TX USA

Following the original concept of converting the 1975 Nag Hammadi, Egypt, Soderberg Aluminium Smelter to prebake technology, a 160,000tpy prebake anode production facility was installed and commissioned in June 2000 at Egyptalum's site. The originally planned conversion program was modified after the installation of one prebake potline, requiring the supply of only approximately 27,000tpy of baked anodes. With a diversification project, Egyptalum has to produce 85,000tpy of baked anodes of size 915×535×475mm for delivery to a US smelter. The paper describes the technical conversion of the modern, highly automated, state-of-the-art, 1 line, 1 size (1650×725×600mm) anode production plant to a multi-dimensional production facility, ready to supply anodes of various dimensions to external customers.

11:35 AM

Neural Network Model of Anode Production Department as a Basis of Green Anodes Improvement: *A. I. Berezin*[1]; O. O. Rodnov[1]; P. V. Polyakov[1]; V. A. Klykov[1]; V. L. Krylov[2]; [1]Scientific Technological Center, Light Metals, Post Box 14144, 95 Krasnoyarsky Rabochy St., Krasnoyarsk 660025 Russia; [2]OKSA, Sayanogorsk Russia

Carbon anodes are an essential part of the cost of primary aluminum. The minimization of excess consumption of anodes by means of improving their quality is a key industrial goal. At the OKSA aluminum plant in Russia, the quality and quantity of raw materials changes frequently, leading to variations in the production process. The paper describes a neural network model, which is used for an in-depth analysis of the technology of anodes and definition of optimum parameters of green anode production. The estimation of adequacy and accuracy of model has shown, that the error between actual and predicted quality indicators of green anodes did not exceed 3%, and the error of definition of control actions is not more than 8%. The neural network model of mixing and pressing operations in the anode production department has adequate accuracy for the solution of practical problems.

Cast Shop Technology: Melting

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: David H. DeYoung, Alcoa Technical Center, Alcoa Center, PA 15069 USA; John F. Grandfield, CSIRO Australia, Preston, Victoria 3072 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday AM — Room: 6A
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Paul Van Zyl, Bayside Aluminium, PO Box 284, Richards Bay 3900 S. Africa; Robert Jenkins, Thorpe Technologies Inc., PO Box 1759, 124 Cove Ave., Gulf Shores, AL 36542 USA

8:30 AM Keynote

Aluminum Melting Technology-Current Trends and Future Opportunities: *Donald L. Stewart*[1]; [1]Alcoa Technical Center, Ingot & Solidification, 100 Technical Dr., Alcoa Center, PA 15069 USA

An estimated 20 million metric tons of aluminum scrap, RSI and primary ingot is melted annually worldwide. Much of the metal is melted in batch reverberatory furnaces using air and natural gas, while high-surface-area scraps such as swarf and UBCs are melted in sidewell or induction melters. Over the past decade, a number of advanced melting technologies have been developed and reported in the literature. The objectives of most of the new processes have been reduced metal loss, improved melt rate, increased energy efficiency or reduced emissions. In this paper, a review of major technical developments over the recent past will be presented. Additionally, the author's view of needs and opportunities for further development in aluminum scrap melting will be discussed.

9:00 AM

Efficiency Evaluation of Melting Aluminum Furnaces: *Frank L. Beichner*[1]; [1]Bloom Engineering Company, Inc., 5460 Horning Rd., Pittsburgh, PA 15236 USA

Increasing fuel prices have placed an increased emphasis on the cost to melt aluminum in the cast house. This brings to bear questions like, what is the most cost effective method to melt aluminum? Should we consider cold air, regenerative, oxy/fuel or even air/oxy/fuel? Can I improve the efficiency of my existing operation, since my current energy costs have increased dramatically in the last few months? This paper provides evaluates the application of cold air, oxy/fuel, enriched oxygen combustion and regenerative considering 1) overall costs incurred in the combustion process 2) determination of the effect on furnace efficiency and 3) calculation of emission factors. In most cases there is not a straight answer to the question of which technology should be utilized. The questions that you will need to answer to make your best engineering recommendation will be presented so your company's needs are attained.

9:25 AM

Heating Patterns Produced by Different Industrial Flame Types: *Laszlo Istvan Kiss*[1]; Geza Walter[1]; Vincent Goutiere[1]; Andre Charette[1]; Wesley Stevens[2]; [1]UQAC Université du Québec à Chicoutimi, Dépt. des Sciences Appliquées, 555 blvd. de l'Université Chicoutimi, Chicoutimi, Québec G7H 2B1 Canada; [2]Alcan International, Ltd., Arvida R&D Ctr., 1955 blvd. Mellon, Jonquiere, Quebec G7K 5J7 Canada

The terminology used to classify industrial burners often reflects certain easy-to-perceive characteristics of the flame produced by them. In the present study short and long, low and high velocity flames together with a so-called "envelope" type flame were compared experimentally. The luminosity of the flames varied strongly among the different settings. One of the two burners used in the study was a traditional, generic device while the other was a low NOx, injection type burner. The aim of the study was to determine the distribution of the total heat load along the length of the different flames as well as to analyze the mechanism of the heat transfer between the flame and charge. The results include the axial distribution of the total heat flux as well as that of its radiative and convective components on a parallel surface under the flame. The analysis offers help for the better selection of burners for various industrial heating jobs.

9:50 AM

Furnace Operation Optimization Via Enhanced Bath Circulation: *Alan M. Peel*[1]; Rifat Alchalabi[2]; Fanli Meng[2]; [1]EMP Technologies, Ltd., Beedes House, Easton Ave., Stretton, Burton-on-Trent, Staffordshire VE13 OVB England; [2]Chinook Sciences, LLC, NJ USA

Aluminum re-melting furnaces fuel and production efficiency highly depends upon furnace design, operational practices, scrap pretreatment selection, furnace control system, and choice of furnace equipment. However, it is customary that the production requirement, and available scrap type changes with time. Along with these changes comes the necessity to change the current melting practice through expansions and the addition of high technology equipment that can be easily retrofitted to the furnace. Accordingly, it is widely customary that furnace equipments require enhancements and changes and then adaptation to these new changes. However, the new equipment selection decision, typically, is done separate and independent from the furnace design, control system, and even from the choice of the other equipments. In this paper a technical evaluation to the importance of equipment selection-compatibility, and operational compatibility to the furnace fuel and production efficiency are evaluated. More specifically this paper will evaluate technically the impact of using appropriate and compatible electromagnetic pumping circulation units (EMP System–Electromagnetic Pumping System) will be presented and supported with installation results detailing production gains, improvements in furnace operational flexibility and reduction in both fuel consumption and dross formation. The technical evaluation will be further supported by state-of-the-art detailed furnace computer physiochemical-modeling. The furnace computer modeling analysis will evaluate the impact of the furnace bath molten metal flow on the efficiency of heat transfer and the overall furnace fuel and production efficiency. Overall, this technical review will emphasize on how the electromagnetic pumping technology has enabled producers to increase their production, improve operational flexibility and lower the cost of producing aluminum.

10:15 AM Break

10:30 AM

Development of a Pilot "Top Charge" Melt Furnace to Examine the Fundamental Melting Phenomena in Aluminum: *Wesley D. Stevens*[1]; Jean-Yves Fortin[1]; [1]Alcan International, Ltd., Arvida R&D Ctr., PO Box 1250, Jonquiere, Quebec G7S 4K8 Canada

A pilot scale «top charge» melt furnace has been developed at Alcan's Arvida Research and Development Centre to study the fundamental melting phenomena taking place in the full scale industrial melt furnace process. The scaled equipment includes a high precision electronic balance permitting in-situ measurement of the rate of formation of oxide for different alloys and scrap forms being melted. High temperature glass observation ports were installed to allow both video and still photographic imagery of the melt process. The gas train permits a single gas burner to be positioned in one of seven locations, the burner and observation ports being interchangeable. Of particular interest to Alcan was the visual documentation of the actual melting phenomena. Almost impossible to carry out in full scale furnaces, we have been able to photograph the melt process as it actually occurs with a variety of materials including cold and hot mill edge trim, extrusion billets, foil rolls, and solid ingot pieces. This information is particularly important for modelling the melting process to determine the time relationship of the change in position of the solid-gas phase boundary.

10:55 AM

Experimental Investigation on the Wear Resistance of Refractories: Effect of the Nature of the Exposed Surface: *N. Ntakaburimvo*[1]; *C. Allaire*[1]; [1]Ecole Polytechnique of Montreal, CRIQ Campus, 8475, Christophe-Colomb, Montreal, Quebec H2M 2N9 Canada

In aluminum confinement units such as holding and melting furnaces, refractories are deteriorated by the action of corrosion and mechanical abuse. Among the later, abrasion and erosion intervene during charging, cleaning, drossing, stirring and others. This paper presents a new experimental set-up for testing the high temperature wear resistance of refractories in presence of molten aluminum alloys. Results on aluminosilicate refractories, including amorphous silica-based castables are presented. The effect of some material characteristics, such as the mechanical properties, the surface texture and others is also discussed.

11:20 AM Cancelled

Optimising the Top Charge Melt Furnace Process-Effect of Alloy and Gauge on Melt Loss: *Wesley D. Stevens*

11:45 AM

New Pump for the Aluminum Industry: *Chris T. Vild*[1]; David V. Neff[1]; Richard S. Henderson[1]; [1]Metaullics Systems Co. LP, 31935 Aurora Rd., Solon, OH 44139 USA

A new family of molten metal pumps has been developed by the leading manufacturer of pumps for the molten aluminum industry. These new patented pumps, designated Tensor™ Series pumps, are designed to last up to a full year between rebuilds. The novel design uses ceramic and steel to replace the traditional graphite posts normally used in molten metal pumps. The result is a very cost-effective method to stir or transfer molten aluminum. Tensor technology developed by Metaullics is a new way to apply ceramics to molten metal pumps. The ceramic post is reinforced by a high temperature alloy steel rod. The rod is loaded in tension which in turn loads the ceramic in compression. This takes advantage of the ceramic's maximum strength and enables the use of a lower cost ceramic. Impact toughness is improved and the material used is not prone to thermal shock. The goal of the patented Tensor technology is to make molten metal circulation and transfer economical and reliable. The Tensor™ Series pumps now being introduced achieve this goal, and ongoing developments at Metaullics will assure even more progress in the future.

Cast Shop Technology: Cast Structures

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: David H. DeYoung, Alcoa Technical Center, Alcoa Center, PA 15069 USA; John F. Grandfield, CSIRO Australia, Preston, Victoria 3072 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday AM — Room: 608
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Philipe Jarry, Pechiney, Centre de Recherches de Voreppe, 725 rue Aristide Berges-BP 27, Voreppe 38341 France; John Taylor, University of Queensland (CAST), Dept. of Minls., Minls. & Matls. Eng., Brisbane, QLD Australia

8:30 AM

Evolvement of Primary Particles in DC-Cast AA 3003 Alloy during Heating and Homogenization: *Yanjun Li*[1]; *Lars Arnberg*[1]; [1]Norwegian University of Science and Technology, Dept. of Matls. Tech. & Electrochem., NTNU, 7491, Trondheim Norway

The evolvement of primary particles in DC cast 3003 alloy during heating and homogenization at 500 and 600°C has been investigated. The size distribution, area fraction and particle density of the particles as well as the electrical conductivity of the alloy are measured. Different mechanisms, breaking, growing and coarsening, compete in the evolvement of the primary particles during the heating and homogenizing process. The size and area fraction of the particles increase with homogenization time. The precipitation of Mn onto the particles from the supersaturated matrix plays a very important role in the size evolvement of the particles. The transformation from Al6(Mn,Fe) to Al(MnFe)Si and its reverse transformation during heating and homogenization are also investigated.

8:55 AM

Investigating the Alpha Transformation–A Solid-State Phase Change of Dispersed Intermetallic Particles from an Al_6(Fe,Mn) Phase to an Alpha-Al(Fe,Mn)Si Phase: *D. T.L. Alexander*[1]; R. G. Hamerton[2]; H. Cama[2]; A. L. Greer[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Cambridge CB2 3QZ UK; [2]Alcan International, Ltd., Banbury Lab., Southam Rd., Banbury OX16 7SP UK

Homogenisation of direct chill (D.C.)-cast AA3104 can beverage stock aluminium alloy causes intermetallic constituent particles to transform from a tetragonal $Al_6(Fe,Mn)$ phase to a cubic alpha-Al(Fe,Mn)Si phase. Electron microscope techniques have been used to investigate the tetragonal $Al_6(Fe,Mn)$ to cubic alpha-Al(Fe,Mn)Si transformation in samples of a model Al-0.5wt.%Fe-1.0wt.%Mn-0.2wt.%Si alloy during isothermal heat-treatments at 500°C. Elemental-mapping using energy-filtered transmission electron microscopy, together with convergent-beam electron diffraction, has revealed that transformation is eutectoid; diffusion of silicon from the aluminium matrix into $Al_6(Fe,Mn)$ particles causes transformation to an alpha-Al(Fe,Mn)Si/aluminium eutectoid. Nucleation is thought to be a key factor controlling the overall transformation rate. After transformation, intragranular eutectoid aluminium coalesces and ripens to become intergranular 'Al-spots'. Resultant effects on particle morphology have been observed by examining deep-etched samples in a FEGSEM. In addition, manganese diffuses into transformed particles, segregating to grain boundaries.

9:20 AM
Spheroidization of Silicon and its Influence on the Mechanical Properties of the Eutectic Al-12%Si Alloy: *Ruyao Wang*[1]; Weihua Lu[1]; Hsienyang Yeh[1]; Henry H.E. Yeh[1]; [1]Donghua University, Dept. of Mechl. Eng., 1882 W. Yan'an Rd., Shanghai 200051 China

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

9:45 AM
Microstructure of Aluminum-Lithium Alloys Obtained with Directional Solidification: Alicia Esther Ares[3]; Carlos T. Rios[2]; Rubens Caram[2]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Sci., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]Universidade Estadual de Campinas, Dept. Engenharia de Materiais, CP 6122, Campinas, Sao Paulo 13083-970 Brasil; [3]CONICET, Post-Doctoral Fellow, CP 6122, Campinas, Sao Paulo 13083-970 Brasil

The Aluminum-Lithium Alloys are of important use in the auto and aerospace industry. It is generally accepted that good solidification structures are required for performance. In the present report the solidification structure of these alloys are studied experimentally. Alloys containing Al, Li and Cu were directionally solidified under different conditions of temperature gradients and velocities; the alloys also contained grain refiners. The alloys which were added with refiners were completely equiaxed. The dendritic structure were analyzed with scanning electron microscopy and image analyzer. The secondary dendritic space were measured and the results were compared and correlated with the models available in the literature.

10:10 AM Break

10:25 AM Cancelled
Self-Modified Structure in Direct Electrolytic Al-12%Si Alloy: *Ruyao Wang*

10:50 AM
Columnar to Equiaxed Transition in Al-2%Cu, Al-4%Cu and Al-10%Si-2.5%Cu Alloys: Alicia Esther Ares[3]; Rubens Caram[2]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Sci., Posadas, Misiones 3300 Argentina; [2]Universidade Estadual de Campinas, Dept. Engenharia de Materiais, CP 6122, Campinas, Sao Paulo 13083-970 Brasil; [3]CONICET, Posadas, Misiones 3300 Argentina

The columnar-to-equiaxed transition was investigated in Al-2%Cu, Al-4%Cu and Al-10%Si-2.5%Cu alloys solidified directionally from a chill face. The transitions occur when the temperature gradient in the melt ahead of the columnar dendrites decreases to -3.89°C/cm in Al-2%Cu, to -0.55°C/cm in Al-4%Cu and to 0.49°C/cm in Al-10%Si-2.5%Cu; at the time of the transition the liquidus interface velocities were about 0.9 cm/seg, 0.33 cm/seg and 0.048 cm/seg, respectively. The observations indicate that the transition is the result of a competition between coarse columnar dendrites and finer equiaxed dendrites. The results are presented and discussed.

11:15 AM
Dendrite Spacing in Al-Cu and Al-Si-Cu Alloys as Function of the Growth Parameters: Alicia Esther Ares[3]; Carlos T. Rios[2]; Rubens Caram[2]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Sci., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]Universidade Estadual de Campinas, Dept. of Matls. Eng., UNICAMP CP 6122, Campinas, Sao Paulo 13083-970 Brasil; [3]CONICET, Post-Doctoral Fellow, 1552 Azara St., Posadas, Misiones 3300 Argentina

Aluminum alloys containing Copper and Silicon were directionally solidified in a range of velocities and thermal gradients producing dendritic structures. The primary and secondary spacings were measured and correlated with the main solidification parameters. The results were also compared with the main theoretical models from the literature. The results are presented and discussed in the frame of the models available. As a main conclusion of the present investigation the results agree with the general observations reported in the literature that an increase in the growth velocity produce a decrease in the primary spacing, and an increase in the local solidification time produce an increase in the secondary spacing.

11:40 AM
Modelling the Metallurgical Reactions during Homogenisation of an AA3103 Alloy: *Arild Håkonsen*[1]; Dag Mortensen[2]; Steinar Benum[1]; Tanja Pettersen[1]; Trond Furu[1]; [1]Hydro Aluminium A.S., R&D Matls. Tech., PO Box 219, Sunndalsora N-6601 Norway; [2]Institute for Energy Technology, Kjeller N-2027 Norway

The as cast microstructure of a DC cast AA3103 alloy consists of equiaxed grains with a cellular structure. The periphery of the cells contains high volume fractions of intermetallic phases and it is large variations in the solid solution level across the cells. During a typical homogenisation heat treatment the material is heated at 50 to100°C/hour up to a temperature of 500-600°C and held there for some hours. The material is then cooled to room temperature (extrusion ingot) or fed into the hot-rolling mill (sheet ingot). A model for the metallurgical reactions occurring in this system is constructed based on a cylindrical cell geometry. The as cast microstructure is adopted from a solidification model (Alstruc) that predicts the micro segregation, the volume fraction and the composition of the primary phases. A thermodynamic description of the two phases Al6(Mn,Fe) and Al15(Mn,Fe)3Si is proposed, assuming matrix to be a dilute solution and the phases to be regular solutions. Fe and Mn are allowed to substi tute each other completely. Precipitation, growth and coarsening of the phases are modelled individually in each position across the cell, each particle is designated to a size class and infinite diffusion is assumed inside particles. Diffusion across the cell is accounted for. Model results are compared with measured number density and size distribution of precipitates and electrical conductivity.

Charles J. McMahon Interfacial Segregation and Embrittlement Symposium: Theory of Segregation and Fracture

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, Jt. Mechanical Behavior of Materials, Jt. Computational Materials Science & Engineering, Texture & Anisotropy Committee
Program Organizers: Vaclav Vitek, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA; Clyde Briant, Brown University, Division of Engineering, Providence, RI 02912 USA; Harvey D. Solomon, General Electric Company, Research & Development Center, Schenectady, NY 12309 USA

Tuesday AM — Room: 307-308
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: David N. Seidman, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL USA; Pavel Lejcek, Academy of Sciences of the Czech Republic, Inst. of Phys., Prague, Czech Republic

8:30 AM Invited
Computational Materials Science Approach to Interfacial Segregation and Embrittlement: *Arthur J. Freeman*[1]; [1]Northwestern University, Dept. of Phys. & Astron., 2145 N. Sheridan Rd., Evanston, IL 60208 USA

It is now becoming increasingly clear that computational materials science is in a golden age of accomplishment and rapidly growing

impact. This was brought about by the dramatic advances in condensed matter theory, especially electronic structure theory, and their successful application to real materials problems made possible by utilizing the continued explosive growth of computer power. These simulations of ever-increasing complexity now serve to fill the increasingly urgent demands of material scientists and engineers and demonstrate the power of this advanced methodology for treating the structural, electronic, magnetic, and mechanical properties of materials, including. Here we present some recent results with illustrative examples: (1) Intergranular embrittlement, often the controlling factor limiting the ductility of high strength metallic alloys, was investigated to determine the role of impurities and alloying additions segregated to the grain boundary (GB) and their effects on the GB cohesion of alloy steels. First principles FLAPW total energy atomic force calculations and the Rice-Wang thermodynamic model were employed to investigate the mechanism by which segregated substitutional additions cause intergranular embrittlement. This is used as the basis for an electronic level phenomenological theory to predict unambiguously the effect of substitutional alloying additions on GB cohesion of metallic alloys for a large number of metals, including 3d, 4d and 5d transition metals. Examples are given for the Fe Σ(111) and Ni Σ5(210) grain boundaries. (2) The segregation of impurities on intrinsic interfaces is an important factor determining mechanical behavior of materials. The interactions of transition metal (TM) impurities (Ti-Co) with intrinsic interfaces-anti-phase boundaries (APB)-in NiAl were investigated using the ab-initio real-space tight-binding LMTO recursion method; impurity-APB interaction energies and segregation energies were calculated. The correlation of the segregation energy with d-electron band filling of impurity elements was demonstrated. The dependencies of the APB energy on impurity concentration were calculated and their effect on the primary slip systems were determined. (3) The effect of alloying with Re and Ti impurities on the fracture toughness of NiAl/Mo eutectic composites was investigated by means of the electronic structure, cleavage and shear energy calculations using the ab-initio FLMTO tin method. It was demonstrated that (i) the coherency strain of mismatched Mo and NiAl phases plays the crucial role in the cohesive properties of the interface, (ii) the clean NiAl/Mo interface itself is not a weak place where a crack may begin to propagate and (iii) segregation of impurities is the main factor controlling the interface debonding. Work performed in collaboration with W. T. Geng, G. B. Olson, Y. N. Gornostyrev, O. Y. Kontsevoi and N. I. Medvedeva, and was supported by the ONR and AFOSR.

9:05 AM Invited
Interfacial Adhesion and Structure of Grain Boundaries in bcc Transition Metals with Segregated Interstitial Impurities: *Christian Elsässer*[1]; Rebecca Janisch[1]; Thorsten Ochs[1]; [1]Max-Planck-Institut für Metallforschung, Seestr. 92, C-70174, Stuttgart, Germany

The influence of interstitial impurities B, C, N, O and H on the structure and stability of Σ5 (310) [001] symmetrical tilt grain boundaries (Σ5 STGB) in bcc Mo and Nb is investigated by ab-initio mixed-basis pseudopotential calculations of the local density functional theory (LDFT). Even in low concentration the impurities affect the translation state and the bonding strength of the Σ5 STGB considerably. For Mo bicrystals loaded with C in high concentration, bulk precipitates and interfacial layers, as observed experimentally, are studied via LDFT calculations for MoC and $MC_{0.5}$ bulk phases and for Mo/MoC {310} heterophase interfaces. As validated earlier for the pure Σ5 STGB, the ab-initio LDFT results are accurate data sets for developments of semi-empirical tight-binding models for the Mo-C system, which provide interatomic interactions for large-scale atomistic simulations of interfacial segregation and embrittlement.

9:40 AM
Impurity-Induced Decohesion: *John L. Bassani*[1]; [1]University of Pennsylvania, Mechl. Eng. & Appl. Mech., 297 Towne Bldg., 220 S. 33rd. St., Philadelphia, PA 19104-6315 USA

Decohesion is analyzed under varying concentration of interfacial impurities. Under stress, the impurity can diffuse into the interface and lower its cohesive energy, a process McMahon terms dynamic embrittlement. A simple phenomenological model is introduced to describe the effects of impurity concentration on cohesive strength. Even under uniform stressing and depending upon the rate of decohesion relative to the rate of diffusion, the impurity concentration can be non-uniform along the interface. Ahead of a crack, the concentration generally will be non-uniform. A cohesive-zone model has been developed that accounts for both impurity diffusion and creep deformation ahead of a crack. The relationship between the applied load (e.g., the stress-intensity factor under small-scale yielding) and crack velocity is predicted as a function of material parameters.

10:15 AM
Brittle Fracture and the Breaking of Atomic Bonds: *Peter Gumbsch*[1]; [1]Max-Planck-Institut für Metallforschung, Seestrasse 92, 70174 Stuttgart, Germany

Recent advances in the microscopic modeling of fracture processes have led to new insights into brittle fracture processes. The atomistic aspects of brittle fracture are summarized and discussed in this paper. Examples are taken from ab-initio simulations and empirical atomistic modeling of brittle fracture which show that the production of metastable fracture surfaces or directional cleavage anisotropy are readily anticipated consequences of the discrete nature of the bond breaking at the crack tip. In this context, the difficulties of modeling bond breaking processes with empirical atomic interaction models become important. Finally, an attempt will be made to relate thermally activated fracture processes and the energies measured in dynamic fracture experiments to the microscopic characteristics of the material.

10:35 AM
A Universal Mechanism of Brittle Compressive Failure: *Erland M. Schulson*[1]; Carl E. Renshaw[1]; [1]Thayer School of Engineering, Dartmouth College, Hanover, NH 03755 USA

Coulombic shear faulting marks terminal compressive failure of ice, rock and other brittle polycrystalline materials when loaded under moderate degrees of confinement. The faults often form as intersecting sets on planes which, for a fiction coefficient appropriate to such materials, are inclined by ~30 to the direction of maximum (most compressive) principal stress and which contain the intermediate principal stress. Coulombic faults are composed of narrow bands of damage which, in ice at least, are characterized in part by out-of-plane extensions to grain boundary cracks, termed wing cracks, and by sets of closely spaced secondary cracks, termed comb cracks, that stem from one side of a sliding primary crack. The faults are triggered, we propose, by the bending-induced failure of slender microcolumns (created by the comb crack), through frictional drag across their free ends (Schulson 2001, Eng. Fract. Mech., in press). In this paper we describe the comb-crack mechanism and show that it accounts quantitatively for the brittle compressive strength of a variety of crystalline materials. The mechanism, we suggest, is a universal one.

10:55 AM
Segregation of Phosphorus Atoms to Grain Boundaries in Ferritic Steels under Neutron Irradiation: *Alexander V. Barashev*[1]; Yury N. Osetsky[1]; David J. Bacon[1]; [1]The University of Liverpool, Dept. of Eng., Matls. Sci. & Eng., Liverpool L69 3GH UK

The segregation kinetics of phosphorus (P) atoms to grain boundaries (GBs) in reactor pressure vessel steels is studied assuming the three-dimensional migration of P atoms via the vacancy and interstitial mechanisms. An analytical expression for the dependence of the GB coverage of P atoms on the irradiation dose, dislocation density, diffusion parameters and P content is derived. The theory is valid below some critical temperature, where the thermal effects are negligible and P atoms are fully adhesive to GBs. The results are compared with other calculations and experimental data. The influence of recombination and clustering of point defects, which occur in displacement cascades, on segregation is discussed. Further clarification of the mechanisms and physical parameters associated with P diffusion requires molecular dynamics simulation and this is in progress.

11:15 AM
Interaction of Grain Boundaries with Point Defects in fcc Metals: *Akira Suzuki*[1]; Yuri Mishin[1]; [1]George Mason University, Sch. of Computl. Scis., 4400 University Dr., MSN 5C3, Fairfax, VA 22030 USA

Interaction of vacancies and interstitials with several tilt grain boundaries in Cu and Al is studied using molecular statics, harmonic lattice dynamics, and the embedded-atom method. Equilibrium defect concentrations in the grain boundaries are calculated from their formation energies and entropies, the latter being computed in the harmonic approximation. Point defect formation energies at grain boundaries are in average lower than in the regular lattice, but variations from one position to another are very significant. Vacancies can be either localized at certain sites in the boundary core or be delocalized over several sites. Some atomic positions in the boundary core are unable to support a stable vacancy. Interstitial atoms can either occupy open positions between atoms or form split dumbbell configurations. The relation of these effect to grain boundary diffusion is discussed.

11:35 AM
Atomistic Simulations of Segregation of Alloying Elements to a Grain Boundary in bcc Fe: *Diana Farkas*[1]; Renata N. Nogueira[2]; Margarita Ruda[3]; [1]Virginia Tech, Dept. of Matls. Sci. & Eng., Blacksburg, VA 24060 USA; [2]USP, Escola Politecnica, Sao Paulo Brazil; [3]CNEA, Centro Atomico Bariloche, Bariloche, Argentina

Atomistic simulations of the segregation of various substitutional and interstitial alloying elements to a grain boundary in Fe were performed. The simulations employ embedded atom interatomic potentials to study the energetics of the segregation phenomenon and the fracture process directly. We studied Ni, Cr, C and H impurities in a (210)[001] symmetrical tilt boundary. The particular sites in the grain boundary that are preferred by each of these impurities was studied. These results and the energetics of segregations are correlated with the effects observed in the fracture mechanisms. A discussion is presented on the various factors that contribute to the effect of the impurities on grain boundary fracture behavior.

Commercial Mini-Sessions

Sponsored by: Light Metals Division,
Program Organizers: David V. Neff, Metaullics Systems Company, Solon, OH 44139 USA; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday AM Room: 401
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chair: David V. Neff, Metaullics Systems Company, 31935 Aurora Rd., Solon, OH 44139 USA

Computational Modeling of Materials, Minerals & Metals Processing: Tuesday AM Plenary Session 9:00 AM – 9:45 AM

Sponsored by: Materials Processing & Manufacturing Division,
Program Organizer: Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Tuesday AM Room: 619-620
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chair: James W. Evans, University of California, Dept. of Matls. Sci. & Minl. Eng., 585 Evans Hall, Berkeley, CA 94720-1760 USA

Keynote
Phase Field Methods for Modeling Microstructure: *James A. Warren*[1]; [1]National Institute of Standards and Technology, Metall. Div. & Ctr. for Theoretl. & Computl. Matl. Sci., Gaithersburg, MD 20899 USA

The phase field method has been successfully employed as both a tool to model heterogeneous materials and as numerical method for calculating the motion of interfaces and phase boundaries without explicitly tracking those interfaces. The method has been used to model a diverse suite of problems describing the microstructural evolution in materials. These models are derived from thermodynamic arguments and symmetry principles, and usually guarantee positive local entropy production for systems out of equilibrium. Descriptions of how phase field methods can be applied to the problems of solidification, grain growth, and electroplating will be presented.

Computational Modeling of Materials, Minerals & Metals Processing: Track A - Structure

Sponsored by: Materials Processing & Manufacturing Division,
Program Organizer: Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Tuesday AM Room: 619
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chair: Jim Warren, NIST, CTCMS & Metall. Div., Gaithersburg, MD 20899-8554 USA

9:45 AM
Microporosity Evolution and Interdendritic Fluid Flows during Solidification: Adrian S. Sabau[1]; Srinath Viswanathan[1]; [1]Oak Ridge National Laboratory, Matls. & Cer. Div., Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

The occurrence of microporosity during metal casting is due to the combined effects of solidification shrinkage and gas precipitation. The governing equations for fluid flow and hydrogen evolution indicate that porosity formation and fluid flow are strongly coupled. However, in most studies on microporosity, it is considered that the porosity formation does not influence the fluid flow in the mushy zone. In this study, a computational methodology is presented for the numerical simulation of microporosity evolution and interdendritic fluid flow. The solution algorithm presented includes a fully coupled, implicit treatment of microporosity and local pressure in the mushy zone. It is shown that neglecting the effect of porosity formation on the pressure in the mushy zone yields higher pressure drops and an overprediction of final porosity. By its growth, microporosity compensates partially for the solidification shrinkage, reducing the feeding demand. Therefore, in order to accurately describe casting defects, comprehensive models of fluid flow, heat transfer, solidification, must include the effect of microporosity as well.

10:10 AM
Cellular Automata Computer Model of Polycrystalline Plastic Deformation: *Alexander V. Spuskanyuk*[1]; Yakiv Beygelzimer[1]; Victor M. Varyukhin[1]; [1]Donetsk Physical & Technical Institute of the NAS of Ukraine, High Press. Phys. & Adv. Tech. Dept., 72 R. Luxembourg St., Donetsk 83114 Ukraine

Effects stipulated by interdependence of microlevel and macrolevel of plastic deformation processes were analyzed. Using computing mechanics instead of constitutive relationships the adequate computer models were used, which was opened by cellular automata approach. By means of numerical experiments, the cellular automata allowed to study the macrobehavior of the ensemble of cells at the macrolevel depending on the local microscopic laws that define evolution of each cell and its interaction with the closest environment. A cellular model of the plastic deformation of polycrystalline aggregate was proposed and comprehensively described. Representative volume of the deformed solid body deformed was described as a population of interconnected units which, in turn, consisted of lower scale level units. Sliding along the various allowed sliding systems deforms simple units, which do not have an internal structure. For consideration of stress distribution within the limits of components, the approach of self-consistent field was used. Rotation of units and moment stresses connected with it were taken into account. Results of computer experiments are analyzed, software is described.

10:35 AM Break

10:50 AM
Simulations of Microstructural Evolution: *Martin E. Glicksman*[1]; Kegang Wang[1]; P. Crawford[1]; [1]Rensselaer Polytechnic Institute (CII-9111), Matls. Sci. & Eng., 110 8th St., Troy, NY 12180-3590 USA

Predicting microstructure evolution in alloys remains a keystone of materials science. The mean-field theory of phase coarsening, in the (impractical) limit of zero volume fraction, was first formulated by Lifshitz and Slyozov, and by Wagner (LSW). Numerous attempts have been made to extend LSW theory toward microstructures with nonzero volume fractions. The successes achieved with analytical theories, however, have been limited, due primarily to the difficulties of characterizing interactions among particles and the matrix, and accounting for stochastic variations in the microstructural locale surrounding each particle. Such theories predict unrealistic particle size distributions (PSDs) when compared with experimental observations. The importance of large-scale simulation of microstructures was realized with concurrently increasing capability of computer hardware and software. Since the 1980s we formulated and solved multiparticle diffusion equations to simulate the dynamics of phase coarsening. These simulations provide insight into the nature of diffusion interactions and multiparticle stochastics. The rate constants, PSDs, and higher-order correlations can all be extracted by simulation. “Snap shot” simulation techniques, developed recently permit study of microstructure size at various volume fractions. Gradually, a bridge has been built connecting fundamental theory and experiment through computer simulations. Some recent examples of progress in simulating microstructure evolution will be discussed.

11:15 AM
Ab Initio Calculations of Theoretical Tensile Strength in Metals and Intermetallics: *Mojmir Sob*[1]; Ligen Wang[1]; Martin Friak[1]; Vaclav Vitek[2]; [1]Institute of Physics of Materials, Zizkova 22, Brno 616 62 Czech Republic; [2]University of Pennsylvania, Dept. of MSE, 3231 Walnut St., Philadelphia, PA 19104-6272 USA

Fully self-consistent ab initio electronic structure calculations of the theoretical tensile strength in metals and intermetallics loaded uniaxially along several crystallographic directions are performed using the full-potential LAPW method. It turns out that the theoretical tensile strength and elastic anisotropy at higher strains are closely connected with the presence or absence of higher-symmetry structures along corresponding deformation paths. Total energy calculations show that all higher-energy cubic structures studied are locally unstable with respect to tetragonal and/or trigonal deformation modes. In intermetallics, there may or may not be symmetry-dictated energy extrema corresponding to cubic lattices depending on the atomic ordering. However, other energy extrema along the deformation paths besides those required by symmetry occur. Configurations corresponding to energy minima on the deformation paths may represent metastable structures that can play an important role in interfaces and other extended defects. As a specific example, tensile strength of single-crystalline tungsten loaded uniaxially along the [001] and [111] directions is analyzed. Although tungsten is elastically nearly isotropic for small deformations theoretical tensile strength exhibits a marked anisotropy. This anisotropy is explained in terms of structural energy diferences between bcc, fcc and simple cubic structures which occur on the calculated deformation paths. Theoretical results compare favorably with available experimental value obtained for tungsten whiskers grown along the [110] direction. Further examples include computer simulations of a tensile test for single-crystalline NiAl, where the theoretical tensile strength for the "hard" orientation [001] differs very significantly from that for the [111] orientation. Again, this anisotropy may be understood in terms of higher-symmetry structures present or absent along the deformation paths.

11:40 AM
Modeling of Interdendritic Strain and Interdendritic Cracking Phenomena during Dendritic Solidification Processes: *Mostafa El-Bealy*[1]; [1]Material Processing International, 100 Trade Centre Dr., #103, Champaign, IL 61820 USA

A one-dimensional mathematical model to calculate the interdendritic cracking tendencies for low alloyed carbon steel casting processes is described. The model combines an interdendritic strain model with concept of the effect of alloying element on the solidification behaviour, segregation of carbon, and therefore, critical elementary interdendritic area EIA. A susceptibility of cracking of different steels is modelled by using El-Bealy approach. Model predictions were performed to explain the effects of various alloying elements on the solidification and cracking phenomena. Some typical cases in conventional casting processes related to increase cracking susceptibility are discussed. It is shown that there is a satisfactory degree of correlation between prediction a practical casting knowledge. Possible solutions to these problems based on the adjustment of chemical composition are proposed.

Computational Modeling of Materials, Minerals & Metals Processing: Track B - Melting & Solidification - II

Sponsored by: Materials Processing & Manufacturing Division, *Program Organizer:* Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Tuesday AM — Room: 620
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Dan Cook, Virginia Commonwealth University, Mechl. Eng. Dept., 601 W. Main St., Rm. 312, Box 843015, Richmond, VA 23284-3015 USA; Mark Jolly, University of Birmingham, Birmingham, Great Britian B15 2TT UK

9:45 AM
Wax Injection in the Investment Casting Industry: Jean-Christophe Gebelin[1]; Alexander Cendrowicz[1]; *Mark R. Jolly*[1]; [1]The University of Birmingham, IRC in Matls., Edgbaston, Birmingham, W. Midlands B15 2TT UK

Injection of wax patterns is the first stage of the multi-stage process of investment casting. The quality of the final casting and its dimensional accuracy is highly dependent on this stage of the process. Pattern waxes used in the industry behave in a complex visco-elastic-plastic manner. The modelling of such material behaviour is therefore not simple. In this paper a number of configurations of die will be shown in which the injection of the wax has been carried out for a range of processing conditions. Some of the dies are transparent and the movements of wax front observed can be compared with those predicted by simulation. Surface defects in final components will also be shown and compared with the location predicted by simulation software. Some discussion will be presented on the limitations of the software used and the time-scales achieved for practical use as a design tool within the foundry environment.

10:10 AM
Micro/Macro Modeling of Ingot Cooling Processes for Ni-Cu-S Alloys: *Apostle Mouchmov*[1]; Mark Cross[2]; Koulis Pericleous[1]; [1]University of Greenwich, Sch. of Compg. & Mathl. Sci., 30 Park Row, Greenwich, London SE10 9LS UK; [2]The University of Greenwich, Ctr. for Numl. Modlg. & Proc. Analy., Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Copper–nickel–sulphide alloys are typically cooled and solidified in 4, 8 and 16 tonne ingots. These ingots exhibit a variation of grain size distribution and macro segregation of the prime alloy components throughout. The former is assumed to be primarily due to the dominant relation between grain growth rate and the cooling profile, whilst the latter is heavily influenced by buoyancy driven residual convection. The objective of this research program is to examine the extent to which it is possible to develop a 'broad brush' computational model of this ingot cooling process, that can predict some 'integral' measure of the grain size (e.g. average diameter) and the macro-segregation as a function of operating conditions. A computational modeling software framework, PHYSICA+ is used to simulate the complex process of ingot casting, which involves coupling between different physical phenomena. The entire model involves: (i) a 'broad brush' grain growth model that could be used in the prediction of micro/macro-structure of alloy ingot cooling processes, (ii) a heat transfer and solidification model which takes into account a second phase transformation of Ni3S2 to heazlewoodite, and (iii) NS fluid flow simulation which provides a good basis for further micro/macro segregation modeling. At this stage some results of the 3D convection driven thermal cooling and solidification profiles will be shown, together with the 'integral' model for the grain size prediction.

10:35 AM Break

10:50 AM
Comparison of Numerical Models of Solidification Behavior in Direct Chill Casting with Experiments: Christopher J. Vreeman[2]; David Schloz[3]; *Matthew John M. Krane*[1]; [1]Purdue University, Sch. of Matls. Eng., W. Lafayette, IN 47907 USA; [2]Boeing North American, Rocketdyne Div., 6633 Canoga Ave., Canoga Park, CA 91309 USA; [3]Wagstaff, Inc., 3910 N. Flora Rd., Spokane, WA 99216 USA

Numerical results from a continuum mixture model of the Direct Chill casting process is compared to experimental results from industrial scale aluminum billets. The model, which includes the transport of free-floating solid particles, is used to simulate the effect of a grain refiner on macrosegregation and fluid flow. It is applied to an Al-6wt%Cu alloy and the effect of casting speed, grain refiner, and assumed mushy zone permeability on predicted macrosegregation, sump profile, and temperature fields are presented. Three 45 cm diameter billets were cast under production conditions with and without grain refiner and at two casting speeds. Temperature and composition measurements and sump profiles are compared to the numerical results. The comparison shows qualitative agreement and limitations of application of the model to industrial processes are discussed.

11:15 AM
Two-Phase Predictive Finite-Element Flow Model for Semi-Solid Slurries: *Frédéric Pineau*[1]; [1]National Research Council Canada, Indl. Matls. Inst., Proc. Modlg. & Instrm., 75 de Mortagne Blvd., Boucherville, Québec J4B 6Y4 Canada

Semisolid metal alloys have a special microstructure of globular grains suspended in a liquid metal matrix. This particular physical state of the matter can be exploited to produce near-net-shape parts with improved mechanical properties. However, the behavior of the slurry is strongly influenced by the local solid fraction and state of agglomeration. Different flow instabilities associated with the combined flow and solidification process result, which make difficult the application of semisolid processing in the casting industry. Moreover, the rheology of semisolid materials is not well understood. Most of the theory has been derived from experimental data, which are somewhat difficult to measure. A model that accounts for the multiphase nature of the slurry is required to get more insight into such complex flows. This paper thus describes a mixture model for semisolid slurries. It assumes that the mixture of liquid-solid components behaves as a single fluid as far as overall mass and momentum balances are concerned. The coupling force between the phases is derived on the assumption that the slurry is a fluid saturated isotropic media. The proposed methodology is implemented in a finite element code. The filling of an industrial-scale

capillary flow viscometer is investigated numerically. Segregation patterns are obtained and discussed.

11:40 AM
CFD Simulation of Continuous Charging and Melting of Small Metallic Particles in a Melting Reactor: *Stefan Pirker*[1]; Oszkar Biro[3]; Philipp Gittler[1]; Peter Mittag[2]; Bernard Aigner[2]; [1]Johannes Kepler University, Altenbergerstr. 69, Linz A-4040 Austria; [2]VOEST ALPINE Industrieanlagenbau GmbH, Postfach 3, Linz A-4031 Austria; [3]Technical University Graz, Kopernikusgasse 24, 8010 Graz Austria

This paper considers CFD modelling of processing and melting of small metallic particles by means of electrical heating. The particles fall continuously onto a liquid metal bath which is heated by an electric arc. After melting of the particles liquid metal is tapped. The charging behavior of the particles in the supply unit as well as in the reactor freeboard is studied by means of Euler-Euler granular simulations. The flow situation in the metal bath due to gas injection and magnetic fields is calculated by combining Navier-Stokes and Maxwell solvers. The macroscopic melting process occurring during the continuous charging of the particles is studied by kinetic laws for melting reactions. The temperature field is evaluated by balancing heat sources due to Joule's heating and heat losses due to latent heat of melting as well as convection and radiation. As a result of these simulations the fully three-dimensional flow fields of particles and gas in the atmosphere is obtained. Furthermore the flow field as well as the magnetic field in the metal bath can be studied. As a main result the three-dimensional concentration field of the still unsolved particles in the liquid metal can be evaluated.

12:05 PM
Numerical Simulation of Wax Pattern Dimensions in Investment Casting: Adrian S. Sabau[1]; *Srinath Viswanathan*[1]; [1]Oak Ridge National Laboratory, Matls. & Cer. Div., Bldg. 4508, MS 6083, Oak Ridge, TN 37831 USA

Dimensional Changes between a pattern die and its corresponding investment cast part occur as a result of complex phenomena such as thermal expansion/contraction and hot deformation (elastic, plastic, and creep) during the processing of the pattern material (wax), mold material (shell), and solidifying alloy. Determining the pattern tooling dimensions is crucial to the dimensional control of the investment casting process. To date, there are no computational methodologies available for predicting dimensional changes during investment casting. This paper deals with the evaluation of wax pattern dimensions, which is one important factor in determining the pattern tooling dimensions in investment casting. Cerita 29-51, an industrial wax is considered in this study. The wax pattern dimensions are affected by its thermophysical and thermomechanical properties, restraint of geometrical features by the metal die, and process parameters such as dwell time, platen temperature, injection pressure and injection temperature. Numerical simulation results for the wax pattern dimensions are compared with experimental measurements. Critical variables that determine dimensional changes associated with the wax system are identified.

Computational Phase Transformations: Modeling Solidification Microstructures

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Jt. Computational Materials Science & Engineering, Thermodynamics & Phase Equilibria Committee
Program Organizers: Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; Mark Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; James Aaron Warren, NIST, CTCMS and Metallurgy Division, Gaithersburg, MD 20899-8554 USA

Tuesday AM — Room: 201
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Jim Warren, NIST, CTCMS & Metall. Div., 100 Bureau Dr., MS 8554, Gaithersburg, MD 20899-8554 USA

8:30 AM
On the Stability of Eutectic and Peritectic Coupled Growth and the Dynamics of Solid-Solid-Liquid Trijunctions: Blas Echebarria[1]; *Alain Karma*[1]; [1]Northestern University, Phys., 360 Huntington Ave., Boston, MA 02115 USA

This talk will summarize the results of recent numerical and analytical studies that shed new light on, and provide a unified picture of, the stability of coupled growth in eutectic and peritectic alloys. It is theoretically well-established since the pioneering analyses of Hillert and Jackson-Hunt that steady-state eutectic growth in non-faceted/non-faceted materials can occur for a continuous range of lamellar spacing, with the temperature vs spacing curve having a maximum at a fixed growth rate. Moreover, so far, the part of this curve with a positive slope has been assumed to be morphologically unstable, with the consequence that the stability limit of coupled growth at small spacing coincides with the point of maximum temperature of the steady-state temperature-spacing curve. As showed by Langer in 1980, the points of marginal stability and maximum temperature do indeed coincide if one assumes, as originally proposed by Cahn, that lamella grow locally perpendicular to the envelope of the composite interface. A close examination of the results of phase-field simulations of directional solidification of eutectic alloys have led us to the conclusion that this assumption does not strictly hold for typical growth conditions of experimental relevance. Our results show that the trijunctions can slide along the front (i.e. perpendicularly to the solid-solid interfaces) with a velocity that depends on the local gradient of the lamellar spacing. When this sliding motion is included, the point of marginal stability and maximum temperature no longer coincide, which has important consequences for the stability of coupled growth in eutectic alloys, both with and without a ternary impurity present, and in peritectic alloys. This main finding is confirmed by recent experiments by Akamatsu and Faivre in a transparent organic eutectic alloy, which we discuss.

9:00 AM
Simulation of Dendritic Growth with Fluid Flow: Jun-Ho Jeong[1]; *Jonathan A. Dantzig*[1]; Nigel D. Goldenfeld[2]; [1]University of Illinois, Mechl. & Indl. Eng., MC-244, 1206 W. Green St., Urbana, IL 61801 USA; [2]University of Illinois, Dept. of Phys., MC-704, 1110 W. Green St., Urbana, IL 61801 USA

Understanding pattern selection during dendritic solidification is an important problem for materials scientists and engineers. This paper describes recent computations using phase-field models to directly simulate 3D dendritic growth with fluid flow. We employ an adaptive gridding procedure for solving the phase field equations, where high resolution is available near the interface, and more appropriate grid dimensions are used to resolve the diffusion and velocity fields. Fully three-dimensional simulations of growth of pure materials are presented, and it is demonstrated that the three dimensional aspects of the flow are essential. Parallel implementation of the code is also described. Direct comparisons of computational and experimental results are presented, demonstrating the fidelity of the simulations.

9:30 AM
Modeling Elasticity in Crystal Growth: *Ken R. Elder*[1]; Mark Katakowski[1]; Mikko Haataja[2]; Martin Grant[2]; [1]Oakland University, Phys., Rochester, MI 48309-4487 USA; [2]McGill University, Phys., Montreal, Quebec H3A 2T8 Canada

A new model of crystal growth is presented that describes the phenomena on atomic length and diffusive time scales. The former incorporates elastic and plastic deformation in a natural manner, and the latter enables access to times scales much larger than conventional atomic methods. The model is shown to be consistent with the predictions of Read and Shockley for grain boundary energy, and Matthews and Blakeslee for misfit dislocations in epitaxial growth.

10:00 AM Break

10:15 AM
Phase Field Modeling of Electrochemistry: *Jonathan E. Guyer*[1]; William J. Boettinger[1]; James A. Warren[1]; Geoffrey B. McFadden[2]; [1]NIST, Metall. Div., 100 Bureau Dr., Gaithersburg, MD 20899 USA; [2]NIST, Infor. Tech. Lab., 100 Bureau Dr., Gaithersburg, MD 20899 USA

We present the first application of phase field modeling to electrochemistry. This work was motivated by the mathematical analogy between the governing equations of solidification dynamics and electroplating dynamics. For example, the solid-liquid interface is analogous to the electrode-electrolyte interface. The various overpotentials of electrochemistry have analogies with the supercoolings of alloy solidification: diffusional (constitutional), curvature and interface attachment. Dendrites can form during solidification and during electroplating. The crucial presence of charged species in electrochemistry, however, leads to rich interactions between concentration, electrostatic potential, and phase stability. The present model properly pre-

dicts the charge separation associated with the equilibrium double layer at the electrochemical interface and its extent in the electrolyte as a function of electrolyte concentration. A single set of equations describes electron conduction in the solid and ionic conduction in the electrolyte. The goal of the approach is to treat the complex geometry, including void formation, that occurs during plating in vias and trenches for on-chip metallization.

10:45 AM
Modeling Solidification of Crystals Floating in a Moving Liquid: *Adam C. Powell*[1]; David M. Dussault[1]; [1]MIT, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139-4307 USA

The phase-field and fluid-structure interaction approaches are coupled in a model of elastic crystals solidifying from a flowing liquid. Motion is governed by a mixed elastic-viscous stress formulation, in which elastic modulus is set to zero in the fluid, and viscosity to zero in the solid, with intermediate values of both in the diffuse interface. Crystal surface energy and growth kinetics are both anisotropic, and elastic properties may be anisotropic as well, with crystal orientation rotated according to the local vorticity. An Eulerian approach is taken using finite differences in two dimensions, and extension to three dimensions is conceptually straightforward. Results are presented for several anisotropic surface energy and kinetics models.

11:05 AM
Modeling Reduction of Liquid Metal from Liquid Electrolyte in Three Dimensions: *David M. Dussault*[1]; Adam C. Powell[1]; [1]MIT, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 4-117, Cambridge, MA 02139-4307 USA

A three-dimensional phase-field electrochemical model is presented which describes the transport-limited reduction of liquid metal from a liquid electrolyte. This model assumes zero charge density everywhere, and thus does not capture space charge distribution across the double-layer. For this reason, the methodology cannot be used to model electrolysis under charge transfer limiting conditions. However, the double-layer related phenomenon of electrocapillarity can be represented by a gradient penalty term which includes the electric potential gradient, such that interfacial energy is a function of the electric field normal to the interface. When reaction rate is limited by metal ion transfer to the cathode, finger-like "liquid dendrites" of metal grow into the electrolyte and then break up into metal droplets which float or sink depending on the relative densities of the two liquids. Capturing dendrite breakup in the model requires three dimensions, as the liquid dendrite shape is stable in two.

11:25 AM
Phase-Field Modeling of Eutectic Growth: *Daniel J. Lewis*[1]; [1]NIST, Metall. Div., 100 Bureau Dr., MS 8555, Rm. B164, Gaithersburg, MD 20899 USA

Many eutectic classification schemes have been completed where the focus on interface velocity and thermodynamic parameters is used to classify the resultant microstructures. Although the kinetic component of phase growth is dominant at higher interface velocities, understanding the morphology selection process requires that the thermodynamics be well studied. Thermodynamic parameters such as interface energy, molar volume, entropy of solution, and entropy of freezing have been used to classify binary eutectics, but very little has been done to classify ternary eutectic alloys. This is due in large part to the wealth of microstructures that can be formed in ternary eutectics due to the presence of three solid phases. In order to understand the eutectic morphology selection process a phase-field model of eutectic solidification has been developed and simulation results will be presented.

11:45 AM
Phase-Field Modelling of Spontaneous Grain in Rapidly Solidified Melts: *Andrew Martin Mullis*[1]; [1]University of Leeds, Dept. of Matls., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK

The origin of spontaneous grain refinement in deeply undercooled metallic melts is of enduring interest within the solidification literature. We present the results of phase field simulations of dendritic growth into pure undercooled melts, at growth velocities up to 35 m/s. We find that, at low growth velocities, dendrite morphologies are self-similar with increasing growth velocity. However, above 15 m/s the initiation of side-branching moves closer to the dendrite tip with increasing growth velocity. This is related to the level of kinetic undercooling at the tip. Once side-branch initiation begins to occur within 1-2 radii of the tip, profound morphological changes occur, leading to severe thinning of the dendrite trunk and ultimately repeated multiple tip-splitting. This process can be invoked to explain many of the observed features of spontaneous grain refinement in deeply undercooled metallic melts. Comparison between simulation results and experimental microstructure and velocity data is made.

Creep Deformation: Fundamentals and Applications: Fundamental Behavior - III

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, Jt. Mechanical Behavior of Materials, Powder Materials Committee
Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA; Sai V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA

Tuesday AM — Room: 214
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: F. A. Mohamed, University of California, Dept. of Cheml. & Biocheml. Eng. & Matls. Sci., 744J EGR, Irvine, CA 92697-2575 USA

8:30 AM Invited
Creep Deformation of Ti-6Al-2Sn-4Zr-2Mo: *Robert W. Hayes*[1]; Babu Viswanathan[2]; Mike Mills[2]; [1]Metals Technology, Inc., 19801 Nordhoff St., Northridge, CA 91324 USA; [2]Ohio State University, 2041 N. College Rd., Columbus, OH 43210 USA

It has recently been shown that trace levels of Fe,Ni and Co when present in solid solution in the alpha phase of Ti increase the rate of Ti self-diffusion and the rate of Al substitutional diffusion as well. We have recently shown that the magnitude of primary creep strain as well as the steady-state strain rates of the commercial alloy Ti-6Al-2Sn-4Zr-2Mo (wt.%) are increased in the presence of trace levels of Ni in the same manner as the diffusivity in alpha Ti. This suggests a strong link between the diffusion and the creep behavior in this alloy. In addition, the apparent activation energy for creep is decreased with increasing levels of Ni which is also consistent with the effects of Ni on the activation energy for self and Al substitutional diffusion in alpha Ti. When analysed in terms of well known phenomenological creep laws, the present results fit best with a power law representation with dislocation climb as rate limiting. A more detailed analysis based upon deformation substructure has led to a mo re precise description of the specific creep process. We show that a strong correlation between the effects of trace levels of Ni on Ti self-diffusion in alpha Ti and on creep in Ti-6-2-4-2 does exist. However, the creep behavior in the alloy is somewhat more complex.

8:55 AM
The Use of Creep Test in Probing the Initiation of Plastic Strain in Nearly-Dislocation Free Crystals: *Y. Q. Sun*[1]; [1]University of Illinois, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA

How plastic strain by slip is initiated in crystals with low initial dislocation density is very important to several key mechanical properties of crystalline materials. In this paper we show that the method of creep test can be used to detect sensitively the onset of plastic strain in these materials. The creep test overcomes a chief weakness of the usual constant-strain-rate tests in which the yield point does not mark the initiation of plastic strain. We have applied the anisothermal creep method to probe the initiation of slip in several materials with low initial dislocation density, including intermetallics, silicon and HCP metals. The results show that slip is initiated abruptly in these materials with a well-defined, rate-independent critical temperature. These new experimental results are explained in terms of dislocation nucleation mechanisms and are contrasted with predictions by dislocation multiplication models.

9:15 AM
Grain Boundary Sliding during Creep: Role of Boundary Misorientation and Strain Compatibility: *Askar D. Sheikh-Ali*[1]; Jerzy A. Szpunar[2]; Hamid Garmestani[1]; [1]NHMFL-FSU, Lab. for Micromech. of Matls., 1800 E. Paul Dirac Dr., Tallahassee, FL 32310 USA; [2]McGill University, Dept. of Metlgcl. Eng., 3610 University St., Montreal, Quebec H3A 2B2 Canada

Zinc bicrystals of different types with symmetrical tilt boundaries have been tested in creep conditions. Observations of grain boundary sliding along boundaries with different misorientations have been made. It has been established that Sigma 9 coincidence boundary slides faster than boundaries of general type. The sliding along coincidence bound-

ary is activated at higher stresses and only in the presence of intragranular slip. Also, sliding along coincidence boundary is accompanied by regular boundary migration. Negative grain boundary sliding has been observed near one of the boundary ends in different bicrystals as well as boundary curving. Dislocation reactions in compatible and incompatible bicrystals have been analyzed and different mechanisms of the influence of intragranular slip on sliding have been revealed.

9:35 AM
The Elevated Temperature Stress-Strain Behavior of Ti and TiNi Alloy Produced by High Pressure Torsion: *Alla V. Sergueeva*[1]; Ruslan Z. Valiev[2]; Amiya K. Mukherjee[1]; [1]University of California, Cheml. Eng. & Matl. Sci., 1220 Bainer Hall, Davis, CA 95616 USA; [2]Ufa State Technical University, Inst. of Adv. Matls., 12 Marks str., Ufa 450000 Russia

The tensile stress-strain behavior of commercially pure Ti and TiNi alloy subjected to severe plastic deformation by high pressure torsion has been investigated over a range of elevated temperatures and strain rates. That treatment leads to formation an ultrafine-grained structure in Ti and partial amorphization in TiNi with strong refinement of the residual crystalline phase. High stresses and intensive work hardening were observed in both materials at "conventional" strain rates and at temperatures below 0.3Tm. The increase in temperature (or decrease in strain rate) leads to transition to steady-state flow of the materials but the flow stress remains much higher than that for Ti and TiNi alloy with microcrystalline structure at the same testing conditions. Results were analyzed in terms of a rate equations describing deformation behavior with respect of microstructural evolution. This investigation was supported by a grant from the US National Science Foundation (NSF-DMR-9903321).

9:55 AM
Novel High Temperature Creep Control Mechanism for the Region of High Applied Stresses: *Oleksandr Illich Dekhtyar*[1]; [1]Kurdyumov Institute of Metal Physics of National Academy of Science of Ukraine, Div. of Strength & Plasticity of Nonhomogeneous Alloys, 36 Vernadsky Blvd., Kiev, Kiev Region 03142 Ukraine

Transition from the region of low stresses, where the stress exponent is equaled to 4-5, to the region of high stresses, where the stress exponent is equaled to 8-9, has been determined by the average space between jogs created on dislocations in subboundaries by their mutual intersections. At high stresses the most of subboundaries consists from two or more sets of the edge and mixed dislocations with different Burgers vectors. In this stress region the jog space is lower than free path of vacancy drift along dislocation core before its vapor into the bulk. The directed vacancy flow from one system of dislocations, which are the vacancy sources, to other system of dislocations, which are the vacancy run-off, has appeared due to the chemical potential gradient between these dislocation systems. As result the mechanism of collective climb of dislocations within subboundaries is realized.

10:15 AM Break

10:25 AM Invited
Study on the Superplastic Deformation Mechanism using Magnesium-Based Materials: *Kenji Higashi*[1]; *Toshiji Mukai*[2]; Hiroyuki Watanabe[2]; [1]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1,Gakuen-cho, Sakai, Osaka 599-8531 Japan; [2]Osaka Municipal Technical Research Institute, 1-6-50 Morinomiya Joto-ku, Osaka 536-8553 Japan

Investigations to develop correct models for understanding the superplastic flow mechanism are very much in order. In the present paper, two problems in the fine-grained superplasticity, whose details are indispensable to understand the nature of superplastic flow, were discussed using magnesium-based materials. First, the effect of temperature and grain size on superplastic flow was investigated for the inclusive understanding of the dominant diffusion process. We precisely determined the effective diffusion coefficient for superplastic flow involving lattice diffusion coefficient and grain boundary diffusion coefficient. A phenomenological constitutive equation for superplastic flow using the effective diffusion coefficient was developed. Second, the effect of intergranular and intragranular particles was examined. Two critical strain rates for the elimination of the effect of these particles were developed respectively by considering the suitable deformation models. In addition, constitutive equation for superplastic flow under the influence of intragranular particle was proposed.

10:50 AM
Internal Stress Creep-Plasticity Due to Dynamic Hydrogen Gradients in Ti-6Al-4V: *Christopher Schuh*[1]; David C. Dunand[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA

Internal-stress plasticity is a Newtonian deformation mechanism which operates at low applied stress levels, when there is a concurrent internal stress. Common sources of internal stress are thermal expansion or phase transformation mismatch; in this work we explore the possibility of chemically-induced internal stresses. We report tensile creep experiments on the BCC beta-phase of Ti-6Al-4V at 1030°C, in which dynamic gradients of hydrogen concentration were introduced through cycling of the test atmosphere (between Ar/H2 mixture and pure Ar). As expected, low applied stresses lead to Newtonian deformation which is characteristic of internal stress plasticity, and which reflects a large enhancement in the average creep rate. Also, we present an analytical model which considers chemical, elastic, and creep strains during chemical cycling, and find good agreement with the experimental results.

11:10 AM
Time-Dependent Inelastic Deformation of Pure Copper at Low Homologous Temperature: *Chen-Ming Kuo*[1]; Yao-Chang Huang[2]; [1]I-Shou University, Dept. of Mechl. Eng., 1, Section 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan; [2]I-Shou University, Dept. of Matls. Sci. & Eng., 1, Section 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

Deformation mechanisms of time-dependent inelastic behavior of pure metals are attributed to the dislocation motions and their interactions with each other or other kinds of obstacles such as impurities. Mathematical modeling of these mechanisms is generally considered as thermally activated energy of dislocation past obstacles and structural evolution underway activated motion of dislocations. However, it has been proposed that other mechanism such as stress-rate consideration as mechanically activated energy shall be included as well. In this study, stress-rate change experiments are conducted during room temperature creep test of pure copper. Numerical calculations based upon thermally activated kinetic flow and structural evolution laws are compared with experimental data. Excellent agreement is observed between these two comparisons. It concludes that the mathematical models of thermally activated deformation theory and the structural evolution can completely describe the phenomena of metallic time-dependent inelastic flow at low homologous temperature.

11:30 AM
Cavity Characteristic in High-Strain-Rate Superplastic Aluminum Matrix Composite: *Hiroyuki Hosokawa*[1]; Mamoru Mabuchi[1]; Hajime Iwasaki[2]; Kenji Higashi[3]; [1]National Institute of Advanced Industrial Science and Technology, Function-Adding Recycle Tech. Grp., Hirate-cho, Kita-ku, Nagoya, Aichi 462-8510 Japan; [2]Himeji Institute of Technology, Dept. of Matls. Sci. & Eng., Col. of Eng., Shosha, Himeji, Hyogo 671-2201 Japan; [3]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., Col. of Eng., Gakuen-cho, Sakai, Osaka 599-8531 Japan

It has been demonstrated that many aluminum matrix composites show superplastic behavior. Superplastic behavior of composites is related to high strain rate sensitivity or low stress exponent. However, it is recognized that largest elongation doesn't coincide with high strain rate sensitivity; because cavitation behavior is also associated with elongation. In superplastic deformation, the fracture is generally associated with the cavitation. The cavitation also deteriorates the performance of mechanical properties after deformation. Therefore, it is required that cavity forming is inhibit in order to obtain large elongation and to maintain the mechanical properties. It is known that liquid phase is important to inhibit cavity forming as an accommodation helper. However, when stress concentrations are relaxed satisfactorily, a liquid phase is not need to inhibit cavity forming, resulting in large elongation. In the present investigation, the critical size of the reinforcement for cavity forming is investigated in high-strain-rate superplastic aluminum matrix composite.

11:50 AM
Superplasticity-Like Behavior of Ultralight bcc Beta Phase Based Mg-11.8wt%Li-2.65wt%Zn Alloy: *F. R. Cao*[1]; F. Lei[1]; J. Z. Cui[1]; J. L. Wen[1]; [1]Northeastern University, Dept. of Matls. Forming & Control, Sch. of Matls. & Metall., Shenyang 110006 China

The mechanical behavior of Mg-11.8wt%Li-2.65wt%Zn alloy (bcc beta phase based) at elevated temperatures was investigated at 473-623K and initial strain rate of 5×10^{-2}/sec-5×10^{-4}/sec. The maximum elongation to failure of 250% has been demonstrated in such coarse-grained alloy (average grain size 66.7 μm after holding time before high temperature deformation) at 573K and initial strain rate of 5×10^{-2}/sec. The coarse-grained alloy exhibits a stress exponent of approximately 3, and fractured with significant development of necking. Optical microstructure shows that subgrain exist heterogeneously in the deformed coarse-grained interior. Transmission electron microscope

examination of its structure reveals that many dislocations exist inside the grain. Above evidences such as the stress exponent 3, necking fracture appearance, existence of subgrain and dislocation indicate that the alloy exhibits superplastic-like or quasi-superplastic behavior and its deformation mechanism at ex perimental condition is dislocation creep (climb/glide) with subgrain, rotational dynamic recrystallization and limited grain boundary sliding instead of grain boundary sliding-dominated mechanism in classical superplastic material.

David L. Davidson Symposium on Fatigue: High Temperature Fatigue

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Jt. Mechanical Behavior of Materials, High Temperature Alloys Committee
Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; Thomas Zogas, Carpenter Technology Corporation, Reading, PA 19612-4662 USA

Tuesday AM — Room: 208
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Session Chair: Richard S. Bellows, Solar Turbines, Inc., M&PE, M/Z R-1, 2200 Pacific Hwy., San Diego, CA 92101 USA

8:30 AM Invited
Fatigue Properties of High Temperature Cast Blade Alloys: *Jacqueline B. Wahl*[1]; Ken Harris[1]; [1]Cannon-Muskegon, PO Box 506, Muskegon, MI 49443 USA

A consistent goal of gas turbine hot section technology development has been increased temperature capability to produce both improved fuel efficiency and higher thrust levels. This goal has been accomplished through the combination of design improvements (including cooling hole technology and the use of thermal barrier coatings) and casting improvements: both process and alloy developments. In recent years, the cost of introducing advanced technology has also become an important factor. Critical (and often competing) criteria in alloy development include creep resistance, oxidation performance, castability and microstructural stability. Although fatigue properties are not the primary alloy design property for hot section components, certainly fatigue performance is an important component design criteria. Process developments have introduced directionally solidified (DS) and single crystal (SX) casting technology. Alloy development has capitalized on the opportunities through the introduction of alloys specifically tailored to these processes, and families of DS and SX alloys have thus emerged. This paper will trace the progression of alloy development in the industry using Cannon-Muskegon alloys as representative of the various alloy families and examine pertinent fatigue properties (LCF, HCF, TF & TMF) of these alloys.

9:00 AM
The Effect of Crystallographic Orientation on Strain Localization in a Ni-Based Superalloy: *Luis E. Forero-Gomez*[1]; Fereshteh Ebrahimi[1]; [1]University of Florida, Matls. Sci. & Eng. Dept., 180 Rhines Hall, PO Box 116400, Gainesville, FL 32611 USA

Plastic strain localization usually reduces the resistance to fatigue crack initiation and propagation of metallic alloys. In single crystals, the degree of strain localization is dependent on the crystallographic orientation. The crystallographic orientations of both the loading (primary) and the crack front (secondary) directions affect the development of plasticity and hence the resistance to fatigue cracking. In this study the development of plasticity as functions of deformation and crystallographic orientation in single-crystal samples of a Ni-Based superalloy is investigated. Smooth dog-bone shaped and double-notched tension specimens were tested at room temperature. The evolution of slip localization was studied using optical profiling and scanning electron microscopy. The surfaces of the samples were electropolishesed and characterized before loading. Analyzing the perpendicular specimen surfaces identified the activated slip systems in the smooth samples. In this presentation the results for <100> and <110> loading directions with various crack front orientations are discussed.

9:20 AM
Cyclic Behaviour of Single Crystal Superalloys: *Pedro Dolabella Portella*[1]; [1]BAM, Dept. V, Unter den Eichen 87, Berlin D-12205 Germany

Single crystal superalloy find a wide application as blades or vanes in landbased and airborne gas turbines, especially in the highly loaded first stages. Due to their very regular microstructure it is possible to characterize the changes due to high temperature deformation and correlate them to the mechanical response of these materials. In this work we discuss the microstructural changes in single crystal superalloys observed during cyclic loading at high temperatures. The influence of hold times on cyclic softening, asymmetry in the hysteresis loops and failure mechanisms can be derived from the coarsening in the gamma/gamma structure. Parallel to this discussion it is possible to modify consistently the constitutive equations designed to predict the mechanical behaviour of these materials in order to simulate these phenomena. We further show the effect of crystal anisotropy and artificially introduced defects on crack initiation in uncoated specimens of single crystal superalloys. Using the constitutive equations derived for these materials was possible e.g. to determine the crack initiation sites in notched specimens.

9:40 AM
Micromechanisms of Fatigue Crack Growth in Structural Aerospace Alloys: Christopher Mercer[1]; Sassan Shademan[2]; Weimin Shen[1]; J. Oh[3]; *Winston O. Soboyejo*[1]; [1]Princeton University, Princeton Matls. Inst. & the Dept. of Mechl. & Aeros. Eng., Engineering Quadrangle, Olden St., Princeton, NJ 08544 USA; [2]The Ohio State University, Dept. of Matls. Sci. & Eng., Columbus, OH USA; [3]The Ohio State University, Dept. of Mechl. Eng., Columbus, OH USA

This paper presents an overview of fatigue fracture modes in structural aerospace alloys. These include mechanisms of fatigue crack growth in the near-threshold, Paris and high-delta K regimes obtained from titanium alloys, nickel-base superalloys and an aluminum alloy (Al 7050). Fatigue fracture modes in these materials are shown to be strong functions of the stress intensity factor range, delta K, and the maximum stress intensity factor, Kmax. Fatigue mechanism maps are also presented to show the parametric ranges of delta K and Kmax corresponding to the different fatigue fracture modes.

10:00 AM Break

10:15 AM
Thermo-Mechanical Fatigue Properties of a Third Generation Single Crystal Superalloy TMS-75: *Hao Zhou*[1]; Hiroshi Harada[1]; Yoshikazu Ro[1]; Yutake Koizumi[1]; Toshiharu Kobayashi[1]; Ikuo Okada[2]; [1]National Institute for Materials Science, High Temp. Matls. 21 Proj., 1-2-1 Sengen, Tsukuba Science City, Ibaraki 305-0047 Japan; [2]Mitsubishi Heavy Industries, Ltd., Takasago R&D Ctr., 2-1-1 Shinhama Arai-cho, Takasago, Hyogo 676-8686 Japan

Thermomechanical fatigue (TMF) behaviour of a single crystal superalloy TMS-75 has been studied. It was shown that the lifetimes of TMF for the samples with a dwell time in the compression phase drop greatly by an order of magnitude as compared with ones without a dwell time. The microstructure and the rupture surface were observed by scanning electron microscopy. Quantitative analyses of dislocations and stacking faults were performed by transmission electron microscopy. It was found that the dwell period played an important role for the fracture process. A transition from elastic strain to plastic strain due to a stress relaxation is responsible for yielding a plastic deformation in the tension phase and causing a drastic reduction of the TMF lifetime. This work suggests that designing single crystal superalloys with higher resistance to compressive stress relaxation is an effective way to improve the TMF property.

10:35 AM
Effects of High Temperature Exposures on Fatigue Life of U720: *Tim P. Gabb*[1]; Jack Telesman[1]; Peter T. Kantzos[2]; Joseph W. Sweeney[3]; Paul F. Browning[4]; [1]NASA Glenn Research Center, MS 49-3, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [2]Ohio Aerospace Institute, MS 49-6, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [3]Gilcrest Company, MS 49-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [4]Solar Turbines, Inc.

Low cycle fatigue tests conventionally used to characterize the low cycle fatigue resistance of disk superalloys are usually performed at cyclic frequencies of 0.33 Hz or faster. This is so in the interests of time and cost. However, service conditions for disks in some aerospace applications and land-based gas turbine engines can produce major cycle periods extending to hours and days. Over service life, this can produce total service times exceeding 1,000 hours for aerospace applications and exceeding 100,000 hours for land-based applications. The costs of running strain-controlled low cycle fatigue tests in this

manner would be prohibitive from both time and cost considerations. However, some aspects of the surface effects of realistic total exposure times can be considered economically. The purpose of this study was to examine the effects of extended exposures on the near-surface fatigue resistance of a disk superalloy. Powder metallurgy processed supersolvus heat treated U720 fatigue specimens were exposed in air at temperatures of 650 to 705C, for times of 100 to 1000h. They were then tested using conventional fatigue tests at 650C, to determine the effects of exposure on fatigue resistance. The exposures significantly affected fatigue life. The exposures reduced life and increased the scatter in life, compared to unexposed levels. Fractographic evaluations indicated the failure mode was shifted by the exposures from internal to surface crack initiations.

10:55 AM
Thermographic Detection of Fatigue Crack Growth Behavior in a Superalloy: *Hsin Wang*[1]; Lijia Chen[2]; D. L. Klarstrom[3]; Peter K. Liaw[2]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., Bldg. 4515, MS-6064, Rm. 231, Oak Ridge, TN 37831-6064 USA; [2]University of Tennessee, Matl. Sci. & Eng., 327 Dougherty Bldg., Knoxville, TN 37996 USA; [3]Haynes International, Inc., Kokomo, IN 46904 USA

A high-speed, high-sensitivity infrared (IR) camera has been employed to monitor fatigue crack growth behavior of ULTIMETâ superalloy. Temperature maps around the crack tip were used to determine the crack tip location and plastic zone. Temperature oscillation surrounding the crack tip was also observed during tension-tension fatigue tests. IR images were taken to monitor the propagation of a fatigue crack. The fatigue crack growth rate was determined from the IR images. Infrared thermography provides a powerful addition to study fatigue crack propagation characteristics.

11:15 AM
Effect of Inclusions on Low Cycle Fatigue Behavior of P/M Nickel-Base Superalloy Rene95: *Xishan Xie*[1]; Lina Zhang[1]; Jianxin Dong[1]; [1]University of Science & Technology-Beijing, Matls. Sci. & Eng., 30 Xueyuan Lu, Beijing 100083 China

Inclusions in nickel-base superalloy Rene95 can be considered as the crack initiation sites at low cycle fatigue (LCF) tests. It is critically harmful not only for LCF properties of P/M Rene95 but also on the service lives of P/M Rene95 disks of aero-engines. For understanding the inclusion behavior in P/M Rene95, the LCF tests were conducted with commercial P/M Rene95. Experimental results show the evidence of crack initiation at inclusions, which accelerated crack propagation and shortened LCF lives. Special prepared P/M Rene95 doped with alumina seeds was focused to study the inclusion effect on LCF of P/M Rene95. In consideration of direct observation on inclusion behavior at LCF, SEM in-situ fatigue test and simultaneous observation were conducted. Experimental results show the crack initiation mode at inclusions and further crack propagation. The inclusion effect on crack initiation and propagation in P/M Rene95 was discussed and the physical mode of inclusion effect on LCF was suggested.

Fundamentals of Advanced Materials for Energy Conversion: Fuel Cells

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee
Program Organizers: Renato G. Bautista, University of Nevada-Reno, Department of Chemical and Metal Engineering, Reno, NV 89557-0136 USA; Dhanesh Chandra, University of Nevada-Reno, Metallurgical & Materials Engineering, Reno, NV 89557 USA

Tuesday AM Room: 613
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Andrew Payzant, Oak Ridge National Laboratory, HTML, 1 Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6064 USA; Jai-Young Lee, Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., Kusong-dong 373-1, Taejon 305-701 S. Korea

8:30 AM Invited
Bismuth Oxide Based Ceramics for Oxygen Ion Transport-Crystallography and Properties: *Edward Andrew Payzant*[1]; Stephen D. Nunn[1]; Fred C. Montgomery[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., 1 Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6064 USA

Alkaline earth doped rhombohedral beta-Bi2O3 exhibits oxygen ion conductivity that is much higher than yttria-stabilized ZrO2 (YSZ) up to a maximum operating temperature of about 800°C. The conductivity increases by nearly ten-fold due to a subtle phase change in the temperature range of 560°C to 745°C, depending upon the amount and type of dopant in the composition. The ionic conductivity of the high temperature form of beta-Bi2O3 is 35 to 65 times greater than that of YSZ at the same temperature, but poor stability in reducing atmospheres focuses its applications on gas separation rather than SOFC. Other Bi2O3-based compositions include two-phase compositions that contain pure Bi2O3 in addition to the beta-Bi2O3 phase. At elevated temperatures, the two-phase compounds transform to the cubic delta-phase, which has very high ionic conductivity. X-ray and neutron diffraction analysis studies will be presented with emf and oxygen ion flux measurements through the solid electrolyte.

9:00 AM Invited
Oxidation Behavior and In-Cell Performance of Developmental Cladding Alloys for Intermediate Temperature Solid Oxide Fuel Cell (SOFC) Metallic Interconnectors: Michael P. Brady[1]; *Bruce A. Pint*[1]; Timothy R. Armstrong[1]; Chris E. Milliken[2]; Eric D. Kreidler[2]; [1]Oak Ridge National Laboratory, MS 6115, Oak Ridge, TN 37831-6115 USA; [2]Technology Management, Inc., Cleveland, OH USA

Metallic interconnectors offer the potential for better mechanical properties and significantly lower cost than perovskite-type ceramic interconnectors in planar SOFC's. However, they react in the SOFC operating environments to form oxide scales, which can significantly degrade electrical conductivity and cell performance. Metals also tend to have higher thermal expansion coefficients than the zirconia-based SOFC electrolyte, which can lead to failure during thermal cycling. A single alloy may be unable to meet all of the requirements for interconnector materials. Development of ferritic and Ni-base cladding alloys for use as overlays on lower thermal expansion metallic substrates may provide an answer. The results of an evaluation of oxide scale formation on a series of microalloyed Cr2O3-forming ferritic and NiO-forming Ni-base alloys will be presented. This data will be compared to relative in-cell performance in model stacks operated at 850°C using humidified hydrogen as fuel.

9:30 AM Invited
Inorganic Proton Conductors as Fuel Cell Electrolytes: *Sossina M. Haile*[1]; [1]California Institute of Technology, Matls. Sci., 138-78, 1200 California Blvd., Pasadena, CA 91125 USA

Polymer electrolyte fuel cells, those most viable for mobile applications, suffer from the humidification requirements of the polymer, which limits the temperature of operation to ~100ºC, and from its permeability to methanol and hydrogen, which lowers fuel efficiency. We report here the operation of fuel cells based on inorganic proton-conducting electrolytes, specifically, "solid acids", that address many of the issues facing polymer fuel cells. Solid acids are compounds whose chemistry and properties are intermediate between those of a normal acid and a normal salt. The proton conductivity of these materials can reach values as high as 10^{-2} S/cm when heated to slightly elevated temperatures. The transport process does not require humid atmospheres and the materials are stable to temperatures as high as 250ºC. Ultimately, such fuel cells may greatly simplify the fuel cell balance of plant by eliminating humidification hardware, relaxing CO removal requirements and enabling operation of high efficiency direct-methanol fuel cells.

10:00 AM Invited
Synthesis and Processing of Nanocrystalline Thin Films: *Michael Z.-C. Hu*[1]; Paul F. Becher[2]; Junhang Dong[1]; E. Andrew Payzant[2]; Timothy R. Armstrong[2]; [1]Oak Ridge National Laboratory, Cheml. Tech. Div., 1 Bethel Valley Rd., POB 2008, Oak Ridge, TN 37831-6224 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., 1 Bethel Valley Rd., POB 2008, Oak Ridge, TN 37831-6064 USA

If operated at low temperature, solid oxide fuel cells (SOFC) can be highly efficient to generate electric power for both stationary and mobile applications. However, conductivity of electrolyte thin layer has to be high enough to make a practical fuel cell. SOFC commonly use yttria-stabilized zirconia (YSZ) films as electrolytes, which have a thickness around 20-500 μm and grain size from a few microns to over ten microns in conventional system. High temperatures (normally about 1000°C) are necessary for operation of the current SOFC. Recent research has demonstrated that much higher ionic conductivities at significantly lower temperatures (<800°C) can be achieved by nanocrystalline YSZ thin films (with thickness ~1 μm and crystallite size 10-30 nm). Therefore, the nanocrystalline YSZ thin films can be a key in development of new-generation SOFC that can be operated at a temperature as low as 600°C. However, fundamental understanding of nanocrystallite growth under material processing or operation con-

ditions and its impact on conductivity are far from being developed. In addition, the currently reported nano-phased thin YSZ film fabrication method (i.e., spin coating of polymeric precursor) requires unreasonably large number of coating times (>20) and thus is impractical for efficient, cost-effective, well-controlled, large-scale fabrication. In this work, we will report a new synthesis method for crack-free nanocrystalline YSZ films, which significantly reduces the numbers of coating times and improves film quality control. Results on the kinetics of nanocrystal grain growth in thin YSZ films under various temperatures and atmospheres, as well as grain-size effect on ionic conductivity will be also discussed.

10:25 AM Break

10:35 AM Invited
Water-Gas Shift in a Palladium Membrane Reactor: *Stephen N. Paglieri*[1]; Stephen A. Birdsell[1]; [1]Los Alamos National Laboratory, ESA-TSE, PO Box 1663, MS-C348, Los Alamos, NM 87545 USA

A palladium membrane reactor (PMR) holds promise for efficient production of H_2 for fuel cell use from feedstocks such as gasified coal while simultaneously concentrating the product CO_2 for sequestration. The water-gas shift reaction was carried out using a thin film palladium composite membrane at pressures up to 200 psia over several catalysts including an Fe-Cr-Cu oxide high temperature shift catalyst. The Pd/alumina composite membrane had higher H_2 flux but lower permselectivity than previously used 178 micron thick Pd-Ag tubes. The influence of variables on PMR performance such as H_2O:CO ratio, residence time, membrane permselectivity, and temperature will be discussed. The impact of sulfur (H_2S) on both membrane and catalyst was evaluated.

11:00 AM
PEM Fuel Cell Bipolar Plate-Material Selection, Design and Integration: *Atul Kumar*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Metlgcl. & Matls. Eng., A-129, Bevill Bldg., 126 Seventh Ave., Box 870202, Tuscaloosa, AL 35487 USA

In the present research a two-cell polymer electrolyte membrane fuel cell stack (PEMFCS) with SS-316 bipolar plates has been tested. Different flow field designs were studied and a new multi-parallel channel design is proposed. The stack is stable within ±3 mV even after 1000 hours of continuous operation, indicating efficient water management and effective removal of corrosion products from the stack. Also no iron was detected in membrane electrode assembly (MEA) by EDAX analysis. Experiments for studying the effect of pressure and temperature on the stack were carried out. Results indicates that high pressures beyond 138 kPa did not significantly increase the performance of cell, however added much to the pressurization cost of the system. Tests at different temperatures showed that at temperatures below 80°C external humidification could be completely. The prototype stack was also tested under varying load conditions simulating highway-driving schedule. Three-dimensional numerical modeling was done using the commercial CFD software (FLUENT 5.5). The experimental efficiency of PEMFCS is 49%, which is in good agreement with the model. Results showed that 59% of the total losses is due to ohmic polarization in the cell. These losses are addressed by optimizing the compaction pressure of stack and surface modification of bipolar plates. Application of the stack in automotive transportation system is in progress.

11:25 AM
A Wet Gel Crystallization Method for Synthesis of Zeolite Membranes and Coatings: *Junhang Dong*[1]; Michael Z.-C. Hu[1]; Edward Andrew Payzant[2]; David W. DePaoli[1]; [1]Oak Ridge National Laboratory, Cheml. Tech. Div., 1 Bethel Valley Rd., POB 2008, Oak Ridge, TN 37831 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., 1 Bethel Valley Rd., Oak Ridge, TN 37831 USA

Zeolite membranes are a new class of separative and catalytic materials with great potentials in gas separation and hydrogen generation. Current synthesis methods, including in-situ crystallization and vapor-phase transport methods have serious drawbacks that obstruct the implementation of large-scale production. In the new method of this study, a thin layer of templated, wet-gel precursor is precoated on a porous alumina substrate. Then the gel layer is converted to an MFI zeolite film by vapor-phase treatment at elevated temperatures. High quality MFI zeolite films were formed on porous α-alumina substrates by this new approach. Effects of the templating molecules and vapor phase compositions on the resultant zeolite structure and membrane quality have been investigated. The method developed here has the advantages of improved controllability of synthesis process, minimal waste generation, and reduced chemical consumption that are highly desirable for large-scale production of zeolite membranes.

11:45 AM
LaCrO3-Based Coatings on Ferritic Steel as Solid Oxide Fuel Cell Interconnect: *J. H. Zhu*[1]; Y. Zhang[1]; A. Basu[1]; M. Paranthaman[2]; D. F. Lee[2]; E. A. Payzant[2]; C. J. Rawn[2]; [1]Tennessee Technological University, Dept. of Mechl. Eng., 115 W. 10th St., TTU Box 5014, Cookeville, TN 38505 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., Oak Ridge, TN 37931 USA

The application of chromia-forming ferritic steels for solid oxide fuel cell (SOFC) interconnect must address a number of problems such as Cr migration from the interconnect onto the cathode side and insufficient protectiveness of the thermally grown chromia oxides, which lead to rapid degradation of the cell performance. A thin layer of doped lanthanum chromite on ferritic steel may act as a protective coating to mitigate these problems and facilitate the use of metallic interconnect in SOFC. In this paper, novel approaches including sputter deposition and sol-gel processing are used for synthesizing doped LaCrO3 coatings. The coating formation mechanism, compositional homogeneity and microstructure, etc. are characterized in detail. The oxidation resistance and electronic conductivity, long-term stability of the coated ferritic steel under fuel-cell operating environments are also evaluated. Based on the experimental results, the suitability of doped LaCrO3 coated ferritic steel as an interconnect material for SOFC application is discussed.

Fundamentals of Structural Intermetallics: Deformation Behavior of Intermetallics

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials
Program Organizers: Young-Won Kim, UES, Inc., Materials & Processing Division, Dayton, OH 45432 USA; Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Vijay K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Tuesday AM — Room: 615-616
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Vijay Vasudevan, University of Cincinnati, Dept. of Matls. Sci. & Eng., Cincinnati, OH 45221-0012 USA; Mark Aindow, University of Connecticut, Metall. & Matls. Eng., 97 N. Eagleville Rd., Unit 3136, Storrs, CT 06269-3136 USA

8:30 AM Invited
Nonconservative Dislocation Mechanisms for Yield Strength Anomaly: *Man H. Yoo*[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., Oak Ridge, TN 37831-6115 USA

Dislocation microstructures developed by {110}<111> slip in B2 FeAl, the pyramidal slip in D019 Ti3Al, and the equivalent slip systems in refractory-metal disilicides (MSi2) of C11b, C40, and C54 structures, reported in the literature, indicate that immobilization processes of non-screw dislocation segments may be attributive to the measured critical resolved shear stresses (CRSSs). In this work, the activation enthalpy for the formation of a jog pair on edge and 60° dislocations is formulated in terms of the elastic interaction energies, APB/SISF energies, and internal climb stresses. The critical stress for dynamic breakaway from pinning points (cusps) created by vacancy-dislocation contact interaction is derived for the two limiting cases of low/high temperature and high/low stress. Available data on the anomalous temperature dependence of CRSSs in Fe-39Al, Ti3Al, MoSi2 and NbSi2 single crystals will be discussed in view of the proposed mechanisms.

9:00 AM
Theory of Anomalous Yield Stresses: *John J. Gilman*[1]; [1]UCLA, Matl. Sci. & Eng., 6532 Boelter Hall, MC 159510, Los Angeles, CA 90095-1595 USA

In several substances, the yield stress increases with increasing temperature; e.g., nickel aluminide. The conventional explanation of this phenomenon is in terms of Kear-Wilsddorf dislocation reactions resulting in traps which decrease net dislocation mobilities. However, this requires a considerable amount of statistical coordination, limiting its plausibility. Another possibility is a variation on a theme proposed by Ardley and Cottrell long ago. Namely, that the kinks associated with dislocation motion in superlattices have complex structures and therefore large configurational entropies (unlike kinks in simpler struc-

tures). As a result, the free-energy of kink motion becomes increasingly negative with increasing temperature in the temperature interval of interest. Thus there is an intrinsic decrease of dislocation mobility, and an increasing yield stress. This accounts for the large mechanical stabilities of these substances.

9:20 AM
Decomposition of <111> Superdislocation as a Possible Mechanism of the Yield Strength Anomaly (YSA) in B2-Ordered FeAl Alloys: *Anna Fraczkiewicz*[1]; Olivier Calonne[1]; Francois H. Louchet[2]; [1]Ecole des Mines de St-Etienne, Centre SMS, URA CNRS 1884, 158 Cours Fauriel, St-Etienne 42 100 France; [2]LTPCM/INPG, Domaine Universitaire, BP 75, St-Martin d'Heres 38 054 France

Dislocation structures in deformed B-doped B2 FeAl were studied in situ (TEM straining experiments) and post mortem (conventional TEM). Deformation is strongly localised in the regime of YSA and becomes homogeneous above the YS peak. In the domain of YSA, decomposition of a <111> superdislocation is observed (<111>• <110>+<001>). Moreover, <110> dislocations, stretched in their edge direction, are present. Their anisotrope shape suggests high mobility of the screw segments and of a lower mobility of the edge parts: this anisotropy is supposed to be closely related to the segregation of boron to dislocation lines. This phenomenon explains also the very important shift of the YS peak (+175°C, +200 MPa) due to boron in FeAl [1]. These observations confirm that the strengthening of FeAl in the YSA domain may be due to the decomposition of <111> superdislocations (as proposed previously be Yoshimi and confirmed by Morris[2]). Moreover, they are consistent (at a microscopic scale) with (macroscopic) mobile dislocation exhaustion-multiplication models [3]. [1] O. Calonne, A. Fraczkiewicz, F. Louchet, Scripta Metall. Mater., 43, 69-75, (2000). [2] D. G. Morris, M. A. Morris, Intermetallics 5, 245, (1996). [3] F. Louchet Phil. Mag. A, 72(4), 905, (1995).

9:40 AM
The Role of Dislocation Generation in the Deformation of B2 Intermetallics: *Wei-Jun Zhang*[1]; Seetharama C. Deevi[1]; Rangaraj S. Sundar[1]; [1]Chrysalis Technologies, Inc., Rsrch. Ctr., 7901 Whitepine Rd., Richmond, VA 23237 USA

The brittleness of B2 compounds at room temperature has been attributed to various causes such as environmental effects in FeAl, insufficient slip in NiAl and weak grain boundaries in FeCo. However, questions remain in understanding some of the experimental results regarding the ductility and strength of B2 compounds. For example, (a) why does the yield strength of Fe-40Al single crystal decrease with the ductility as the strain rate decreases. (b) Why does the pre-straining and surface polishing increase the ductility but decrease the yield strength of NiAl single crysta. (c) Why does the presence of fine borides in FeCo-2V alloy improve the ductility but concurrently decrease the yield strength. In this paper, an alternative mechanism is proposed to explain the above results in terms of dislocation generation in B2 compounds. The analysis suggests that the ease of dislocation generation has a significant influence on the deformation of B2 intermetallics. The proposed model is supported by our experimental results on FeCo-base and FeAl-base alloys.

10:00 AM Invited
First Principles Simulation of Ordinary Screw Dislocations in Gamma-TiAl: *Christopher Woodward*[1]; Satish I. Rao[1]; [1]UES, Inc., Matls. Rsrch. Div., 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

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

10:30 AM
A 3D Mesoscopic Simulation of Dislocation Behaviour in Gamma TiAl in the Stress Anomaly Domain: Marc C. Fivel[1]; *Francois H. Louchet*[2]; Marc Verdier[2]; [1]INPG, GPM2, Domaine Universitaire, BP 46, St. Martin d'Heres 38402 France; [2]INPG, LTPCM, Domaine Universitaire, BP 75, St. Martin d'Heres 38402 France

A 3D mesoscopic simulation of dislocation behaviour is adapted from the case of FCC crystals. Screw dislocations are supposed to experience a Peierls type frictional stress and profuse cross slip, while edge motion is slowed down by athermal friction. It is shown that ordinary dislocation motion essentially proceeds through a series of pinning and unzipping processes on screws. Intrinsic pinning occurs on cross-slip generated jogs. Cusp unzipping restores the screw character of dislocations. The obtained microstructure, and particularly the characteristic cusped screw dislocations, fully agrees with TEM observations. The increase of pinning rate with temperature eventually results in mobile dislocation exhaustion, which confirms the Louchet-Viguier anomaly model. The simulation is also run in the case of several interacting dislocations, in order to evidence a possible collective dislocation behaviour. The influence of extrinsic pinning points, often debated in the literature as a possible reason for stress anomaly, is also tested.

10:50 AM Invited
The Critical Temperature in the Initiation of Plastic Strain in NiAl along the [001] Hard Orientation: *Y. Q. Sun*[1]; [1]University of Illinois, Dept. of Matls. Sci. & Eng., 1304 W. Green St., Urbana, IL 61801 USA

In intermetallic compound NiAl, [001] is a special orientation along which the yield strength is several times larger than the other orientations. In this work, the anisothermal creep method is used to probe the initiation of plastic strain in this orientation. The experiments show that the plastic strain is initiated at a well-defined temperature. The initiation temperature is rate-independent at low heating rates and defines a critical temperature. The dislocation structure in the deformed crystals has been observed in TEM and showed that the initiation of plastic strain is controlled by the non-<100> dislocations. The mechanisms responsible for the abrupt onset of plastic strain are discussed. The competition between this mechanism and dislocation multiplication is analyzed using the experimental results.

11:20 AM
Dislocation and Twinning Strain Transfer at Grain Boundaries in Equiaxed TiAl Alloys: *Darren E. Mason*[1]; Benjamin A. Simkin[1]; Thomas R. Bieler[1]; Martin A. Crimp[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., E. Lansing, MI 48824-1226 USA

The nature of grain boundary deformation transfer versus microcrack initiation has been examined in equiaxed gamma-gamma TiAl alloys as a function of grain orientation and grain to grain misorientation. The orientations of grains on the tensile surface of 4-point bend specimens have been determined and mapped using electron back scattered diffraction (EBSD) complemented with selected area channeling patterns (SACPs). The specimens were subsequently deformed either in-situ SEM or ex-situ to initiate microcrack nucleation. Electron channeling contrast imaging (ECCI) in SEM was then used to image near surface dislocations and microtwins in the deformed specimens. In particular, locations where grain to grain deformation transfer or microcrack initiation have occurred have been studied. The active deformation systems at these locations have been identified based on trace analysis of the {111} dislocation slip and twinning planes in conjunction with known crystal orientations derived from SACP and EBSP information. In general, grain boundaries where extensive deformation transfer has occurred have been found between grains where both have high Schmid factors for the active deformation systems and small to moderate misorientations between the grains. Also, a directionality criterion for deformation transfer has been identified for conditions involving deformation microtwins. That is, impingement of deformation twinning on a grain boundary may induce either twinning or dislocation slip on the other side of the boundary, but the reverse process may not be favorable due to the unidirectionality of twinning. Complimenting these experimental observations, a new multi-scale analytical model of slip transfer will be discussed. This approach couples within a nonlinear functional a nonconvex variational model of dislocation distribution evolution in individual grains to phenomenological representation of the influence of grain boundary geometry, crystal disorientation, and mesoscopic stress state on slip transfer. Preliminary results from the application of this hybrid model to a simple bicrystalline system will be discussed. This work has been supported by the Air Force Office of Scientific Research under Grant No. F49620-01-1-0116 and the Michigan State University Composite Materials and Structures Center.

11:40 AM
Static and Dynamic Strain Ageing in Two-Phase Gamma Titanium Aluminides: *Ulrich Fröbel*[1]; Fritz Appel[1]; [1]GKSS Research Centre, Inst. for Matls. Rsrch., Max-Planck-Strasse, Geb 45, WFP, Geesthacht, D-21502 D-21502 Germany

The deformation behaviour of two-phase titanium aluminides was investigated in the intermediate temperature interval 450-750K. The observed deformation characteristics include discontinuous yielding and negative strain rate sensitivity, which are indicative of the operation of the Portevin-LeChatelier effect. The pinning processes occurring at the dislocations and associated with these phenomena were studied by static strain ageing experiments. A wide range of alloy compositions and microstructures was investigated in order to identify the relevant defect species. Accordingly, dislocation pinning occurs with fast kinetics and is characterized by a relatively small activation energy of 0.7 eV, which is not consistent with a conventional diffusion process. Furthermore, the strain ageing phenomena are most pronounced in Ti-rich alloys. This gives rise to the speculation that antisite defects are involved in the pinning process. The implications of the ageing processes on the deformation and fracture behaviour of two-phase titanium aluminide alloys will be discussed.

General Abstracts: Extractive Metallurgy

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA

Tuesday AM — Room: 601
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Mike Wadsley, Austherm Pty. Ltd., North Brighton, Victoria 3186 Australia

8:30 AM
Fluidity of Silicate Slags from a Hazardous Waste Incinerator: Zhijing Zhang[1]; *Mario Arenas*[1]; Ramana G. Reddy[1]; [1]University of Alabama, Metlgcl. & Matls. Eng. Dept., PO Box 870202, Tuscaloosa, AL 35487 USA

The management of hazardous wastes is important in a society which is increasingly concerned with environmental issues. This task can be achieved using incinerators such as the slagging rotary kiln which is utilized in many plants in the Unites States and other industrialized countries. However, in order to assure a smooth operation of the rotary kiln, the melt should have both a low melting point and high fluidity. In this study, the fluidity of various hazardous waste rotary kiln slags was measured in terms of length of flow using the crucible test technique. It was found that the slags have melting points ranging from 1372 to 1424°C while their fluidities at 1450°C varied from 61-102 mm. The effect of addition of oxides on the fluidity of a slag containing 47% SiO_2 was also investigated. For this slag, additions of SiO_2, FeO and Na_2O increased fluidity, whereas the addition of CaO and Al_2O_3 resulted in lower fluidity values.

8:55 AM
Carbothermic Reduction of Alumina using Plasma Quench: *Alan Donald Donaldson*[1]; Ronald Alan Cordes[1]; [1]Plasma Quench Technologies, Inc., 101 Technology Dr., Idaho Falls, ID 83401 USA

Abstract unavailable

9:20 AM
Dissolution Rate of Alumina into Molten CaO-SiO2-Al2O3 Slag: *Ja-Yong Choi*[1]; Hae-Geon Lee[1]; [1]Pohang University of Science and Technology, Dept. of Matls. Sci. & Eng., San-31, Hyoja-dong, Nam-Gu, Pohang, KyungBook 790-784 S. Korea

The dissolution rate of alumina into CaO-SiO2-Al2O3 slag system was investigated at 1873K which was determined with the new rotating cylinder method by measuring torque variation on alumina cylinder using viscometer. The reliability of a new dissolution rate measuring method using viscometer was ascertained by comparing the results of the new method with the experimental results. It was empirically found that the torque on rotating alumina cylinder is proportional to the square of the cylinder radius. The dissolution rate increases with temperature and the rotation speed of cylinder. The activation energy of rate constant was found to be 22kcal/mol for 51.9CaO-48.1Al2O3 slag in mass %. The dissolution rate increases by increasing the CaO content and decreasing SiO2 content. It was found that the dissolution rate is highly dependent on the viscosity of slag rather than concentration difference. Using the experimental results, the map of iso-dissolution rate lines was constructed for dissolution of alumina into CaO-SiO2-Al2O3 slag system. It was concluded that the dissolution of alumina into present slag system is controlled by slag phase mass transfer.

9:45 AM Cancelled
Sulfuric Acid Leaching of Turkish Chromite Ores: *Ahmet Geveci*

10:10 AM
A Kinetic Behavior of Desulfurization in Dynamic Conditions of Fe-S Liquid Drops Falling through Slag Layer: *Sin-Myoung Kang*[1]; *Hea-Geon Lee*[1]; [1]Pohang University of Science and Technology, Dept. of Matl. Sci. of Eng., San 31, Hyoja-dong, Nam-gu, Pohang S. Korea

This research was begun to develop the desulfurization process twice as fast than the present one, which is generally operated in hot metal pre-treatment. For this, new concept was needed. In present desulfurization process, the operation has being injected fine powders (slag) and blown a gas into the iron containing sulfur. However, in this research, it had been tried to speed up the desulfurization rate by falling the steel droplet containing sulfur into slag pool. This report is represented a laboratory research as the first step of that. This experiment was carried out under Fe-0.035 ~ 0.006 mass% S, CaO-38 ~ 52 mass% Al2O3-5 ~ 13 mass% MgO, 1873 ± 2 K and purified Argon atmosphere. And, whole process of experiment was recorded on videotape by using the X-ray fluoroscope. The desulfurization ratios are changed in the range of 93 ~ 10 % of with changing of slag component and the initial sulfur content and the size of droplets during the passing time. An area increasing by the liquid droplet deformed by drag, buyer and friction force was compensated base on Grace and Wairegi's proposal [1]. The desulfurization rate, although the reaction area is compensated, increases from 3×10-5 ~ ×10-4 mole-S/cm2sec with increasing of droplet diameter from 0.3 to 0.58 cm at any given condition which is minimal 5 times or maximal 35 times faster than Choi et. al.'s results [2]. Through the theoretical kinetic study, the reason of that is known as the stream in the droplet increases with increasing of droplet size. And, the rate determining step is also known as the metal phase mass transfer.

10:35 AM
Elecrothermal DC Furnace with Polarization of Bottom Phase (PDF Furnace): *G. S. Nus*[1]; A. V. Tarasov[1]; V. M. Paretsky[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., 129515, Moscow Russia

This furnace has been designed for processing of secondary raw materials (intermediate products, slags and wastes) at non-ferrous metallurgical plants and in similar processes. The furnace constitutes an electrothermal furnace with a large amount of slag and fed from a source of rectified current. Graphite or self-baking electrodes are combined into groups with different polarity, one of which is submersed into the molten metallic bottom phase (matte or metal) and the other one into molten slag. An automatic system ensures stable temperature control of the bottom phase irrespective of variations in the melt volume during the furnace operation. The system makes it possible to: realize the idea of a furnace bottom electrode without disturbing the sealing protective accretion layer, but preventing at the same time excessive formation of accretions on the bottom; concentrate the release of power introduced into the furnace primarily in the vicinity of electrodes submersed into the slag, creating thereby conditions of local overheating of molten slag and facilitating sublimation of volatile components of the slag (zinc, lead, etc.) with a constant temperature at the periphery of the melt, and as a consequence, without affecting the integrity of the furnace lining; combine during cathodic polarization of the bottom phase the zones of local overheating and natural sparging due to formation of carbon monoxide as a result of electrolysis, ensuring thereby additional intensification of metals reduction and sublimation of volatile components. Cleaning of converter slag from a copper smelter in a pilot plant using the PDF technology has ensured complete elimination of excessive bottom accretions, reduction in losses in discard slag: copper by 1.5 times, lead by 1.8 times and zinc by 2 times, as well as improvement of the furnace performance with respect to slag (an increase by 2 times) as compared with a similar furnace using AC. This development makes it possible to take advantage on a full industrial scale of the electrolysis of molten media, including electrochemical metals reduction, intensification of sublimation of volatile components and electrocapillary effect in relation to metallurgical processes for recovery of heavy non-ferrous metals; improvement of recoveries of valuable metals and recycling of difficult-to-treat secondary raw materials. Development of an engineering method for calculation of the PDF furnace parameters requires additional investigations of the process of natural convective heat ex-

change in a fluid (melt) with non-linear physical characteristics, including determination of a temperature field in a bath, and in particular, temperatures at the boundary with solid media (lining before accretion formation) in the presence of intensive internal unequally distributed sources of Joulean heat in a three-dimensional hollow with a complex configuration of boundaries. It boils down to a numerical solution of a non-linear three-dimensional system of differential equations with particular derivatives, possibly of elliptical type, under specific conditions of unambiguity. There are no similar developments in the world practice.

11:00 AM
The Characteristics of the Rare Earth Silicide Alloy Prepared in the Laboratory: *Zhao Qun*[1]; Xie Yanli[1]; Tu Ganfeng[1]; [1]Northeastern University, Sch. of Matl. & Met., PO Box 117, No.11, Ln. 3 Wenhua Rd., Heping Dist., Shenyang, Liaoning 110004 China

In this work, high grade rare earth silicide alloy was prepared in the laboratory. During the smelting procedure, little waste slag was produced and the bottom of the furnace was kept steady, as well as the reaction in the furnace that took place completely, almost all RE element was enriched in the alloy. At same time, the behaviors of phosphors as the main harmful contaminants in raw were studied during the carbon-thermal reduction process and some improvements were proposed to reduce the P content in the alloy. The RE silicide alloy prepared has special hydration-resistance and its disintegration property was also different from the conventional idea. The microstructure of the alloy was observed by SEM and the distribution of elements were also studied. As result, a hypothesis was set up to explain the distinct characteristics of the alloy.

11:25 AM
Synthetic Gypsum Production from Spent Sulfuric Acids of Copper Metallurgy: *Andrzej Chmielarz*[1]; [1]Institute of Non-ferrous Metals, Environment Protection, Sowinskiego 5, Gliwice Poland

The paper presents results of investigations on the process parameters for synthetic gypsum production from sulfuric acid blowdown streams of copper metallurgy (washing acid, tankhouse spent acid) by neutralization with limestone. The aim of the research works carried out was to create the optimal process conditions for gypsum crystals growth to meet requirements of the cement industry - final humidity below 10% and proper impurities concentration. There was investigated influence of retention time, concentration of acid and limestone suspension, solids concentration in a reactor, mixing conditions on obtained gypsum particles size and final humidity of gypsum. Experiments with different solid/liquid separation techniques (vacuum and pressure filtration, centrifugation) allowed to determine proper conditions for the process. Finally tests of gypsum properties as the additive in the cement production were carried out. It was found that synthetic gypsum made from spent acids could replace natural gypsum in the investigated application.

General Abstracts: Composite Materials

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA

Tuesday AM — Room: 209
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Nik Chawla, Arizona State University; Warren Hunt, Aluminum Consultants Group, Inc.

8:30 AM
Thermodynamics of In-Situ Processing Aluminum Alloy Matrix Composites: *Qingjun Zheng*[1]; Ramana G. Reddy[1]; [1]University of Alabama, Dept. of Metlgcl. & Matls. Eng., PO Box 870202, Tuscaloosa, AL 35487-0202 USA

Stability of ceramic reinforcements in Al alloy matrix and the effects of the materials and process variables on the in-situ formation of Al alloy matrix composites using gas bubbling method were modeled based on Gibbs energy minimization theorem. The experiments were carried out based on the results of thermodynamic modeling with methane, nitrogen, and ammona as the gaseous precursors in temperature range from 1223 to 1473 K. The composites were characterized using OM, SEM, XRD, and EDXA. The stability of reinforcements is dependent on temperature and the composition of Al alloy. The thermodynamic stability of AlN is higher than SiC in the Al alloys with low content of Si at high temperatures. The effects of the materials and process variables on the in-situ processing are discussed. Oxygen in nitrogen gas precursors is notably detrimental to the formation of AlN reinforcement.

8:55 AM
Utilization of Surface Forces and Transport Phenomena in Predicting Particle Incorporation in Electrodeposited Metal Matrix Composites: *Michael L. Free*[1]; [1]University of Utah, 135 S. 1460 E. Rm. 412, Salt Lake City, UT 84112 USA

Electrodeposition is an effective method of producing metal matrix composite coatings. Most existing models for predicting particle incorportation are based upon empirical expressions that are derived on the basis of convective mass transport to the deposition site. In the present study a different particle incorporation model, which involves a combination of mass transport and surface forces, will be discussed.

9:20 AM
Modeling of Glass Cenosphere Filled Epoxy Composites: *Nikhil Gupta*[1]; Eyassu Woldesenbet[1]; [1]Louisiana State University, Mechl. Eng. Dept., 2508, CEBA Bldg., Baton Rouge, LA 70803 USA

Incorporation of hollow particles (cenospheres) gives advantage of reduced density and increased specific strength of the composite material. Glass cenospheres tend to break easily under compressive loading conditions. The present study deals with developing a modeling approach for brittle cenosphere filled polymeric materials. Specific issues such as reduction in total volume of the material and generation of new surface due to the fracture of cenospheres are discussed. Ratio of inner to the outer radius (radius ratio parameter) of the cenospheres is an important parameter in deciding the stress state in the composite after the fracture of the particle. A critical value of the radius ratio parameter is calculated which separates two different types of stress states in the composite.

9:45 AM
Mechanical Properties of BSCCO and BSCCO/Ag Composites at 77K and 300K: *Javier LLorca*[1]; Alicia Salazar[1]; José Ygnacio Pastor[1]; [1]Polytechnic University of Madrid, Dept. of Matls. Sci., E. T. S. de Ingenieros de Caminos, Madrid 28040 Spain

The mechanical properties at ambient and cryogenic temperatures were measured in BSCCO 2212 rods produced by laser-induced directional solidification and in BSCCO 2223 and BSCCO 2223/Ag plates manufactured by hot pressing. Tensile tests in the longitudinal and transverse direction as well as flexure and fracture tests were performed in different orientations to ascertain the effect of various microstructural parameters (grain texture, porosity, dispersion of Ag particles) on the overall mechanical performance of the composites. It was found that the strength perpendicular to the c axis was significantly lower than in the a or b directions, and the textured samples were highly anisotrpic. In addition, the presence of Ag improved the strength and toughness but this effect was offset by the reduction in density owing to difficulties to manufacture fully-dense BSCCO/Ag composites. Similar results were obtained at 77 K and 300 K, indicating that the cryogenic temperatures did not modify the mechanical performance.

10:10 AM Break

10:45 AM
Electromagnetic Wave Absorption Properties of Amorphous Alloy-Ferrite Composites: *Kyung Mook Lim*[1]; Moon Chul Kim[2]; Chan Gyung Park[1]; [1]Pohang University of Science and Technology, Matls. Sci. & Eng., Namgu Hyojadong san 31, Pohang, Kyungbuk 790-784 Korea; [2]Research Institute of Science & Technology, Hyojadong Namgu, Pohang, Kyungbuk 790-785 Korea

In order to achieve sufficient electromagnetic wave absorption in high frequency range wave, absorbers made from the spinel-type ferrites requires the thick layer (>7mm) because of the permeability decreasing with increasing frequency. Amorphous magnetic materials have usually large permeability values over the Sneok's limit in high frequency ranges. The absorbing properties of amorphous metal flakes are, however, very poor due to a large difference between their permeability and permittivity. The synergetic effect of improved electromagnetic wave absorbing properties can, therefore, be expected by means of mixing spinel-type ferrite powder and amorphous alloy flakes. In the present study, a composite (less than 3mm in total thickness) of spinel-type ferrite (Ni-Zn) and amorphous alloy (FeSiBC) with an epoxy resin binder has been proposed. The wave absorbing property has been investigated in terms of complex permeability (mr), permit-

tivity (er) and reflection loss measured by a network analyzer in MHz-GHz frequency. The optimum fabrication condition for the composite with good wave absorption will be proposed.

11:10 AM Cancelled
High Temperature Oxidation of MoSi2/SiC Composites: *Dong Bok Lee*

11:35 AM
Studies on the Mechanical Behaviour of Aluminium-Zircon Composite: *P. V. Krupakara*[1]; [1]R V College of Engineering, Dept. of Chem., Mysore Rd., Bangalore, 560059 India

The present investigation aims to evaluate the mechanical properties of Aluminium-Zircon composites. Zircon particulates reinforced varying from three to seven percent by weight in steps of two percent under dry conditions. Al 6061 contains 1% magnesium, 0.6% copper, 0.255% of manganese, chromium 0.1%, balance aluminium. Composites are prepared by using liquid melt metallurgy technique using vortex method. Castings were cut, tuned and shaped into the required size to prepare the specimens for evaluation for the mechanical properties such as tensile, compression impact, fracture toughness, strength as per standards. Hardness test was also carried out. After testing the following results were noticed. Compression strength was increased by 11.29%, hardness increased by 48%, tensile strength increased by 13%, impact decreased by 17%, ductility reduced, rigidity increased, fracture toughness is also improved.

12:00 PM
Synthesis of Nanostructured Mn-Znferrite Powders by using a Novel Chemical Method: *Narendra N. Ghosh*[1]; [1]Birla Institute of Technology and Science, Chem., Vidya Vihar, Pilani, Rajasthan 33303 India

Research in the field of nanostructured ceramic powders have gained immense importance because of their potential application in many areas of technology. Technologically, fine-particle ferrites have been of interest due to their application in the preparation of high density ferrites at low temperatures, pigments and as catalysts. The surface properties and the microstructures of such powders, which control most of the parameters required for any particular application, often depend on the method of their preparation. The conventional ceramic method for the preparation of ferrites, though successful for large scale production of bulk powders because of its low cost and easy adaptability, have several limitations. Long heating schedules and high temperatures, the requisites for the ceramic route, sinter the final product and result in the loss of the fine particle nature of the powders. This method is thus affected by a poor control of the particle size, morphology and microscopic homogeneity. In the present investigation, an attempt has been made to establish a new chemical route, which is both affordable and versatile, for synthesis of the nanostructured mixed oxide ferrite powders. By using this chemical method a variety of ferrite powders with different compositions, such as ZnxMn1-xFe2O4, ZnxMn1-xFe2-y AlyO4 has been prepared. In this method nitrate salts of the different metals were used as starting materials. The aqueous solutions of the metal nitrates were mixed according to the molar ration of the compositions. Then the mixtures were mixed with an aqueous solution of water soluble polymer. This mixture after drying yield a brown floppy powders. These powders were then calcined at different temperatures ranging from 6000C to 9000C. Nanostructured powders were obtained from the thermal decomposition of the brown powders. The powders, prepared by calcinations at different temperatures, were characterized by using X-Ray diffraction analysis, thermal analysis (DTA/TGA), IR spectroscopic analysis. To study the influence of calcinations temperature on the particle size of the powders Transmission Electron Microscopic (TEM) study of the powders was performed. It was observed that the average particle size of the powders are in nanometer scale with a narrow size distribution. The average particle size of the powders was increased with the increase of calcinations temperature. This chemical method has proved to provide a convenient process for the preparation of nanostructured ceramic powders at comparatively low temperatures and offers the potential of being a simple and cost-effective route.

12:25 PM
Machinability Studies of PM Metal Matrix Composites on EDM: *P. Laxminarayana*[1]; V. S.R. Murti[2]; [1]Osmania University, Mech. Eng., Univ. College of Tech. (Autonomous), Hyderabad, Andhra Pradesh 500 007 India; [2]Osmania University, Mech. Eng., Univ. College of Eng. (Autonomous), Andhra Pradesh 500 007 India

In this study the influence of grain size and composition on the machinability of particulate aluminium matrix composites on electrodischarge machining (EDM) process are investigated. This powder metallurgy (PM) composite has an aluminium alloy matrix with reinforcement of silicon carbide particles. The machining was performed on CHARMILLES Electrodischarge machine with kerosene dielectric and copper electrode. The SiC content had a variation for 5 to 35 percent with 5 microns to 180 microns grain size variation. All machining parameters were kept uniform. The results broadly indicate the erosion mechanism in PM composites to include thermal shock & spalling, leading to high erosion rates. Higher grain size and lower percentage of SiC produces higher machining rate. The studies also include the tool wear and surface finish. Unlike conventional machining processes, the EDM of PM components exhibits inverse relationship between roughness and tool wear with machining rate.

General Abstracts: Ferrous Materials: Alloy and Stainless Steels

Sponsored by: TMS
Program Organizers: TMS, Warrendale, PA 15086 USA; George T. Gray, Los Alamos National Laboratory, Dynamic Properties Team, Los Alamos, NM 87545-0001 USA; Mark E. Schlesinger, University of Missouri, Department of Metallurgical Engineering, Rolla, MO 65409-0001 USA; Dan J. Thoma, Los Alamos National Laboratory, Materials Science and Technology, Los Alamos, NM 87545-0001 USA

Tuesday AM — Room: 211
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Aytekin Hitit

8:30 AM
Nucleation and Growth during Rapid Solidification of Fe-Si-B: *Halim Meco*[1]; Ralph E. Napolitano[1]; Matt J. Kramer[1]; [1]Ames Laboratory, Metall. & Cer. Prog., 235 Wilhelm Hall, Iowa State Univ., Ames, IA 50011 USA

Rapid solidification of an Fe-Si-B alloy was achieved by melt spinning. The effect of wheel speed, quenching atmosphere and quench media (i.e. wheel material) on the resulting microstructure was observed. Optical microscopy, scanning electron microscopy, transmission electron microscopy and x-ray diffraction of the melt-spun ribbons revealed that heterogeneous nucleation of a crystalline phase occurs on the wheel side of the ribbon, at lower wheel speeds. At higher wheel speeds, no surface crystallization on the wheel side was observed. SEM studies showed that the crystals have a hemispherical shape growing outwards from the wheel side towards free side of the ribbon. Making use of the data collected from the imaging of the melt pool geometry and the observed microstructures, nucleation and growth kinetics are discussed.

8:55 AM
Austenite Decomposition in the Bay-Forming Fe-0.30C-6.3W Steel: *R. E. Hackenberg*[1]; D. G. Granada[2]; G. J. Shiflet[3]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87544 USA; [2]Nacional de Ingenieros Electromecanica, Tegucigalpa Honduras; [3]University of Virginia, Dept. of Matls. Sci. & Eng., Charlottesville, VA 22904-4745 USA

The kinetics, morphology and elemental distributions associated with the isothermal decomposition of austenite in Fe-0.30C-6.3W were examined in the bay region of its TTT diagram using optical and electron microscopy. Similar to other bay-forming alloys, precipitation of nonlamellar alloy carbides in association with ferrite occurred at all temperatures. The overall product morphology showed an abrupt change on crossing the bay temperature, where changes in the carbide phase and morphology also took place. Furthermore, the density of carbide precipitation increased drastically at the late stages of the transformation, as a dark-etching product (also containing nonlamellar carbides) consumed the last remaining pools of austenite, consistent with the observation of a rising carbon level in the remaining austenite with time. These results highlight the non-equilibrium reaction pathway taken by this system, which is attributed to kinetic factors arising from crystallographic and interfacial structure considerations.

9:20 AM
Supersolidus Liquid Phase Sintering of Steel Powders for Full Density Applications: *E. C. Young*[1]; W. J. Poole[1]; I. V. Samarasekera[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., Vancouver, BC V6T 1Z4 Canada

Supersolidus liquid phase sintering (SLPS) is an excellent candidate for the production of full density ferrous products using traditional single press and sinter technology. In SLPS, prealloyed metal powders

are heated to a temperature between the liquidus and the solidus in order to nucleate liquid within the powder particles, with volume fraction and composition of the liquid determined by the specific sintering temperature. Due to the compositional similarity of the two phases, the liquid has excellent wetting and spreading capabilities, allowing rapid and effective densification to occur. In this study, SLPS of an Fe-C-Mo alloy was investigated with particular attention to density and porosity evolution. Differential Scanning Calorimetry (DSC) was used to identify the solidus and liquidus temperatures and characterize the phase transformation behavior of the alloy, while a high temperature experimental furnace was used to provide information on density evolution. Final densities in excess of 7.4 g/cc were achieved.

9:45 AM
Crystallographic Texture Evolution in Magnetostrictive Fe-Ga and Fe-Al Alloys during Thermomechanical Processing: *Siva Guruswamy*[1]; Nakorn Srisukhumbowornchai[1]; [1]University of Utah, Metlgcl. Eng., 135 S. 1460 E., Rm. 412, Salt Lake City, UT 84112-0114 USA

Highly magnetostrictive materials are of interest for use as actuators and sensors. In a recent work, we have shown that bcc Fe-Ga alloys have large low-field room temperature Joule magnetostriction, good mechanical strength, large ductility (similar to mild steel), and minimal hysteresis. The directionally grown Fe-27.5 at.% Ga alloy rod with a near [001] texture exhibits a saturation magnetostriction value of 271 x10-6. A relatively inexpensive thermomechanical processing approach that results in [001] textured polyctystalline alloy is investigated here. Parallel thermomechanical processing and texture evolution studies were carried out for polycrystalline Fe-15Ga and Fe-15Al alloys. Texture evolution in these alloys during a sequence of hot rolling, warm rolling reductions, and subsequent texture anneal were compared using pole figures, inverse pole figures and other data obtained from OIM-SEM examination. It is shown that texture annealing in the temperature range of 1150-1300°C can result in {hkl} <001> texture.

10:10 AM
Effect of Austenitizing Temperature on the Toughness and Strength of a 0.015C/12Cr/12Co/5Mo/4.5Ni/1.7Si Martensitic Precipitation Strengthened Stainless Steel: *Aytekin Hitit*[1]; Warren M. Garrison[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

In this work a silicon addition of 1.7 wt.% is made to a 0.015C/12Cr/12Co/5Mo/4.5Ni alloy. It was found that silicon addition results in a significant increase in the peak yield strength. However, the Charpy impact energies of the silicon alloy is found to be quite low. SEM investigations on the fractured specimens revealed the presence of undissolved molybdenum rich particles in the alloy containing silicon whereas no molybdenum rich particles were observed in the non-silicon alloy. It is believed that removing these particles by using higher solution treatment temperatures will improve not only the toughness but also the peak yield strength. The increase in yield strength is expected because of the strengthening precipitate, R-phase, is rich in molybdenum and the molybdenum rich particles lower the amount of molybdenum available for the precipitation of the strengthening precipitates. Preliminary investigations showed that after austenitizing at 1200°C for one hour, the molybdenum rich particles dissolve.

10:35 AM
Effect of Silicon Content on the Strength and Toughness of a 0.015C/12Cr/12Co/5Mo/4.5Ni/1.7Si Martensitic Stainless Steel: *Aytekin Hitit*[1]; Warren M. Garrison[1]; [1]Carnegie Mellon University, Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

Martensitic stainless steels containing sufficient amounts of molybdenum and of cobalt exhibit significant age-hardening which is attributed to the precipitation of particles of R-phase. Ferritic stainless steels containing about 5wt.% molybdenum also exhibit pronounced age-hardening if they contain 2 to 4 wt.% silicon and this age-hardening is also attributed to the precipitation of particles of R-phase. For both systems the precipitates contains large amounts of iron, chromium and molybdenum but only small amounts of silicon or cobalt. These results suggest that silicon and cobalt play similar roles in the precipitation of R-phase. The objective of this work is to determine whether or not silicon additions can enhance precipitation strengthening in low carbon martensitic stainless steels modified by cobalt additions to achieve R-phase precipitation strengthening. To investigate this possibility, the effects of an addition of 1.7 wt.% silicon on the strength and toughness of a 0.015C/12Cr/12Co/5Mo/4.5Ni alloy have been investigated. Precipitation reactions in these two compositions have been studied.

11:00 AM
The Influence of Ferrite Content on the Hardness, Galling Resistance, and Fracture Toughness of Iron-Base Hardfacing Alloys: *Brian V. Cockeram*[1]; [1]Bechtel-Bettis, ZAP 08D/MT, PO Box 79, W. Mifflin, PA 15122 USA

Iron-base weld hardfacing deposits are used to provide a wear resistant surface for a structural base material. Iron-base hardfacing alloys that are resistant to corrosion in oxygenated aqueous environments contain high levels of chromium and carbon, which results in a dendritic microstructure with a high volume fraction of interdendritic carbides that provide the needed wear resistance. The ferrite content of the dendrites depends on the nickel content and base composition of the iron-base hardfacing alloy. The amount of ferrite in the dendrites is shown to have a significant influence on the hardness and wear resistance. A galling wear test based on ASTM G98 methods is used to quantify the wear resistance of the iron-base hardfacing alloys. Fracture toughness (Kic) testing in accordance with ASTM E399 methods was used to quantify the damage tolerance of various iron-base hardfacing alloys. Fractographic and microstructure examinations were used to determine the influence of microstructure on the wear resistance and fracture toughness of the iron-base hardfacing alloys. A crack-bridging toughening model was shown to describe the influence of ferrite content on the fracture toughness. Higher ferrite content in the dendrites reduces the tendency for plastic stretching and necking of the dendrites, which results in improved wear resistance and lower fracture toughness values.

11:25 AM
Magnetostrictive Fe-Based Alloys Containing No Rare Earths: *S. Guruswamy*[1]; N. Srisukhumbowornchai[1]; [1]University of Utah, Dept. of Metlgcl. Eng., 135 S. 1420 E., Rm. 412, Salt Lake City, UT 84102-0412 USA

Low cost Fe-based magnetostrictive alloys in particular those containing no rare-earth additions are of great current interest. Magnetostrictive alloys show anisotropic linear expansion in the presence of a magnetic field and are used for actuation and sensing. Ga additions in the range of 15 at.% to 27.5 at.% Ga in Fe are shown here to improve the magnetostriction of the disordered bcc phase of Fe by as much as an order of magnitude. The applied fields for saturation magnetostriction and the hysteresis observed were small. Magnetostriction values as high as 271 x 10-6 were obtained in polycrystalline Fe-27.5 at.% Ga rods prepared using a directional growth (DG) process at a growth rate of 22.5 mm/h. This process, which is essentially a seedless vertical Bridgman technique, resulted in near [110] textured polycrystalline Fe-Ga alloys. Small substitution of 5 at.% Al for Ga in the Fe-20 at.% Ga alloy increases the magnetostriction in Fe, and the value is slightly larger than that of the Fe-20 at.% Ga alloy. Higher substitution amount of Al tends to decrease the magnetostriction. The paper also presents comparative evaluations of other binary alloys of Fe with Al, Sn, Mn, Mo, Re, Rh, B, and other elements.

11:50 AM Cancelled
Effect of Chromium and Molybdenum on the Oxidation of Fe3Al at 1000ºC: *Dong Bok Lee*

12:15 PM
Mechanical Properties of Sintered 316L Stainless Steel Produced by Metal Injection Molding: *You Hwan Lee*[1]; [1]POSTECH, Matls. Sci. & Eng., San 31, Hyoja, Namgu, Pohang, Kyungbuk 790-784 Korea

In this work, the tensile and the fatigue properties of the metal injection molded 316L stainless steel (mean particle size-about 10.8μm-), were studied with the variation of the pore amounts. It was found that increasing sintering temperature and time resulted in a decrease in the porosity along with an increase in grain size. A quantitative analysis on yield strength as functions of grain size and porosity was accomplished by considering the classical Hall-Petch equation with Gurson's model. Tensile strength and elongation were found to be largely dependent on the porosity rather than on the grain size since the fracture would be controlled by microvoid growth and coalescence mechanisms. The fatigue crack growth resistance (especially at low ΔK level) was found to increase with decreasing porosity. It was mainly attributed to the higher intrinsic resistance resulted from the delay in linking between pores ahead of the crack tip.

High Performance Metallic Materials for Cost Sensitive Applications: Titanium Alloys - II

Sponsored by: Structural Materials Division, Structural Materials Committee, Titanium Committee
Program Organizers: Edward Y. Chen, TiTech International, Inc., Pomona, CA 91768 USA; Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; F. H. (Sam) Froes, University of Idaho, Institute of Materials and Advanced Processes, Moscow, ID 83844-3026 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Tuesday AM
February 19, 2002
Room: 213
Location: Washington State Conv. & Trade Center

Session Chairs: Rodney Boyer, The Boeing Company, Seattle, WA 98124 USA; J. R. Wood, Allegheny Technologies, Monroe, NC 28111 USA

8:30 AM Invited
Innovative Titanium Aircraft Structures: *Donald L. Masingale*[1]; [1]The Boeing Company, Wichita Div., Product Definition R&D, PO Box 7730, MS K21-18, Wichita, KS 67277 USA

Titanium has long been used in hot sections and exhaust areas of aircraft engines. In the past few years application of titanium for other than hot areas of the aircraft has inspired new design concept generation, structural analysis, a great deal of new testing, resulting in new structural applications. Superplastic forming and diffusion bonding, developed in the '70 and '80's, for titanium forming and joining paved the way to applying titanium to aircraft structure as well as enhanced applications to hot sections of engines and aircraft structure. On-going effort in developing structural applications has enabled other methods of production (HIP'd Ti. investment castings, small Ti. die castings, and Laser Deposited Structure, etc.), making titanium more economically feasible and practical for designing titanium airframe structures. This paper and presentation provides a view of innovative designs for aircraft structure using creative and innovative approaches for integrating titanium material into aircraft structure.

9:00 AM
Using Superplastic Forming as a Means of Achieving Cost Benefits as Well as Enhancing Aircraft Performance: *Larry Dean Hefti*[1]; [1]The Boeing Company, BCA/Mfg. R&D, PO Box 3707, MC 5K-63, Seattle, WA 98124-2207 USA

This paper will explore the ways that the Superplastic Forming (SPF) and the Superplastic Forming and Diffusion Bonding (SPF/DB) processes are being used at Boeing Commercial Airplanes to reduce the cost of aluminum and titanium components as well as enhancing performance. These innovative manufacturing technologies are used to reduce detail part count as well as the number of fasteners required, reduce assembly time, improve dimensional tolerances and reduce the weight of components all of which leads to cost savings for the aircraft. The current SPF applications include internal stiffening details for doors, structure around the hot sections of the aircraft including the engine strut and auxiliary power unit areas, as well as close out features on wings. Future applications for SPF/DB components include door assemblies and control surfaces to minimize assembly and improve damage tolerance as well as corrosion resistance.

9:20 AM
Superplastic Behavior of Fine Grained Ti-6Al-4V: *S. P. Escobedo*[1]; S. N. Patankar[2]; David Field[1]; F. H. (Sam) Froes[2]; [1]Washington State University, Sch. of Mechl. & Matls. Eng., Pullman, WA 99164-2920 USA; [2]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

Superplastic behavior of extremely fine grained Ti-6Al-4V with an average grain size of 300 nm was studied by performing elevated temperature uniaxial tensile tests. The strain rate was varied from 10-5/s to 10-1/s to estimate the value of the strain rate sensitivity coefficient, m. Tensile testing was conducted in the temperature range of 700°C-900°C to compare the ductility (in terms of percentage elongation) of the fine grained Ti-6Al-4V specimens with a control set of conventional superplastic Ti-6Al-4V specimens with an average grain size of 3 mm. The tensile data obtained was used to investigate deformation mechanisms.

9:40 AM
Relation of Superplasticity Index and Technology Parameter of Titanium Alloy: *Zhou Tianrui*[1]; Wang Lijuan[1]; [1]Nanchang University, Inst. of Mechl. & Elect. Eng., Nanchang 330029 China

This paper investigates the superplasticity behavior of Ti-6%Al-4%V alloy. Relationship between temperature, rate of deformation, true flow stress, extensibility and strain rate sensitivity has been obtained. The present investigation has provided the theoretical and practical evidence in determining the rational technological parameters in the processing super-plastic forming of titanium alloy.

10:00 AM
Enhanced Superplastic Forming of Ti-6Al-4V: *G.* Salishchev[1]; P. Comley[2]; F. H. (Sam) Froes[3]; D. Field[4]; [1]Institute for Metals Superplasticity Problems, Khalturin Str. 39, Ufa, Bashkotostan Region 450001 Russia; [2]Boeing CA, Superplastic Forming Grp., PO Box 3707, MC 5K-63, Seattle, WA 98124 USA; [3]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [4]Washington State University, Sch. of Mechl. & Matls. Eng., 239C Dana Hall, Pullman, WA 99164-2920 USA

The superplastic forming of Ti-6Al-4V is now seeing widespread use in aerospace applications. However, the temperatures necessary for superplastic forming (SPF) 1600°-1700°F, can result in problems relating to alpha case (oxygen enrichment at the surface), excessive tooling wear and increased press maintenance. In this paper, a comparison between SPF of Ti-6Al-4V with a conventional grain size (~ 6 microns) and ultra-fine grain (~300 nanometers) will be made. A considerable decrease in SPF temperature (by as much as 300°F) or increase in strain rate, results from the decrease in grain size. The commercial advantage of this reduced grain sized material will be discussed.

10:20 AM Break

10:30 AM Invited
Dynamic Deformation of Ti and Ti-6%Al-4%V: *Marc Andre Meyers*[1]; Qing Xue[1]; V. F. Nesterenko[1]; [1]University of California-San Diego, Dept. of MAE, MC 0411, LaJolla, CA 92093 USA

The evolution of multiple adiabatic shear bands was investigated in commercially pure titanium and Ti-6Al-4V alloy through the radial collapse of a thick-walled cylinder under high-strain-rate deformation (~104 s-1). The shear bands nucleate at the internal boundary of the specimens and construct a periodical distribution at an early stage. The evolution of shear-band pattern during the deformation process reveals a self-organization character. The differences of mechanical response between the two alloys are responsible for significant differences in the evolution of the shear band patterns. The number of shear bands initiated in Ti (spacing of 0.18 mm) is considerably larger than in Ti-6% Al-4%V (spacing of 0.53 mm); on the other hand, the propagation velocity of the bands in Ti-6%Al-4%V (~556 m/s) is approximately three times higher than in Ti (~153 m/s). The experimental shear-band spacings are compared with theoretical predictions that use the perturbation analysis and momentum diffusion; the shortcomings of the latter are discussed. A new model is proposed for the initiation and propagation that incorporates some of the earlier ideas and expands them to a two-dimensional configuration. The initiation is treated as a probabilistic process with a Weibull dependence on strain; superimposed on this, a shielding factor is introduced to deal with the disactivation of embryos.

11:00 AM
Microstructure Evolution in Hydrogenated Ti-6Al-4V: *Javaid I. Qazi*[1]; J. Rahim[1]; O. N. Senkov[2]; S. N. Patankar[1]; F. H. (Sam) Froes[1]; [1]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA

Ti-6Al-4V samples were hydrogenated to 10, 20 and 30at.% hydrogen levels by holding in a hydrogen atmosphere. Microstructure analysis of the hydrogenated samples after different heat-treatments was carried out using optical microscopy and transmission electron microscopy techniques. The microstructure of hydrogenated samples, heated above the beta transus temperature and step cooled to lower temperature and annealed for various lengths of times was studied. Microstructure of aged hydrogenated samples was also studied by quenching the hydrogenated samples from above the beta transus temperature and ageing at different temperatures for various lengths of time. Vacuum annealing was carried out on the heat-treated samples to dehydrogenate them and the resulted microstructure was compared with the starting and the heat-treated microstructures.

11:20 AM
Martensite Decomposition in Ti-6Al-4V-xH Alloys: *Javaid I. Qazi*[1]; O. N. Senkov[2]; S. N. Patankar[1]; F. H. (Sam) Froes[1]; [1]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [2]UES, Inc., 4401 Dayton-Xenia Rd., Dayton, OH 45432-1894 USA

Ti-6Al-4V samples were hydrogenated to 10, 20 and 30at.% hydrogen levels by holding in a hydrogen atmosphere. The kinetics of the martensite decomposition in the hydrogenated samples were studied by aging the β solution treated and quenched samples at different temperatures for varying lengths of time. Optical microscopy, transmission electron microscopy and microhardness testing techniques were utilized to study the phase transformations. The nose temperature for the start of the martensite decomposition curve decreased from 750°C to 625°C when the hydrogen concentration was increased from 10 to 30at.%. The nose time for the start of the martensite decomposition curve was found to be independent of the hydrogen concentration.

11:40 AM
Titanium Castings for Cost-Sensitive Applications: *Edward Y. Chen*[1]; Scott D. Sitzman[1]; Raymond J. Tisler[1]; Douglas R. Bice[1]; [1]TiTech International, Inc., 4000 W. Valley Blvd., Pomona, CA 91769 USA

Titanium castings are successfully being used as cost-effective alternatives to forging and wrought products for increasingly cost-sensitive applications such as military aircraft airframes. In some instances, castings have been produced for half the cost of a comparable forged-machined part. For much of the last 20 years, investment casting has been the preferred manufacturing route for sophisticated titanium castings due to advantages such as high dimensional tolerance capability. Lesser-known and less expensive casting methods such as by rammed graphite molding are also available and can offer additional competitiveness as market forces press for further cost savings. This presentation examines the two primary titanium castings approaches in use today, investment and rammed graphite, to gauge affordability, process capability, and microstructure-properties against the requirements set forth for forged and wrought parts. Challenges to further cost reduction and more widespread titanium castings use in cost-sensitive applications will be discussed.

Hume-Rothery Award Symposium: CALPHAD and Alloy Thermodynamics: Materials Stability and Reaction

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Robert D. Shull, NIST, Magnetic Materials, Boyds, MD 20841-9015 USA

Tuesday AM — Room: 204
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Antonios Gonis, Lawrence Livermore National Laboratory, C.&M.S. Direct. (L-353), PO Box 808, Livermore, CA 94551 USA; Catherine Colinet, LTPCM-ENSEEG, Domaine Universitaire, Saint Martin d'Hères 38402 France

8:30 AM Invited
Structural Instabilities in High Temperature Phases: *R. E. Watson*[1]; M. Weinert[1]; G. Schneider[1]; [1]Brookhaven National Lab, Phys. Dept., Upton, NY 11973 USA

The low-temperature structural instabilities of high-temperature phases have been discussed previously.[1,2] These considerations will be extended to consideration of the smart materials NiTi and $GaMnNi_2$, as well as classic Martensitic phases such as AuCd and AuTi. These matters are of some concern to the CALPHAD community, whose constructs assume such phases to be stable. Work supported by the Division of Materials Sciences, US Dept. of Energy under Contract No DE-AC02-98CH10886. 1. P. J. Craievich, M. Weinert, J. M. Sanchez, and R. E. Watson, Phys. Rev. Lett. 72, 3076 (1994). 2. P. J. Craievich, J. M. Sanchez, R. E. Watson, and M. Weinert, Phys. Rev. B 55, 787 (1997).

9:00 AM Invited
Calphad and Ab Initio Approaches to Lattice Stabilities: *Goran Grimvall*[1]; [1]Royal Institute of Technology, Theoretl. Phys., SE-100 44, Stockholm Sweden

The most important quantity in Calphad calculations is the enthalpy difference(lattice stability) between stable and metastable phases of pure solids. The lattice stability derived in Calphad work is sometimes very different from the result in quantum mechanical (ab initio) total energy calculations, for instance in fcc tungsten. Ab initio calculations later showed that the fcc tungsten lattice is dynamically (i.e. mechanically) unstable, rather than metastable. Then the vibrational entropy S is undefined. Hence there is no Gibbs energy G = H - TS for the unstable phase. The paper gives a survey of instabilities as predicted in ab initio calculations, and an account of how the Gibbs energy is described when an instability is approached (e.g. through varying alloy composition). Finally it is argued that the ab initio and Calphad approaches can be reconciled, with no essential changes in the Calphad method.

9:30 AM Invited
The Prediction of Extraordinarily Stable Intermetallics of the Platinum Group Metals: *Leo Brewer*[1]; Karen Krushwitz[1]; [1]University of California-Berkeley, Dept. of Chem., MC 1460, Berkeley, CA 94720 USA

The prediction of platinum group metallic alloy compositions with desired properties for technological development using the Brewer-Engel Model is proposed to characterize petroleum products and catalysts imperative not only to maximize resources, but also with possible applications to render mixed oxide (MOX) fuels clean for disposal. Galvanic cell measurements, X-ray diffraction and wavelength dispersive spectroscopy will be used to assess a wide range of platinum group metal systems. One can prepare alloys as feasible catalysts to determine effect of electron transfer upon the thermodynamic stability of catalytic behaviour using cell measurements. This work is considerably advanced by previous work on lanthanide/actinide phase diagrams, the power of the Brewer-Engel Model, as well as the recent work of colleagues who have provided thermodynamic data for U-Sn-Pd and U, Pu-oxide ternary systems. Prediction of designer metallic alloys compositions using the Brewer-Engel Model will require several steps. Step one: predict bonding energies for generalized Lewis acid-base interactions including both covalent and ionic bonds to evaluate thermodynamic properties, the number of s and p electrons which fix structure, and the relative sizes of atoms. Step two: predict properties of phases such as density, electronic properties and hardness. Final step: extension to multicomponent systems. There are a daunting variety of catalyst choice to consider: multicomponent systems identification makes it possible to limit number of phases that would have the desired properties for a given technological development to a small fraction of multicomponent alloys.

10:00 AM Break

10:30 AM Invited
The Application of Calphad to Phase Transformations in Complex Alloys: *John A. Ågren*[1]; [1]Royal Institute of Technology, Matls. Sci. & Eng., SE-100 44, Stockholm Sweden

The extension of the Calphad technique to cover not only equilibrium thermodynamics but also phase transformations during heat treatment, processing and usage of alloys is reviewed. Models based on diffusion kinetics and local equilibrium are powerful tools when predicting the rate of various phenomena, e.g. carbide dissolution during austenitizing of steels, precipitation of sigma-phase in stainless steels etc. For those alloy classes where good thermodynamic data now are available, e.g. steels, Al-alloys and Ni-base alloys, very accurate predictions of non-equilibrium phenomena may be made provided that also kinetic data will be made available. There is thus a call for assessment of diffusion data using essentially the same approach as in traditional Calphad pioneered by Kaufman. The presentation will give some of the background, some recent examples and discuss the further development to cover also interfacial reactions and deviation from local equilibrium.

11:00 AM Invited
Computational Thermodynamics and Simulation of Phase Transformations: *Gerhard Inden*[1]; [1]Max-Planck-Institut für Eisenforschung, Physl. Metall., Max-Planck-Str. 1, Düsseldorf D-40237 Germany

About 35 years ago computational thermodynamics started with the pioneering work of Larry Kaufman, known today as the CALPHAD approach. The intention was to use numerical techniques for treating phase equilibria in multicomponent systems. The fundamental principles were of course known. Nonetheless, this approach initiated new concepts, e.g. phase stabilities. Meanwhile, a variety of databases and software has been developed covering large parts of the wide field of materials. With this background it is natural to move from equilibrium towards transformations. The thermodynamic properties taken from CALPHAD allow to define the driving forces for reactions and to take the thermodynamic coupling between kinetic parameters, e.g. diffusivities, into account. Again, the fundamentals are well-known, but the complexity of the multicomponent systems, of the associated kinetic parameters and of thermal treatments represent an almost

insurmountable barrier for predictions. It is only with the aid of simulations that reliable predictions can be expected. A variety of examples out of the field of steels will be presented illustrating the capability, but also the limitations of presently available software like DICTRA. Steels are complex systems with a large number of competing phases and elements with differences in their mobilities by orders of magnitude.

11:30 AM Invited
Prediction of Interfacial Reactions using Thermodynamic Calculation and Diffusion Simulation: *Byeong-Joo Lee*[1]; [1]KRISS, Matls. Evaluation Ctr., Yusong PO Box 102, Taejon 305-600 Korea

An interfacial reaction between phase layers may involve formation and growth of new phase layers at the interface as well as constitutional changes in initial layers. The interfacial reactions give great effect on the joint properties of the materials. Therefore, it is of practical and academic importance to understand and predict interfacial reactions between various types of materials: metals, ceramics and semiconductors. Recently, the present author has developed a model for prediction of interfacial reactions. The model has been successfully applied to predict interfacial reaction products in metal/solder, metal/ceramics and metal/Si reactions. The model is based on thermodynamic calculation, diffusion simulation and qualitative consideration of nucleation kinetics. In the present paper, the model will be outlined together with examples for application to Cu/solder, Ti/Al2O3 and Metal/Si interfacial reactions.

Imaging of Dynamic Processes - I

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizer: Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA

Tuesday AM
February 19, 2002
Room: 310
Location: Washington State Conv. & Trade Center

Session Chair: Iver E. Anderson, Iowa State University, Ames Lab., Metall. & Cer., 222 Metals Dvlp., Ames, IA 50011 USA

8:30 AM Invited
Thermal Imaging of Solidification: *William Hofmeister*[1]; [1]Vanderbilt University, Interdisciplinary Matls. Sci./Cheml. Eng., PO Box 1604, Sta. B, Nashville, TN 37235 USA

Thermal imaging has been useful in the determination of solidification kinetics from undercooled melts and in process control of solidification in direct metal deposition. In undercooled melts solidification is accompanied by recalescence, the release of the latent heat of fusion. This release of sensible heat raises the temperature of the solid such that the solidification front can be tracked on the surface of a levitated drop. At Vanderbilt we have used various high-speed and ultra high-speed thermal imaging techniques to determine the relationship between bulk undercooling and solidification velocity in pure metallic materials and alloys. These imaging techniques will be described, and some comments on the data and predictability of solidification kinetics at high undercooling will be elucidated. In the area of direct metal deposition, high-speed thermal imaging has been used to study cooling rates in solidification. In the LENSTM (Laser Engineered Net Shaping) process developed at Sandia National Laboratory, the information from thermal imaging is used to provide feedback control for the process, insuring that the desired cooling rates are produced regardless of the part geometry. This work will also be discussed. This work was sponsored by NASA Office of Microgravity Science and Applications and Sandia National Laboratory. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the US Department of Energy under contract number DE-AC04-94AL85000.

9:00 AM
Visualization of Primary Austenite and Primary Ferrite Solidification Modes in Fe-Ni-Cr Gas Tungsten Arc Welds: *Aaron C. Hall*[1]; Charles V. Robino[1]; John Brooks[2]; Mark Reece[1]; Danny O. MacCallum[1]; [1]Sandia National Laboratories, 1833, PO Box 5800, MS 0889, Albuquerque, NM 87185-0889 USA; [2]Sandia National Laboratories, 8724, PO Box 969, Livermore, CA USA

A technique for imaging the solid-liquid interface in Gas Tungsten Arc (GTA) welds at high-speed and high-magnification has been developed. At high magnification, the dendritic structure of the solid-liquid interface can be clearly seen. Computer image analysis techniques have been developed that allow solid-liquid interface velocity and secondary dendrite arm spacings to be extracted from the video images. This technique has been used to image two Fe-Ni-Cr alloys. One is a known austenite solidifier; the other is a known ferrite solidifier. The imaging and data extraction techniques will be described. High-speed, high-magnification videos of each Fe-Ni-Cr alloy will be shown and compared. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

9:30 AM Invited
Optical Characterization of Front Dynamics in Directional Solidification of Transparent Alloys in a Cylinder: *Haik Jamgotchian*[1]; Nathalie Bergeon[1]; Dominique Benielli[1]; Philippe Voge[1]; Bernard Billia[1]; [1]Université d'Aix-Marseille III, L2MP, UMR CNRS 6137, Faculté de St Jérôme, Case 151, Marseille, Cedex 20 13397 France

Materials properties strongly depend on the microstructure left in the solid during processing. Microstructure formation and dynamics in directional solidification remain largely open. Up to now important progress have been realized on transparent alloys in thin samples, where convection was most often negligible. However, important points should still be definitely clarified, such as the role of convection on the formation and evolution of microstructure and the problem of pattern selection. Therefore, experiments on cylindrical samples are carried out in SOLDIR and SOLDIR1 devices (laboratory models dedicated to directional solidification of model transparent alloys in a cylinder in the future DECLIC facility onboard the International Space Station). The solid-liquid interface is characterized by two complementary optical techniques, through the whole length of the crucible. The direct observation of the transmitted light provides bright field images of the solid-liquid interface. A Mach-Zehnder interferometer set-up gives the shape and the position of the interface. In addition, SOLDIR1 device possesses side observation in transmission. In situ and real-time images are recorded on videotape. For the first time, coupling of convection and solid-liquid interface morphology is analyzed. Experiments are carried out on succinonitrile - 0.2 wt% acetone alloy and on pure succinonitrile with various lengths of solid seed that induce different initial conditions. Dynamics of convection during solidification initial transient are analyzed through the time evolution of the front shape. The present experiments also reveal a strong dependence of propagation of morphological instability and macroscopic front deformation on fluid flow in liquid phase.

10:00 AM Break

10:15 AM
Three Dimensional Microstructural Evolution in Succinonitrile: *Mark A. Palmer*[1]; Martin E. Glicksman[2]; Krishna Rajan[2]; [1]Kettering University, IMEB Dept., 1700 W. Third Ave., Flint, MI 48504-4898 USA; [2]Rensselaer Polytechnic Institute, Matls. Sci. & Eng., CII 9111, 110 Eight St., Troy, NY 12180-3590 USA

The apparent annihilation of four and five sided grains in succinonitrile thin films contradicts the predictions that grains disappear through a sequence of neighbor switching and three dimensional grain annihilation. This observation can be explained as an effect of the third dimension, that is the finite thickness of the film. However these three dimensional processes can be modeled as a series of two dimensional events occurring at either surface or throughout the thickness of the film. Visual examination confirms these predictions. This work was funded by the Jeffres Memorial Trust.

10:45 AM
Studying Changes in Surface Topography by White Light Interferometry: *Borge Holme*[1]; [1]SINTEF Materials Technology, Casting & Metal Forming, Oslo, PO Box 124 Blindern, N-0314 Oslo Norway

White Light Interferometry gives fast and accurate measurements of surface topography. Within 10 seconds one obtains a topographic image with micrometer resolution laterally and nanometer resolution vertically. At SINTEF Materials Technology we have extended the capabilities of our WYKO NT-2000 White Light Interferometer to include studies of changes in surfaces. We make repeated images of a surface that undergoes a change in topography, e.g. by etching, polishing, melting, drying, corrosion or mechanical strain. The sequence of images is made into a video clip, which visualizes the topographic changes in an intuitive manner. Some processes, like drying of paint, can be done in-situ with imaging in real time. Most chemical processes, like etching and corrosion, have to be done in a stepwise manner where the sample is removed and etched in the lab. We have therefore constructed a sample holder for accurate repositioning of the sample.

Special software was written to analyze and visualize the data. Combining th is technique with surface analytical instruments like SEM will make it an even more powerful scientific tool.

11:15 AM
Investigation of Bubble Nucleation Site Density during Quenching Heat Treatment Process using Video Imaging: *M. Maniruzzaman*[1]; SH Ma[1]; R. D. Sisson[1]; [1]Center for Heat Treating Excellence, Matls. Sci. & Eng., Worcester Polytechnic Inst., Worcester, MA 01609 USA

The cooling rate during quenching of steel parts in liquid medium largely depends on the dynamics of the bubble formed on the heated surface and the convection of the liquid medium from the hot surface. In the nucleate boiling regime of the quenching process in the liquid medium, bubble nucleation site density plays a vital role in transferring heat from the heated surface. In this study, the surface-liquid interactions during the nucleate boiling regime of the quenching process and its effect on the nucleation site density and thereby the cooling rate is examined. The nucleation site density for various surface-liquid combinations was measured by using progressive scan 3-CCD digital video camera. A correlation in terms of wall temperature, the Prandtl number, a surface-liquid interaction parameter and dimensionless roughness parameter is proposed and compared with the experimental observations.

International Symposium on Science and Technology of Interfaces in Honor of Dr. Bhakta Rath: Mechanical Behavior

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Physical Metallurgy Committee, Superconducting Materials Committee, Jt. Mechanical Behavior of Materials, Titanium Committee
Program Organizers: Sreeramamurthy Ankem, University of Maryland, Department of Material & Nuclear Engineering, College Park, MD 20742-2115 USA; I. Ovidko, Russian Academy of Sciences, Institute of Problems of Mechanical Engineering, Laboratory for Theory of Defects in Materials, St. Petersburg 199178 Russia; Chandra Pande, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5000 USA; S. Ranganathan, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India

Tuesday AM Room: 617
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: James C.M. Li, University of Rochester, Dept. of Mechl. Eng., 500 Wilson Blvd., 233 Hopeman/River Campus Sta., Rochester, NY 14627 USA; Sreeramamurthy Ankem, University of Maryland, Dept. of Matls. & Nucl. Eng., College Park, MD 20742-2115 USA

8:30 AM Invited
Microstructures and Mechanical Properties of Semi-Solid Materials: Hee-Soo Kim[1]; Ian Stone[1]; *Brian Cantor*[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford OX1 3PH UK

This paper describes the effect of heat treatment on grain growth in a wide range of semi-solid Al and Ni alloys, as well as the related effects on mechanical properties. The paper concentrates on the recently demonstrated unusual effects which are found at high solid fractions with faster grain growth at higher temperatures unlike the low solid fraction regime, and with compressive strength varying dramatically with grain size and anisotropy.

8:55 AM Invited
The Role of Interfaces on Mechanical Behavior of Titanium Alloys: *Sreeramamurthy Ankem*[1]; [1]University of Maryland-College Park, Dept. of Matls. & Nucl. Eng., College Park, MD 20742-2115 USA

Interfaces such as grain boundaries in single phase titanium alloys and two-phase interphase boundaries in two-phase titanium alloys play a significant role on mechanical behavior of titanium alloys. For example, at low temperatures, a large grain size in single phase alpha and beta titanium alloys results in time dependent twinning, which is primarily responsible for ambient temperature creep resistance. In regard to alpha-beta two-phase titanium alloys, a number of factors such as alloying elements and thickness of the alpa platelets determine whether slip is transferred or not across the interfaces. Ambient temperature creep resistance was found to be small in those alloys where slip is easily transferred across the alpha-beta interfaces. These and other effects of interfaces on mechanical properties will be reviewed and optimal microstructures will be identified. This work is being supported by the Office of Naval Research under Grant No. N0001496101819.

9:20 AM
Reevaluation of Enviromental Interactions at the Crack Tip-Metal Interfaces: *Kuntimaddi (Sada) Sadananda*[1]; Ronald L. Holtz[1]; Asuri K. Vasudevan[3]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6323, 4500 Overlook Ave., Washington, DC 20375 USA; [3]Office of Naval Research, 800 N. Quincy St., Arlington, VA 22217 USA

The authors have recently developed a Unified Approach to Fatigue Crack Growth that involves consideration of two load parameters that include Kmax in addition to the conventional *K parameter that describes the stress intensity amplitude. Of the two, Kmax appears to be a more fundamental requirement for all fracture phenomena, while *K arises as a perturbation due to the irreversibility associated with plasticity during unloading. Correspondingly there are two thresholds one in terms of each parameter that need to be fulfilled simultaneously for a fatigue crack to grow. Environmental contributions arise directly through Kmax and indirectly through *K. We present a new perspective of the role of environmental interactions at the crack tip-metal interfaces and how they affect these two driving forces and thus fatigue crack growth rates.

9:40 AM
Hot Ductility in Titanium Alloys-A Review: *M. Ashraf Imam*[1]; Brian K. Damkroger[2]; Glen R. Edwards[3]; [1]Naval Research Laboratory, Matls. Sci. & Tech. Div., Code 6320, 4555 Overlook Ave. S.W., Washington, DC 20375-5343 USA; [2]Sandia National Laboratories, Stockpile Sys. Ctr., Albuquerque, NM 87185-5800 USA; [3]Colorado School of Mines, Ctr. for Welding & Joining Rsrch., Golden, CO 80401 USA

Two phase titanium alloy systems suffer from an abrupt drop in ductility on cooling from beta phase and is sensitive to cooling rate. This loss of ductility is manifested by easy decohesion of polycrystalline aggregates along the grain boundaries of the high temperature beta phase. If the alloy is in a state of tensile stress at the aforementioned temperatures, cracks initiate at the grain boundaries and propagate readily through the alloy, leading to premature failure. This phenomenon requires care in processing of titanium alloys during thermomechanical fabrication and welding. Several mechanisms have been proposed to explain high temperature crack nucleation and growth along the boundaries. A critical review of the phenomenon and possible mechanisms responsible for the observed behavior will be discussed.

10:00 AM Invited
A New Mechanism for Superplasticity: *James C.M. Li*[1]; [1]University of Rochester, Matls. Sci. Prog., Dept. of Mechl. Eng., Rochester, NY 14627 USA

Superplasticity has been discovered for a long time but its mechanism is still not clear. Grain boundary shearing is the usually accepted mechanism except that when the extent of shear is measured carefully along the boundaries, it contributes only about a third of the total strain. The other 2/3 must come from the deformation of the grains. Yet the activation energy is usually that of the grain boundary or interfacial diffusion. The new mechanism treats the interfacial fluid layer as the source of plasticity. Not only it can be sheared to produce shear strain but also squeezed to produce compressive strain or negatively squeezed to produce tensile strain. The latter strains cannot be measured by grain boundary shearing. In fact the shear flow and the compressive or extensional flow are consecutive processes with the slow one controlling. All the flows continue because the interfacial fluids are not exhaustible. Work supported by NSF through DMR9623808 monitored by Dr. Bruce MacDonald.

10:25 AM Invited
Crystallization Behavior and Mechanical Properties of Melt-Spun Al-Ni-Mm Alloys: *S. J. Hong*[1]; H. S. Kim[1]; C. Suryanarayana[2]; B. S. Chun[1]; [1]Chungnam National University, Rapidly Solidified Matls. Rsrch. Ctr., Taedok Science Town, Taejon 305-764 Korea; [2]University of Central Florida, Dept. of Mechl., Matls. & Aeros. Eng., Orlando, FL 32816-2450 USA

There has been renewed interest in recent years on the crystallization behavior of rapidly solidified Al-Ni-Misch Metal alloys. In the present investigation, we have studied the microstructure and mechanical properties of Al-Ni-Mm alloy ribbons both in the as melt-

spun and heat-treated conditions using DSC, XRD, TEM and microVickers hardness methods. Primary crystallization of the alloy results in a mixed fine structure consisting of nanocrystalline Al particles embedded in an amorphous matrix amorphous nanocomposites). The hardness changes with the composition and microstructure after heat treatment. To quantitatively explain the hardening mechanism in the nanocomposites, a rule-of-mixture model based on the volume fraction of the amorphous matrix and Al particles is proposed. The nano-sized particles are treated as perfect materials and the matrix is treated as amorphous material, in which the solute concentration increases as the volume fraction of the Al particles increases. The results obtained using the rule of mixtures using the iso-stress model are in good agreement with the experimental ones.

10:50 AM
Modeling of Die-Workpiece Interface during Hot Forging: *Kalyan Kannan*[1]; Murali Pandheeradi[1]; Suhas Vaze[1]; Steven R. Schmid[2]; [1]Concurrent Technologies Corporation, Johnstown, PA 15904 USA; [2]University of Notre Dame, Dept. of Mechl. & Aeros. Eng., Notre Dame, IN 46556 USA

Hot forging operations involve a variety of die-workpiece interface phenomena, such as lubricant stretching and thinning, surface roughening and/or asperity flattening and heat transfer. Localized lubricant thinning or surface roughening (which are strain-induced) can lead to regions of poor lubrication, where there is contact between the die and workpiece asperities. This can result in local variations of the friction and heat transfer between the die and workpiece, and in turn, affect the metal flow pattern, defect formation and die wear. Most hot forging models assume a constant value of the friction factor (m) and heat transfer coefficient (h), which limits their accuracy. This presentation describes the development of an advanced model, incorporating the above phenomena and allowing for an evolving m and h with workpiece strain. This work includes experimental activities on characterizing friction and heat transfer for various material and lubricant systems, characterization of surface roughening and lubricant thinning, development of a module accounting for variations in m and h with strain, and integration of the module with a commercial FEA code. Results of the model applied to ring compression tests and sample forgings will be presented, highlighting the improvements in modeling of these forging processes. Acknowledgments: This work was conducted by the National Center for Excellence in Metalworking Technology, operated by Concurrent Technologies Corporation under contract No. N00014-00-C-0544 to the US Navy as part of the US Navy Manufacturing Technology Program. It was undertaken as a part of the 'Forging Suppliers Initiative' project, a joint US Air Force/US Navy initiative focussed on reducing the cost of forged aircraft engine components. The authors acknowledge the technical guidance of Dave Furrer and Joe Lemsky of the Ladish Company, Cudahy, WI and Tim Howson of PCC, North Grafton, MA.

11:10 AM
Magnetic Pulse Welding and Forming of Tubular Components: *Victor Shribman*[1]; [1]Pulsar, Ltd., 4 Faran St., Yavne 81103 Israel

The use of a pulsed magnetic field and its advantage over conventional methods in the metalworking industry has been known for some time. Magnetic pulse welding (MPW) is in the group of pressure solid state welding processes together with explosive welding, to which it is closely analogous. Both these processes utilize a high speed impact of two work pieces placed either in parallel or at a small angle one to the other and with a small initial displacement allowing a terminal velocity and a suitable angle to be achieved at impact. The basic principle of the magnetic pulse system is that an extremely high current is discharged through a coil, creating an eddy current in the conductive workpiece. Repulsion between the two magnetic forces (created through the coil and the eddy current in the surface of the workpiece) creates pressure and accelerates the workpiece into a new configuration with a force proportional to the square of the discharge current. As a result the workpiece moves away from the coil at very high speed, pushing the metal well beyond its yield strength and into its plastic region. If the conditions are correct i.e. plate velocity, collision point angle and collision point velocity, jetting of the surface layers at the collision point occurs resulting in a weld. This exciting new technology will be presented along with applications for industry including welding of tubes of various similar and dissimilar combinations. These, among others, will include results for aluminum to low alloy steel, aluminum and copper, copper to copper and brass, and steel to steel. Some forming applications will also be presented.

Lead-Free Solders and Materials Issues in Microelectronic Packaging: Microstructural Characterization and Evolution

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Electronic Materials Committee, Electronic Packaging and Interconnection Materials Committee
Program Organizers: Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; Darrel R. Frear, Motorola, Tempe, AZ 85284 USA; Sung-Ho Jin, Lucent Technologies, Bell Laboratories, Murray Hill, NJ 07974 USA; Sung Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Michael J. Pfeifer, Motorola, Northbrook, IL 60062 USA; Martin Weiser, Honeywell Electronics Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Tuesday AM Room: 612
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Darrel R. Frear, Motorola, SPS, 2100 E. Elliot Rd., Tempe, AZ 85284 USA; Paul T. Vianco, Sandia National Laboratories, MS 1411, PO Box 5800, Albuquerque, NM 87185 USA

8:30 AM Invited
Microstructural Modifications and Properties of Sn-Ag-Cu Solder Joints Induced by Alloying: *I. E. Anderson*[1]; J. C. Foley[1]; B. A. Cook[1]; J. L. Harringa[1]; R. L. Terpstra[1]; J. Anderegg[1]; [1]Ames Laboratory, Metall. & Cer., 222 Metals Development, Ames, IA 50011 USA

A family of near-eutectic Sn-Ag-Cu alloys has emerged from a patented Sn-4.7Ag-1.7Cu (wt.%) eutectic with high potential for replacement of Sn-37Pb as a general use solder. With the benefits of a melting range increase of less than about 2°C above the eutectic temperature of 217°C and increased strength from both Ag₃Sn and Cu₆Sn₅ phases, alloy development has focussed on Sn-(3.5 to 4.0)Ag-(0.5 to 1.0)Cu, with Sn-3.8Ag-0.7Cu and Sn-4.0Ag-0.5Cu as commercial examples. This study consists of a critical comparison of alloys within the Ag and Cu content ranges in terms of melting and solidification behavior and solder joint microstructure and mechanical properties. Also included were 4th element additions, Co, Fe, and Bi, to Sn-Ag-Cu to enhance solidification refinement and thermal stability of the joint microstructure. In addition, the 4th element alloying mechanisms and correlations with mechanical properties will be reviewed. Support received from USDOE-BES, Materials Science Division (contract no.W-7405-Eng-82).

8:55 AM
Evolution of Microstructure of Pb Free Solder Joints: *Eric J. Cotts*[1]; [1]SUNY Binghamton, Matls. Sci./Phys., PO Box 6016, Science 2, Binghamton, NY 13902-6016 USA

The advent of Pb free solders has had a profound effect upon the metallurgy of solder interconnects. The properties of solder interconnects are determined by the nature of the solder, the contact pad metallization, and the phases which form in the bulk of the solder and at the solder/metallization interfaces during reflow and subsequent aging. As metallizations and solders change, new metallurgical phenomena occur, affecting the properties and reliability of the interconnects and the package as well. The Pb in solders has been replaced with constituents (e.g. Cu or Ag) which diffuse rapidly in Sn and can react with metallizations. We report on studies of the evolution of the microstructure Pb free solder (including SnAgCu solder) joints with Ni and Cu metallizations. We focus on the variation of solder joint microstructure with relatively small variations in reflow temperatures and times, in particular those variations which have been shown to cause changes in solder joint reliability.

9:15 AM
The Microstructure Characterization of Ultrasmall Eutectic Bi-Sn Solder Bumps on Au/Cu/Cr and Au/Ni/Ti UBMs: *Un-Byoung Kang*[1]; *Young-Ho Kim*[1]; [1]Hanyang University, Div. of Matls. Sci. & Eng., 17 Haengdang-dong, Seongdong-ku, Seoul 133-791 Korea

Flip chip technology is increasingly used due to its high packaging density and good electrical performance. Recently, the pad size and the pitches of chips get smaller and finer because of further miniaturization and integration of electronic components. Bi-Sn solder has raised great attention for low temperature solder alloy. This paper presents the evolution of microstructure in ultrasmall eutectic Bi-Sn

solder bumps on Au/Cu/Cr and Au/Ni/Ti UBMs. Ultrasmall 58wt%Bi-42wt%Sn solder bumps with the diameter of 25–50° were fabricated using lift-off method and reflowed using RTA system. The Bi phase segregated in the solder surface and two phase-like structure was observed in the Bi-Sn solder bumps formed on Au/Ni/Ti UBM. As the cooling rate increased, the solder surface became smooth and the lamellar structure appeared. For Bi-Sn solder bumps on Au/Cu/Cr UBM, the solder is composed of the large Bi phase and the lamellae of Bi-Sn. Increased cooling rate refined the lamellar structure.

9:35 AM

Reliability of Flip Chip Solder Joints with Sn-Based Pb-Free Solders under Thermal Cycling Test: *Hun Han*[1]; J. Y. Kim[1]; Jin Yu[1]; [1]KAIST, Matl. Sci. & Eng., 373-1 Kusong-dong Yusong-gu, Taejon 305-701 Korea

As microelectronic devices get smaller and I/O densities increase, various chip scale packages with the flip chip technology becomes more important. The flip chip solder joint is the area of stress concentration during the thermal cycle and susceptible to crack initiation and growth. The reliability problem tends to become more complex and severe as the solder pitch and ball size become smaller. This is particularly so because mechanical property of solder depend on the microstructure which varies with the solder ball size. In the present work, using typical Pb-free candidate alloy: Sn-3.5Ag, Sn-3.5Ag-0.7Cu, Sn-0.7Cu, we prepared flip chip packages with varying solder pitch (800, 400, 200, 100μm) and ball size (300, 200, 100, 50μm), and conducted thermal cycling tests. Investigated under-bump-metallurgy (UBM) structure included Ti/Ni, Ti/Cu and Ti/Ni/Cu, etc. Results of the test were examined from the perspectives of the solder microstructure, creep-fatigue interaction, intermetallic formation, and UBM structures.

9:55 AM Break

10:10 AM Invited

The Microstructure and Creep Properties of Lead-Free Solder Joints: *Ho Geon Song*[1]; John W. Morris[1]; Fay Hua[2]; [1]University of California at Berkeley/Lawrence Berkeley National Laboratory, Dept. of Matls. Sci. & Eng., MS 66-200, One Cyclotron Rd., Berkeley, CA 94720 USA; [2]Intel Corporation, Santa Clara, CA 95054 USA

Failure induced by thermal fatigue is one of the most important threats to the integrity of solder joints. Since in many cases the strain cycle is predominantly in shear and the joint is cycled at a high homologous temperature, the major deformation mode that cause fatigue is creep in shear. Therefore it is necessary to study of lead-free solder joints under shear creep conditions in order to use them reliably in microelectronic applications. This work presents creep behaviors and microstructural influences of several lead-free solder joints, including Sn-0.7Cu, Sn-3.5Sn, Sn-10In-3.1Ag, and Sn-3Ag-0.5Cu. Constant load tests at 60, 95, 130°C were performed on single-shear specimens with bare Cu and Cu/electroless Ni/immersion Au substrates. The microstructural change under creep conditions will be discussed as the microstructural responses of each of these alloys to creep deformation. Additionally, the effect of cooling rate during solidification and substrate metallization on the creep behaviors of these alloys will be reported.

10:35 AM

Interfacial Microstructure of Pb-Free and Pb-Sn Solder Ball in BGA Package: Chin-Su Chi[1]; *Ker-Chang K. Hsieh*[1]; C. L. Mark Chung[2]; [1]National Sun Yat-sen University, Inst. of Matls. Sci. & Eng., Kaohsiung Taiwan; [2]ChipMos Technologies, Inc., Assy. Dev. Div., No. 5, Nan-Ko 7th Rd., Science-Based Industrial Park, Tainan Taiwan

BGA samples were aged at 155°C up to 45 days. The formation and the growth of the intermetallic phases at the solder joints were investigated. The alloy contents of solder ball included Sn-3.5Ag-0.7Cu, Sn-1.0Ag-0.7Cu and Sn-37Pb. The solder ball pads were with Au/Ni surface finish on copper substrate. Microstructure analysis was carried out by electron microprobe. The distributions of Au and Cu during aging were carefully examined on solder joints and solder balls. The intermetallic phase formation sequences can be interpreted by diffusion path and ternary phase diagram.

10:55 AM

Effect of Bi Content on the Microstructure and Shear Strength of Ball-Grid-Array Sn-Ag-Bi/Cu Solder Joints: Joo-Youl Huh[1]; Sang-Uk Han[1]; [1]Korea University, Div. of Matls. Sci. & Eng., 5-1, Anam-Dong, Seoul, Sungbuk-Gu 136-701 Korea

We studied the effects of Bi addition (2, 5, 8, 12 wt.%) in eutectic Sn-3.5Ag solder on the microstructural evolution during soldering and aging, and on the shear strength of ball-grid-array (BGA) solder/Cu joints. The ball shear strength of Sn-Ag-Bi solder joints is closely related to the morphology of the intermetallic compound(IMC) layer formed at solder/Cu interface and the bulk solder hardness. Reflow soldering was performed at 240°C and the as-soldered joints was aged isothermally at 120, 150, and 180°C for various times. With increasing the Bi content, both the shear strength and bulk solder hardness increased and the fracture mode shifted from the bulk solder to the solder/IMC interface. Finely dispersed precipitates of Bi, Ag3Sn and Cu6Sn5 in the bulk solder of as-soldered joints coarsened during aging, resulting in significant changes in the shear strength and the bulk solder hardness with increasing the aging time.

11:15 AM

Microstructural Characterization of Diffusion Barriers for Gold/Tin Solder Bonding of Microelectronic/Optoelectronic Devices: *Anqiang He*[1]; James N. Broughton[2]; Douglas G. Ivey[1]; [1]University of Alberta, Dept. Cheml. & Matls. Eng., Edmonton, Alberta T6G 2G6 Canada; [2]Micralyne, Inc., 1911-94 St., Edmonton, Alberta T6N 1E6 Canada

Gold-tin eutectic solders are commonly used in the optoelectronic and microelectronic industries for chip bonding to dies. Au-Sn solder is classified as a hard solder with superior mechanical and thermal properties relative to soft solders, such as the Pb-Sn system. Au-Sn solder can be applied in a number of ways, i.e., as a preform, solder paste, by evaporation and by electrodeposition. Compared with solder preforms and pastes, evaporated and electrodeposited solders are cleaner and provide more precise thickness and positional control. Electroplating has the added advantage of being a low cost process relative to evaporation. An electroplating process has been developed for depositing Au-Sn alloys, over a range of compositions (15-50 at% Sn) and thicknesses (up to several μms), from a single, non-cyanide, slightly acidic, solution. During chip bonding to carriers, such as AlN, there may be metallurgical reactions between the molten solder and the metallizations associated with both the carrier and the chip. The metallizations generally consist of an adhesion layer, a barrier and a capping layer (typically Au). The capping layer will dissolve, at least partially, in the molten solder leaving the barrier exposed to the solder. The goal of this work is to assess the effectiveness of various barriers, including Mo, NiV, TiW and NiCu, in terms of metallurgical behaviour. The phase changes accompanying solder bonding are studied using both scanning and transmission electron microscopy (SEM and TEM).

11:35 AM

Characterization and Modeling of Microstructure Evolution Sn-Ag Solder: *A. Telang*[1]; T. R. Bieler[1]; K. N. Subramanian[1]; [1]Michigan State University, Dept. of Matls. Sci. & Mech., 3536 Engineering Bldg., San Luis Obispo, MI 48824-1226 USA

Evolution of microstructural characteristics in solder joints alters their properties in a continuous manner, and poses significant challenges in developing reliability prediction models. Using Orientation Imaging (OIM), microstructures of the Sn-Ag solder specimens exposed to different aging and thermomechanical fatigue conditions are investigated. The effects of aging and fatigue on refining the grain size and changing the grain misorientations are measured, and compared to models for evolution of high angle grain boundaries from low angle boundaries. A goal will be to understand why creep/stress relaxation studies on these solder joints suggest that a dislocation climb mechanism is rate controlling, while observations of surface features indicate a high significance of grain boundary sliding that is commonly associated with a lower stress exponent than climb. Acknowledgment: Financial support for this study was provided by National Science Foundation under grant NSF-DMR-0081796.

Magnesium Technology 2002: High Temperature Alloy Development - Mechanical Properties - I

Sponsored by: Light Metals Division, Magnesium Committee, International Magnesium Association

Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA; Gerald S. Cole, Ford Motor Company, Ford Research Laboratories, Dearborn, MI 48121 USA; Rod Esdale; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; John L. Mihelich, Metal Experts International, Winston, GA 30187 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. Pekguleryuz, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Bob R. Powell, General Motor Corporation, NAO Research and Development Center, Warren, MI 48090-9055 USA; Allen Schultz, Hatch, Mississauga, Ontario L5K 2R7 Canada

Tuesday AM — Room: 606
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Mihriban O. Pekguleryuz, Noranda, Noranda Tech. Ctr., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada; Bob R. Powell, General Motor Corporation, NAO R&D Ctr., PO Box 9055, Warren, MI 48090-9055 USA

8:30 AM

New Die-Casting Alloy MRI153 for Power-Train Applications: *Frank von Buch*[1]; Soenke Schumann[1]; Horst Friedrich[1]; Boris Bronfin[2]; Eli Aghion[2]; Barry Leslie Mordike[3]; Menachem Bamberger[4]; Dan Eliezer[5]; [1]Volkswagen AG, Matls. Rsrch., K-EFFS, Letter box 1511, Wolfsburg 38436 Germany; [2]Dead Sea Magnesium, Ltd., Magnesium Rsrch. Inst., Potash House, PO Box 75, Beer-Sheva 84100 Israel; [3]Technical University of Clausthal, Dept. of Matls. Eng. & Tech., Agricolastrasse 6, Clausthal-Zellerfeld 38678 Germany; [4]Technion, Dept. of Matls. Eng., Haifa 32000 Israel; [5]Ben Gurion University of the Negev, Dept. of Matls. Eng., PO Box 653, Beer-Sheva 84105 Israel

For the production of manual transmission housings the conventional magnesium die-casting alloy AZ91 is used in the Volkswagen Group for many years. Due to the fact that the tendency of development follows the trend to higher torque and higher temperatures and from manual to automatic and variable transmissions it is obvious that alloy AZ91 reaches the upper bound regarding high temperature strength and creep-resistance. Therefore in 1996 Volkswagen initiated an extensive research program aimed at developing a low-cost, creep-resistant magnesium die-casting alloy with sufficient high temperature strength. This paper deals with the systematic, the structure, the sequence and the results of this alloy development. The whole chain from first trials in laboratory scale to the production and testing of components will be described in detail. At the end of this development the new magnesium die-casting MRI153 will be commercially available.

8:55 AM

Computational Thermodynamics and Experimental Investigation of Mg-Al-Ca-Sr Alloys: *Yu Zhong*[1]; Koray Ozturk[1]; Alan Luo[2]; Zi-Kui Liu[2]; [1]Pennsylvania State University, Dept. of Matls. Sci. & Eng., Steidle Bldg., University Park, PA 16802 USA; [2]General Motors Research and Development Center, Matls. & Proc. Lab., 30500 Mound Rd., Warren, MI 48090-9055 USA

The thermodynamic properties of the Mg-Al-Ca-Sr system are investigated computationally, based on the relevant binary and ternary systems with most intermetallic compounds treated as stoichiometric compounds. Consequently, the phase relations at any temperature and composition can be readily calculated. Furthermore, Scheil simulations of solidification processes are carried out and compared with experimentally measure fraction of precipitates. The results are used to understand fundamentals of existing alloys and development of new alloys.

9:20 AM

Precipitation Hardening in Mg-Zn-Ca Alloys: *Menachem Bamberger*[1]; Paula Jardim[2]; Guillermo Solorzano[2]; John VanderSande[3]; [1]Technion, Dept. of Matls. Eng., Haifa 32000 Israel; [2]Catholic University of Rio de Janeiro, Matls. Sci. & Metall., 38008 Gavea, Rio de Janeiro, RJ 22452-970 Brazil; [3]MIT, Matls. Sci. & Eng., Rm. 13-5025, 77 Massachusetts Ave., Cambridge, MA 02139 USA

This presentation will address the development of new Mg alloys based on the reduction of the γ-$Mg_{17}Al_{12}$ phase, which exists in Mg-Al based alloys, increasing thereby the properties at elevated temperatures. In the current study, the Al was replaced by Zn and Ca suppressing the undesired phase and allowing for precipitation hardening. Gravity die-cast disks 60mm in diameter and 10mm thick containing various amounts of Ca and Zn were solution treated and aged at 175C. In these Mg-Zn-Ca alloys, the hardness increases with aging time up to 8 hours followed by typical overaging the hardness decrease. Structural analysis and thermodynamic calculations reveals that the change in the hardness can be correlated to the precipitation of $Mg_6Zn_3Ca_2$.

9:45 AM Break

10:05 AM

The Effect of Exposure to Elevated Temperature on the Microstructure and Hardness of Mg-Ca-Zn Alloy: *Menachem Bamberger*[1]; Ludmila Shepeleva[1]; Eugene Rabkin[1]; Amir Finkel[1]; [1]Technion, Matls. Eng., Haifa 32000 Israel

A ternary alloy of Mg-5wt%Ca-6wt%Zn was cast into steel mold and then exposed to 160°C for 40 days. Microstructure analysis, microhardness and hardness measurements conducted in alloy samples once in a couple of days enabled monitoring the thermal stability of the cast alloy. The as cast structure is composed mainly of α-Mg solid solution, and in the grain boundaries 2mm elliptical precipitates of $CaMg_2$ and eutectic structure of Mg and $Ca_2Mg_6Zn_3$ were found. The α-Mg grain size was 10μm and no change was observed as a result of the exposure to elevated temperature. The structure of the alloy did not changed during the treatment, but small increase of the amount of the intergranular phases was observed. Exposure to 160°C was resulted in a decrease in micro-hardness of α-Mg grains, but no change in the overall hardness of the alloy was observed during this time. Microstructural analysis point out on diffusion of the solute elements from the α-Mg grains to grain boundaries resulting in a decrease in the hardness of the α-Mg. This is compensated by the increase in hardness related to inter-granular precipitates.

10:30 AM

Process Parameters and Diecasting of Noranda's AJ52 High Temperature Mg-Al-Sr Alloy: *Donald Argo*[1]; Michel Lefebvre[1]; Robert Bouchard[1]; [1]Noranda, Inc. Technology Centre, Matls. Eng., 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada

Various components have been diecast with the AJ52 high temperature Mg-Al-Sr alloy developed by Noranda. These include typical test bars/plates designed for property evaluations and various industrial components, including several designed for automotive power train applications as well as an engine valve cover. As a consequence, a significant amount of data has been generated as to the best parameters to be employed for successfully diecasting the AJ52 alloy. This paper summarizes the results of these casting evaluations and looks more closely at the effect of various casting parameters such as melt and die temperature, fill time and gate velocity on castability and properties. This is done by means of a designed experiment involving die cast test bars and the use of a modified industrial die designed to include ribs, cored bosses and other typical die casting features which would highlight problems with hot tearing and die sticking.

10:55 AM

The Effect of the Microstructure on the Creep Behaviour of AJ52X (MG-AL-SR) Die Casting Alloys: *Éric Baril*[1]; Mihriban O. Pekguleryuz[1]; Rubens Verni[1]; [1]Noranda, Inc., Tech., 240, Hymus Blvd., Pointe-Claire, Québec H9R 1G5 Canada

The solidification characteristics of AJ52x (Mg-Al-Sr alloys) were determined using the cooling curves of the alloys, the detailed microstructural characterisation results, and subsequent heat treatments. Depending on the strontium content of AJ52x alloy, the alloy contains various types of 2nd phase particles that precipitate at relatively high temperatures (between 510 and 535°C). The thermal stability of the alloys and the 2nd phase particles was studied using high temperature heat treatments. The mechanical properties and the creep behaviour of the heat treated samples were determined and the microstructure was characterised using SEM, TEM and XRD. It is proposed that the presence of thermally stable 2nd phase particles is beneficial to the creep resistance at elevated temperature.

11:20 AM
Effect of Mg2Si Particles on the High Temperature Mechanical Properties of Squeeze Cast Mg-Al Alloys: Nack J. Kim[1]; *Min Soo Yoo*[1]; Jae Joong Kim[2]; Kwang S. Shin[3]; [1]POSTECH, Ctr. for Adv. Aeros. Matls., San 31, Hyojadong, Pohang, Kyungbuk 790-784 Korea; [2]MIT, Dept. Matl. Sci. & Eng., Cambridge, MA USA; [3]Seoul National University, Sch. of Matl. Sci. & Eng., Seoul Korea

Mg alloys offer the potential for significant weight savings in automotive and aerospace components because of their low density. Nevertheless, there is a strong need for Mg alloys with improved high temperature mechanical properties. The main objective of the present research is to investigate the effect of Mg2Si particles on the high temperature tensile and hardness properties of squeeze cast Mg alloys. Mg2Si particles usually have Chinese script morphology which is detrimental to the mechanical properties. The morphology of Mg2Si particles has been modified to the polygon by adding Ca or P to the base AZ61 alloy. It has been shown that the newly developed alloys have better combinations of room temperature and high temperature tensile properties than the conventional AZ and AM alloys.

Materials Processing Fundamentals - I

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee
Program Organizers: P. N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam Powell, Massachusetts Institute of Technology, Cambridge, MA 02139-4301 USA

Tuesday AM Room: 614
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chair: Princewill N. Anyalebechi, Grand Valley State University, Padnos Sch. of Eng., L. V. Eberhard Ctr., 301 W. Fulton, Ste., 617, Grand Rapids, MI 49504-6495 USA

8:30 AM
Materials Processing Software for Educational Use: *Arthur E. Morris*[1]; Richard K. Luu[2]; [1]Thermart Software, 12102 Calle de Maria, San Diego, CA 92128-2720 USA; [2]General Atomics, AAA Div., Los Alamos, NM USA

Process modeling software is used extensively in process engineering, but there is a notable lack of affordable software for educational uses. This paper describes two educationally-oriented software programs for modeling pyrometallurgical processes. Both programs run as Excel spreadsheet files. The first is FREED, a thermodynamic database program containing datafiles for 2500 species. It contains the entire US Bureau of Mines and most of the US Geological Survey database, plus data for scores of other species drawn from various sources. Several modules are available for manipulating and viewing the data. The development of FREED was carried out at the Department of Metallurgical Engineering, University of Missouri-Rolla as part of a project to preserve and enhance the Bureau of Mines database. The second program is THERBAL, for making thermodynamic calculations using FREED data. Equilibrium calculations can be made for systems with non-ideal solutions, for a specified set of pressure, temperature, and input amounts of reactants. An optional heat balance is also calculated. The results are displayed as tables or charts in Excel worksheets. Examples of the use of THERBAL will be given for the reforming of natural gas for DRI production, the direct smelting of lead, and the desulfurization of steel. Some observations will be made on its use in the classroom.

9:00 AM
Application of a Commercial CFD Code to Predicting the Behavior of a Bubble Stirred Melt and Comparison with Experimental Results: Dipak Mazumdar[2]; *James W. Evans*[1]; [1]University of California, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA; [2]Indian Institute of Technology-Kanpur, Dept. of Matls. & Metlgcl. Eng., Kanpur 208016 India

The application of computational fluid dynamics (CFD) to bubble driven flow and bubble behavior has evolved considerably in the past two or three decades. Much of the interest has focussed on the dynamics of liquid steel in gas stirred ladles. Because of this evolution in CFD it is now possible to simulate many real features of the flow (e.g. deformation of the melt surface) that were previously ignored by mathematical modelers. The paper describes the use of FLUENT® to simulate bubbles and bubble driven flow in a molten metal. Although the calculations are simplified to a degree (axisymmetric), the results reveal many real features such as the development of bubbles of a realistic size and the ejection of liquid into the freeboard as the bubbles breach the surface. A water model has been constructed and particle image velocimetry used to measure the acceleration of the water from rest as gas injection is commenced. The results of the measurements are compared with results calculated by FLUENT®.

9:30 AM
Dynamics of Solidification and Microstructure Evolution in Undercooled Co-Cu-Alloys with Metastable Miscibility Gap: *Matthias Kolbe*[1]; Xiaoyu Lu[1]; Chongde Cao[2]; Peter K. Galenko[1]; Jan Fransaer[3]; Dieter M. Herlach[1]; [1]German Aerospace Center (DLR), Inst. of Space Simulation, Linder Hoehe, Koeln D-51170 Germany; [2]North Western Polytechnical University, LMSS, Xi'an 710072 China; [3]Catholic University of Leuven, MTM, Kasteelpark Arenberg 44, Heverlee B-3001 Belgium

Co-Cu exhibits a metastable miscibility gap in the region of the undercooled melt. Specimens have been undercooled and solidified containerlessly into the metastable miscibility gap in an electromagnetic levitation facility (EML) and in drop tube experiments. Due to the high undercooling the velocity of the solidification front is very high and the microstructure is frozen in instantaneously. The microstructure of samples processed in the EML is influenced by the electromagnetic stirring due to the induction of electric currents into the melt. Drop tube experiments, which lead to a rapid solidification under reduced gravity conditions, in contrary allow for the observation of the early stages of demixing: the microstructure shows a homogeneous distribution of spherical particles of the minority phase. The solidification velocity as a function of undercooling has been measured in the EML for the composition Co-18.8at%Cu. The data can be explained in the framework of the LKT/BCT model with a velocity-dependent distribution coefficient as well as within a model of Galenko/Danilov using a kinetic growth coefficient of μ=0.22 m/Ks.

10:00 AM Break

10:30 AM
Direct-Visualization of High Temperature Processes in Metals: *Sridhar Seetharaman*[1]; Alan Cramb[1]; [1]Carnegie Mellon University, Dept. of Matls. Sci. & Eng., 5000 Forbes Ave., Pittsburgh, PA 15213 USA

A unique understanding of phenomenon taking place in molten metals is achieved by observing alloy samples in-situ through a Confocal Scanning Laser Microscope. The confocal optics in combination with a laser source and real time 3D imaging, enables uneven surfaces of molten metal melts and semi-solids to be observed with great clarity and a high signal/noise ratio. This paper presents recent results on the solidification and evolution of inclusions in carbon/stainless steels and superalloys. The collision, agglomeration and particle pushing/engulfment of inclusions is elucidated.

11:00 AM
Using a Magnetic Body Force to Control Convection in Studies of Solidification: *Chris D. Seybert*[1]; James W. Evans[1]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA

Natural convection, caused by differences in density which, in turn, are due to differences in temperature, solute concentration or both, dominates the solidification of liquids in normal gravity. Recently there have been experiments in orbiting laboratories where the reduction of gravity to microgravity nearly halts natural convection so that diffusion of heat and solute become important and may be studied. The paper describes an alternative technique that exploits a strong magnetic field gradient to achieve the same objective. The field gradient results in a magnetic body force that is analogous to gravity in that it has a well defined direction and depends on a well defined property (susceptibility) of the material on which it acts. Consequently there is an analog of buoyancy if the susceptibility varies within a liquid subjected to the field gradient. In this way, a "magnetic buoyancy" occurs and can be made to counterbalance the normal buoyancy. Experimental work on a paramagnetic liquid (manganese chloride) has been completed and convection has been substantially reduced (but not eliminated) in experiments on the surface of the Earth. Unfortunately most liquids are diamagnetic and very powerful magnets are required to halt convection in such liquids. The paper presents the results of calculations of flow in some candidate magnets. The paper also examines the possibility of exploiting this technique in reducing residual accelerations ("g-jitter") in spacecraft where experiments must be carried out with minimal disturbance of a fluid. Research supported by NASA.

11:30 AM
Effect of the Exposed Copper Area/Volume of the Solution Ratio and Aeration on the Formation of an Artificial Patina: *M. A. Llavona*[1]; B. Díaz[1]; A. M. Fernandez[1]; M. C. Garcia[1]; J. L. Ibanez[1]; N. Velasco[1]; R. Zapico[1]; [1]University of Oviedo, Dept. of Matls. Sci., US Mining & Topographic Eng., Reinerio Garcia s/n., 33600, Mieres Spain

When exposed to the atmosphere, some metals form a thin layer of corrosion which on copper is commonly green or greenish-blue. This protective Patina layer is chemically and physically complex structure, the surfaces show a highly porous structure and it is regarded as an aesthetically pleasing as well as a protective coating of the metal substrate. The changing environment has accelerated the processes of Patinas degradation; thus, the exhaustive study of factors capable of affecting the Patina formation, growth and durability has assumed great importance for the industrial activity of copper.

Modeling of Multi-Scale Phenomena in Materials Processing: Solidification Microstructure

Sponsored by: ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, Jt. Computational Materials Science & Engineering, Solidification Committee

Program Organizers: Adrian Sabau, Oak Ridge National Laboratory, MS-602, Oak Ridge, TN 37831-6083 USA; Boyd A. Mueller, Howmet Corporation, Whitehall, MI 49461-1832 USA; Anthony D. Rollett, Carnegie Mellon University, Department of Materials Science & Engineering, Pittsburgh, PA 15213-2890 USA

Tuesday AM — Room: 304
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Boyd A. Mueller, Howmet Corporation, Whitehall, MI 49461-1832 USA

8:30 AM
Comparison of Measured and Predicted Columnar to Equiaxed Transitions in Unidirectionally Solidified Binary Alloys: Marcelo A. Martorano[1]; *Christoph Beckermann*[1]; [1]University of Iowa, Mechl. Eng., 2412 SC, Iowa City, IA 52242 USA

A multi-phase multi-scale model was used to predict the columnar to equiaxed transition (CET) during the directional solidification of binary alloys. The model is similar to that proposed by Wang and Beckermann (1994) and is based on the solution of energy, mass and species conservation equations, coupled with nucleation and growth laws for the various dendritic structures. The directional solidification of three alloys, namely Al-Cu, Cu-Sn and Al-Si, was simulated using a density of solid nuclei based on the measured equiaxed grain size. Model results of cooling curves and CET positions for all simulations were analyzed and compared with experimental measurements. The effect of nucleation undercooling on the CET position provided by the model was also assessed by a parametric study. Finally, a nucleation undercooling resulting in the best agreement between calculated and predicted CET positions was determined for each alloy system.

8:55 AM
Modeling of Coalescence and Separation of Liquid Droplets during Solidification of Immiscible Alloys: Lirong Tong[1]; *Nagy El-Kaddah*[1]; [1]The University of Alabama, Dept. of Metall. & Matls. Eng., Box 87202, Tuscaloosa, AL 35487-0202 USA

A basic understanding of droplet motion, coalescence and solidification is essential for quantitative predictions of dispersion characteristics of cast immiscible alloy composites. This paper presents a mathematical model for describing the growth of nucleated dispersed phase in the two-liquid phase region ahead of the solidification front and the entrapment of these droplets by the moving solid-liquid interface in vertical unidirectional solidification systems. The model has two components. A macro-heat transfer model for describing the temperature profiles and the rate of advance of the solidification front. The dynamic behavior and coalescence and growth of nucleated droplets in the two-liquid phase region under the influence of gravity and thermocapillary forces (Marangoni motion) were represented through the solution the droplet momentum and mass conservation equations in particle space. These two components of the models were coupled through a special algorithm for tracking the particle location and size with respect to moving solidification front in the solidification time scale. The model is applied to study the particle size distribution in unidirectional solidified Zn-Bi alloys. The model predictions were found to be in reasonable agreement with experimental data for this system.

9:20 AM
Solidification Modeling of Atomized Droplets: Arvind Prasad[2]; *Romulo Heringer Ferreira*[1]; *Charles-Andre Gandin*[1]; Hani Henein[2]; [1]LSG2M, Ecole Des Mines, UMR CNRS-INPL-UHP 7584, Parc de Saurupt, Nancy, Cedex F-54042 France; [2]University of Alberta, Dept. of Cheml. & Matls. Eng., 536 CME Bldg., Adv. Matls. & Procg. Lab., Edmonton, Alberta T6G 2G6 Canada

Atomization of liquid is a unique rapid solidification process for the production of metal powders and spray deposition. Of great interest is the prediction of the microstructure and segregation state of the rapidly solidified powders, i.e. the nature and volume fraction of the phases solidified. Such predictions are not trivial due to the strong interaction between the heat flow and the phase transformations, which occur under conditions far from equilibrium (recalescence, temperature dependent heat extraction rate, large nucleation undercooling, phase selection in the coupled zone of the phase diagram, kinetics effects on structure development and microsegregation). Calibrations of the models are also difficult since experimental data recorded during the process are usually not available, or are very limited. Two models of droplet solidification will be presented and compared with experimental results. These are based on in-flight droplet temperature measurements during atomization and X-ray diffraction measurements of the eutectic fraction of the solidified droplets.

9:45 AM
Microstructure Modeling of Investment Cast Nickel Base Superalloys: *Ty W. Hansen*[1]; Boyd A. Mueller[1]; [1]Howmet Research Corporation, Elect. Prod. Tech., 1500 S. Warner St., Whitehall, MI 49461 USA

Process Modeling has been used successfully to reduce the time and cost of developing investment casting processes for nickel base superalloys. The process models examine the macroscopic phenomena of heat transfer and fluid flow. As such, these models are useful for predicting defects such as porosity, primarily by defining gating size and location, and appropriate casting parameters. To date, Howmet has been unsuccessful in predicting grain size or shape in equiaxed or directionally solidified castings. The results were inaccurate because the time dependence of nucleation was not modeled. Recently, the functionality of the commercial CAFÉ stochastic microstructure modeling code has been enhanced such that it now is able to account for the cooling rate dependence of nucleation. This microstructure modeling code, when coupled with a macroscopic heat transfer and fluid flow simulation, is able to accurately predict the types and ranges of microstructures found in typical investment castings. Examples ar e given of predicted grain size and shape in nickel base superalloy investment castings.

10:10 AM Break

10:25 AM
Modeling of Inductive Stirring and Microstructure Formation during Solidification: *Ben Q. Li*[1]; Nirmal Gravinaraju[1]; [1]Washington State University, Sch. of Mech. & Matls. Eng., Pullman, WA 99164 USA

An integrated numerical model is developed to represent complex electrodynamic, fluid flow and heat transfer and microstructure formation in solidifying metals with electromagnetic stirring. The model is developed based the hybrid finite element and boundary element solution of the Maxwell equations and the finite element solution of transport phenomena equations. This macroscale solution is coupled with the Monte-Carlo-Cellular Automata method for the representation of microstructure formation solidifying metals stirred by induction forces. A multi-time and space coupling scheme is presented. Parallel computing algorithm is developed to improve the computational performance. Results are presented that illustrates the effect of electromagnetic stirring on the solidification microstructure formation.

10:50 AM
A Phase-Field Study of Kinetically Limited Dendritic Growth: *Andrew Martin Mullis*[1]; [1]University of Leeds, Dept. of Matls., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK

Dendrites are the most commonly encountered solidification morphology observed in metals. Recent studies have shown that virtually all the important characteristic of dendritic growth (primary trunk radius, secondary arm spacing, surface area, volume, fractal dimension) can be scaled against the tip radius, R. It has previously been universally found that in the solidification of pure metals, R decreases monotonically with increasing undercooling. However, in this presentation we will discuss the results of phase field simulations which indicate that this may not be true in systems with strong interface kinetics. As growth velocity increases we find that a transition from diffusion limited growth to kinetic limited growth occurs and that this

transition is delineated by a minimum value of R. Using parameters appropriate to pure Ni we find that this transition would occur at a growth velocity of 18 m/s.

11:15 AM
3D Modeling of Porosity Formation during the Solidification of Aluminum Alloys, using a Mushy Zone Refinement Method: *Ch. Pequet*[1]; M. Rappaz[1]; [1]Ecole Polytechnique Fédérale de Lausanne, Laboratoire de Métallurgie Physique, Département des Matériaux, Lausanne CH-1015 Switzerland

Porosity, a very severe defect in castings, is caused by solidification shrinkage and gas segregation. Solidification shrinkage induces a pressure drop in the mushy zone while gas is rejected at the solid/liquid interface. If the gas concentration in the liquid locally exceeds the saturation limit (supersaturation), pores can nucleate and grow. A comprehensive model of porosity formation has been developed for 2D and 3D geometry. While the heat and mass transfer phenomena are solved for the entire casting using FEM, the pressure drop is calculated only in the mushy zone by solving the coupled Darcy-mass conservation equations using a dynamic refined FV mesh. For each time step, liquid pockets are identified to fix appropriate boundary conditions at the frontier of the mushy zone. These calculations are coupled with a model of gas segregation and nucleation/growth of pores, therefore predicting microporosity and pressure maps, as well as macroshrinkage cavities and the shape of free surface (pipe shrinkage). Simulations of several aluminum castings are compared with experiments.

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium
Chemical and Other Processing Methods - Session III

Sponsored by: Materials Processing & Manufacturing Division, Jt. Computational Materials Science & Engineering, Powder Materials Committee, Jt. Processing Modeling Analysis & Control Committee, Surface Engineering Committee, Shaping and Forming Committee, Solidification Committee

Program Organizers: Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; T. Dennis Claar, Fraunhofer USA, Newark, DE 19716 USA; T. H. Sanders, Georgia Institute of Technology, Department of Materials Science and Engineering, Atlanta, GA 30332 USA

Tuesday AM Room: 205
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Stephen R. Nutt, University of Southern California, Matls. Sci., 3651 Watt Way, VHE-602, Los Angeles, CA 90089-0241 USA; Wynn S. Sanders, Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 8-135, Cambridge, MA 02139 USA

8:30 AM
Multifunctional Metallic Honeycombs by Thermal Chemical Processing: *Joe K. Cochran*[1]; Jim Lee[1]; David L. McDowell[2]; Tom H. Sanders[1]; [1]Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA; [2]Georgia Institute of Technology, Mech. Eng., 801 Ferst Dr., Atlanta, GA 30332-0405 USA

Thin-walled metallic honeycombs with variable cell geometry and alloy systems are being developed at Georgia Tech. Using conventional powder processing, i.e. extrusion, precise shapes are formed with non-metallic precursors, which are converted to the metallic state by reduction in hydrogen. For intricate geometries requiring good precision, fine powder sizes about 1/50th of the feature dimension are necessary. By controlling the reduction process, parts fabricated formed from brittle powder precursors are converted into a metal product. Alloys that have been successfully reduced include certain stainless steels, maraging steel, Inconel 617 and 718, Super Invar, and a variety of copper alloys. Strength and specific energy absorption of a maraging steel honeycomb at 1.5 gm/cc were approximately 400 MPa and 100 Joules/gm, respectively. Properties at this level are suited for lightweight structural components with high-energy absorption capability in defense and civil transportation areas. Honeycomb structures fabricated from copper alloys show high heat transfer at low pressure drops that would be ideally suited for forced air heat sinks.

8:55 AM
Metal Honeycomb from Oxide Paste: Maraging Steel Structure and Properties: *Justin L. Clark*[1]; Joe K. Cochran[1]; Thomas H. Sanders[1]; [1]Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

A study is under way to produce complex metal honeycombs through a powder oxide extrusion process. The extruded oxide pastes are subsequently reduced in a hydrogen atmosphere to a metallic body. One of the alloy systems being investigated is 18% Ni maraging steels. These steels are well known for their excellent combination of high yield strength and fracture toughness. A focus of this study is the characterization of the bulk maraging steel material produced through the reduction process. As in traditional powder processing methods, porosity, homogeneity, shrinkage, and microstructure must be understood with respect to the various processing steps. Elastic modulus, yield strength, and elongation of the bulk material are required for predicting the performance of the honeycomb. To date, densities approaching 100% theoretical and yield strengths approaching 70% of the reference values have been observed. The relationships between processing, microstructure and properties will be presented.

9:15 AM
Reduction of Fe2O3 - Cr2O3 Powder Mixtures for Metal Honeycomb Structures: *Jason Hayes Nadler*[1]; Thomas H. Sanders[1]; Joe K. Cochran[1]; [1]Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30030 USA

The reduction of Fe2O3 and Cr2O3 powder mixtures has been studied in order to better understand the microstructural evolution of chromium containing alloys for metal honeycomb structures. The effects of sintering, porosity and sample composition are discussed. Samples were prepared by mechanically mixing oxide powders and pressing into pellets. All samples were reduced with hydrogen in an infrared gold image furnace at 1573K. None of the samples exhibited complete reduction of Cr2O3. However, the amount of reduced chromium increased with higher Cr2O3 compositions. The results, in agreement with previous work, suggest that gaseous diffusion is the most prevalent limitation to Cr2O3 reduction. Sample preparation was also observed to have an affect on the amount of Cr2O3 that will reduce. Sintering the pellets prior to reduction yielded higher chromium concentrations when compared to directly reduced powder compacts. Thermodynamics and kinetics of Cr2O3 reduction will be discussed in terms of free energy of formation of reduced solid solutions as well as the rate limitations of gaseous diffusion and interfacial chemical reactions.

9:35 AM
Copper Alloys from Oxide Reduction for High Conductivity Applications: *Benjamin C. Church*[1]; Joe K. Cochran[1]; Thomas H. Sanders[1]; [1]Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA

Extrusion of oxide powders allows fabrication of thin-walled metal articles to produce controlled geometry, low-density copper alloy architectures. Shapes formed with copper oxide powders mixed with alloying oxides are reduced and sintered to produce high relative densities in the thin walls. This technology has produced square cell honeycomb extrusions, which are being characterized for heat exchange applications. This effort is to determine the bulk properties of alloys produced by this type of thermo-chemical powder processing and to explain behavior based on the final chemistry and microstructure of the alloys. Compositions investigated include Cu, Cu-Ni, and Cu-Ag alloys. Alloys have been characterized for relative density, porosity, pore size distribution, and grain size. Mechanical properties, tensile strength and elongation, and thermal conductivity were measured and results were analyzed based and porosity and chemistry of the alloys. Properties were compared to alloys from conventional processing and powder metallurgy.

9:55 AM Break

10:15 AM
Modeling Powder Extrusion Pastes for Forming Light Weight Multifunctional Structures: *K. M. Hurysz*[1]; R. Oh[1]; J. K. Cochran[1]; T. H. Sanders[1]; K. J. Lee[1]; [1]Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30332 USA

The extrusion of pastes of oxide powder mixtures through a thin-wall honeycomb die results in a low-density metallic structure following reduction. These pastes are a combination of two phases: a solid phase composed of the particular oxide or oxide mixture carried by a fluid solution of water, binder, and lubricant. The key to forming high quality, defect free extrudate lies in the optimization of paste properties and is contingent on solids loading and fluid-phase rheology. To extrude efficiently, the paste must be compliant to allow flow through

the die, yet provide a high enough yield stress at low shear rates to avoid deformation following extrusion. To develop a model that predicts paste bulk shear stress, capillary rheometry was used to characterize aqueous binder gels and powder pastes using the binder gels as the fluid phase. Rheology models based on solids loading were successfully applied to permit rapid optimization of solids content and binder gel solutions for extrusion of articles having complex geometry.

10:35 AM
Advances in the Melt Route Production of Closed Cell Aluminium Foams using Gas-Generating Agents: *V. Gergely*[1]; D. C. Curran[1]; T. W. Clyne[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

A brief survey is given of the current state of the art for hydride-based processing of closed cell foams, including the production of precursor material in which gas release has been delayed during dispersion in the melt by the introduction of surface coatings on the powder particles (the FORMGRIP process). A study is then presented of the factors involved in the search for gas-generating agents offering superior performance. These include kinetic and thermodynamic characteristics of potential decomposition reactions, the ease of dispersion of the powder in the melt, the nature and likely effect of decomposition products on melt flow and the availability and cost of the powder concerned. It is shown that there are one or two very promising candidate materials, which can offer advantages compared to hydride powders in virtually all aspects of their performance. It is confirmed that foams can be produced having appreciably finer (<1 mm cell size) and more uniform cell structures than currently-available melt route foams, a lower ceramic content in the cell wall microstructure and dramatically reduced raw material costs. It is also shown that stress-strain curves during compressive loading of such material are much smoother and more progressive than those of previously-available melt route material, which is consistent with the finer and more homogeneous structure and may also be at least partly due to greater ductility exhibited by the cell walls.

10:55 AM
Solid State Processing of Titanium Foams by Transformation Superplasticity: *Naomi G. Davis*[1]; David C. Dunand[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Rm. 2036, Evanston, IL 60208 USA

Solid-state foaming of titanium and titanium alloys can be achieved by hot-isostatic pressing powders with argon gas, followed by high temperature expansion of the resulting high-pressure argon bubbles. This foaming technique, which is currently under industrial development at Boeing Corp. for Ti-6Al-4V as their LDC material, is limited by the low creep rate and ductility of the metal, which lead to slow pore growth and early cell wall fracture limiting the maximum porosity. We address these issues by performing the foaming step under transformation superplastic conditions where the foam is thermally cycled around the α/β allotropic temperature of titanium. This induces superplasticity due to the complex superposition of internal transformation stresses and the external biasing stress from the pore pressure. Variations in processing conditions as well as the initial pore size and backfill pressure are discussed with respect to the foaming kinetics, the terminal porosity and the open/closed porosity ratio.

Recycling – General Sessions: Automotive and Non-Ferrous Materials Recycling

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee
Program Organizers: John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Wolfgang Schneider, VAW Aluminium AG, Reseach & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday AM — Room: 604
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Mark E. Schlesinger, University of Missouri-Rolla, Rolla, MO 65409-0001 USA; Greg K. Krumdick, Argonne National Laboratory, Argonne, IL 60439 USA

8:30 AM
Integrated Technology for Processing Spent Lead Batteries and Other Types of Secondary Lead-Containing Raw Materials by Soda-Less Electric Smelting: *A. D. Besser*[1]; [1]State Research Center of Russian Federation, State Rsrch. Inst. of Non-Ferrous Metals "Gintsvetmet", 13, Acad. Korolyov St., Moscow 129515 Russia

A flow-sheet for waste-less processing of spent lead batteries comprises mechanized separation of batteries, smelting of the metallic fraction in a kettle, electrothermal smelting of oxysulfate fraction, refining of crude lead and processing of reverts to produce commercial products. A review of technologies available worldwide for separation of spent lead batteries has shown that the most suitable and efficient technique for the Russian conditions is the method proposed by the Engitec Technologies Srl. (Italy). The SX-compact unit is an environmentally sound installation, requiring a small volume of construction, low capital investments, short period of time for erection and ensures separation of lead batteries into five fractions (metal, oxysulfate, polypropylene, polyvinyl chloride and ebonite). Smelting of lead-containing fraction is performed in an electrothermal furnace using a processing technology developed in the Gintsvetmet Institute and differing from conventional processes by the fact that it does not require any fluxing agent (soda), no matte is formed in the process and the amount of slag produced is minimal, because its formation depends only on the ash content of coke and the quality of battery scrap separation. The process has been patented. It has been introduced on a commercial scale in 1.8 MVA furnaces with a bottom area of 12 m2 and 13 m2 at the plant of AO "RyazTsvetmet" (Ryazan, Russia). Preparations are in progress to commission another plant in the Russian Federation.

8:55 AM
Household Solid Waste Recycling Induced Production Values and Employment Opportunities in Taiwan: *Esher Hsu*[1]; Chen-Ming Kuo[2]; [1]National Taipei University, Dept. of Stats., 67, Sec. 3, Min-Sheng E. Rd., Taipei 104 China; [2]I-Shou University, Dept. of Mechl. Eng., 1, Sec. 1, Hsueh-Cheng Rd., Ta-Hsu, Kaohsiung 84008 Taiwan

Household solid waste recycling has been the major policy of Taiwan Environmental Protection Administration. What are production values and employment opportunities of these recycling not only the focal points of Taiwan Congress, but also the major implementations of recycling policies for EPA. In this study, production values and employment opportunities induced by the household solid waste recycling are investigated by sampling survey with ratio estimation. The estimated production values in 1998 are respectively 26.9 and 44.6 billion on the collection and sorting business, and recycling industry. Total estimated added value is approximately NT$35 billion, of which, 15.5 and 19.5 billion are from the former business and latter industry, respectively, 0.28% and 0.80% of the service and manufacturing sectors in 1998. That provides 189,551 employment opportunities, of which, 182,538 and 7,013 positions are from the former and latter ones, respectively, 3.69% and 0.27% of the service and manufacturing sectors in 1998.

9:20 AM
A Study of Recycling Ru: S. Kagagiri[1]; M. Takayanagi[1]; *I. Jimbo*[1]; [1]Tokai University Japan

In the recent years, the recycled amount of various precious metals and other useful metals is steadily increasing. A cooperative research work on the precious metal recycling is underway between Matsuda Sangyo Co., Ltd. and Tokai University in Japan. Among these precious metals, ruthenium is focused and the recycle process of this element is under investigation by the authors. In this paper, the industrial practice of wastes treatment is introduced and the result of the laboratory studies on the possibilities for Ru to be separated from other precious metal elements will be discussed.

9:45 AM
Possibilities and Use of Automatic Picking within the Automobile Recycling Industry: *Stefan Mutz*[1]; Thomas Pretz[1]; Andreas Weingart[2]; Eric van Looy[2]; [1]RWTH Aachen, Chair for Procg. & Recycling of Solid Waste Matl., Wuellnerstr. 2, Aachen D-52062 Germany; [2]Separation Systems Engineering GmbH, Rosengarten 10, Wedel D-22880 Germany

Although the structure of automobile recycling in Germany is regulated by law in the year 2015 for example 85% of the materials are materially recycled, for 10% of the materials a thermal utilization is certified there are still some concrete questions not answered concerning the usability of single fractions. The different approaches of e.g. innovative dismantling concepts do not modify anything in the fact that the majority of the materials of used cars are going with the utilization the path over the shredder and the connected processing aggregates. The whereabouts of the so called SLF (Shredder light fraction) which is about 25% w.-t. in used cars are not satisfyingly clarified. Also the dry-mechanical processing creates questions regarding the separation of the non-magnetic heavy fraction from the shredder

which has to be separated in the single metallic fractions like stainless tell, copper, brass, zinc, etc. Attempts of optimization can be found. After analysis of the weak positions of the recent processing of cars in Germany, this paper shows the possibilities of the new technologies like automatic picking. Examples of test series with this technology with the most varying material mixtures from the range of the shredders offer a descriptive summary of the possible applications within the range of the used car recycling.

10:10 AM Break

10:20 AM
The Future of Automotive Aluminium: *Petra Zapp*[1]; Georg Rombach[2]; Wilhelm Kuckshinrichs[1]; [1]Forschungszentrum Juelich, Sys. Analy. & Tech. Evaluation, Juelich 52425 Germany; [2]RWTH Aachen, IME Proc. Metall. & Metal Recycling, Aachen 52056 Germany

This paper presents a scenario of aluminium in automotive applications describing the resulting impact on aluminium production and recycling. Based on detailed process chain modelling the use of aluminium in the German car industry is analysed along the entire life cycle chain. To identify major innovations in the automotive industry several life periods of cars have to be covered, which was considered by choosing 2040 as the target year. A variety of parameters influences the demand of metal (primary and/or recycled) and the availability of aluminium scrap. Investigated are market developments of automobiles in general, design strategies for car components differentiating between conventional and aluminium intensive constructions, and diverse engine concepts like combustion engines, hybrid or fuel cell vehicles. For the analysis the automotive aluminium is classified in casting or wrought alloys, which have to be distinguished with regard to recycling aspects.

10:45 AM
Future Availability of Aluminium Scrap: *Georg Rombach*[1]; [1]RWTH Aachen, IME Proc. Metall. & Metal Recycling, Intzestr. 3, Aachen 52056 Germany

The continuously growing importance of metal recycling, especially of aluminium, as part of the raw material supply is indisputable. Nevertheless there are limitations resulting from future development of applications, recycling quotas or metal losses during production, manufacturing and use which influence the entire material flow of primary and recycled aluminium. The growth of aluminium recycling is, unlike primary production, not determined by the industry itself but mainly by the availability of secondary raw materials. This article focuses on recycling potentials of aluminium in Germany and Europe concerning this availability and the quality of secondary raw materials. Different growth rates in each area of application and the product lifetimes will lead to different amounts of both, fabrication and post-consumer scrap. In the analysed case studies also the development and impact of collection efficiency and processing yield are considered.

11:10 AM
Recycling of the Metallic Phases Resulted after Reclamation of Al/SiC(p) Scrap Composites: P. Moldovan[1]; *Gabriela Popescu*[1]; C. Popescu[1]; [1]University Politehnica of Bucharest

In processing of composite materials results lot scraps, which contain metallic phases that can be recovered and used again in different fields. The purpose of our work was to find and develop a recycling process of different "metallic phases" in order to obtain, by selected metallurgical procedures, one of the standard alloys with adequate composition and properties. The basic criterion to prepare an alloy by recycling a given material was: Basic elements from the "metallic-phase" ought to be found in the final alloy; The amount of elements which are not basic elements (in general impurities) should be smaller than the allowed composition of final alloy. Stating from these conditions we choose five different alloys from national standard, with defined chemical composition and allowed content of impurities. The desired composition of the final alloy obtained from scraps could be reached by adding to initial amount of "metallic phases" of some additional elements. In this point, the paper presents a computer program to calculate the amount of additional elements in order to obtain the final alloy.

Second International Symposium on Ultrafine Grained Materials: Nanostructures

Sponsored by: Materials Processing & Manufacturing Division, Shaping and Forming Committee
Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; Terence G. Langdon, University of Southern California, Department of Mechanical Engineering, Los Angeles, CA 90089-1453 USA; Terry C. Lowe, Technanogy, Newport Beach, CA 92627 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; Michael Jeremi Saran, Case Western Reserve University, Cleveland, OH 44106 USA; S. Lee Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Tuesday AM Room: 210
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Dong H. Shin, Hanyang University, Dept. Metall. & Matls. Sci., Kyunggi-Do, Ansan 425-791 Korea; Yong Seog Kim, Hong-Ik University, Dept. Metall. & Matls. Sci., 72-1, Sangsoo-Dong, Mapo-Ku, Seoul 121-791 Korea

8:30 AM Keynote
High Strain Monotonic Deformation of Metals: *Niels Hansen*[1]; Xiaoxu Huang[1]; Darcy A. Hughes[2]; [1]Risø National Laboratory, Matls. Rsrch. Dept., Frederiksborgvej 399, Roskilde DK-4000 Denmark; [2]Sandia National Laboratory, Ctr. for Matls. & Eng. Sci., Livermore, CA 94550 USA

Monotonic deformation of aluminum and nickel by rolling and torsion has shown that the microstructural evolution with increasing true strain up to about five follows a universal pattern of grain subdivision down to boundary spacing of the order of 100 to 200nm. In parallel the flow stress increases without saturation. This behavior suggests that the effect of even higher strains is explored and investigations have covered aluminum deformed by accumulative roll bonding (ARB) and nickel deformed by high pressure torsion (HPT). By this choice of processes it has been possible to compare and analyze the evolution in microstructure and flow stress during deformation by rolling, torsion, ARB and HPT over a large strain range.

8:55 AM Invited
Microstructure of Cold Deformed and Recrystallized Tantalum: *K. T. Hartwig*[1]; S. N. Mathaudhu[1]; [1]Texas A&M University, Mech. Eng., 319 Eng. Phys. Bldg., Spence St., College Station, TX 77843-3123 USA

Vacuum arc remelted pure tantalum was deformed at room temperature to a strain of 4.6. Deformation was accomplished by multipass equal channel angular extrusion through a tool containing an abrupt 90 degree angle on 25.4 by 25.4 mm square bars at a strain rate of approximately two per second. The sequence of orientations of the tantalum billet with respect to the longitudinal axis was chosen to be one that would produce an equiaxed microstructure. The as-worked microstructure shows a progression from shear bands at a strain of 1.2 to the development of subgrain features with dimensions on the order of hundreds of nanometers at a strain of 4.6. The recrystallized grain size for the material strained to 4.6 is under 20 microns. Hardness, metallography and electron microscopy results are discussed.

9:15 AM
Defect Structures and Deformation Mechanisms of a Nanostructured Al-Mg Alloy Prepared by Cryogenic Ball Milling: *Xiaozhou Liao*[1]; Jianyu Huang[1]; Yuntian T. Zhu[1]; Fei Zhou[2]; Enrique J. Lavernia[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]University of California, Dept. of Chem. & Biochem. Eng. & Matls. Sci., Irvine, CA 92697 USA

A nanostructured Al-Mg wt 7.5% alloy was produced by mechanical alloying under liquid nitrogen. The defect structures of the alloy were investigated using high-resolution transmission electron microscopy, electron diffraction, energy-dispersive spectrometry, and energy filtering imaging. Deformation mechanisms at different crystalline sizes will be discussed based on the defects structures of the nanostructured alloy.

9:30 AM Invited

X-Ray Analysis of SPD Nanostructured Materials: *Igor V. Alexandrov*[1]; [1]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Matls., K. Marks str.,12, Ufa, Bashkortostan 450000 Russian

The present report is focussed on the development and application of X-ray structural analysis for studying the defect structure of nanostructured materials processed by severe plastic deformation (SPD). The main attention is paid to the following topical issues and the recent results obtained: 1. The previously carried out X-ray analysis was mainly concentrated on pure nanostructured metals with the FCC crystal lattice such as Cu and Ni. Lately the development of SPD resulted in the opportunity of the microstructure refinement in hard-to-deform BCC and HCP metals such as W and Ti. In this connection the results of the carried out X-ray structural analysis of these SPD processed BCC and HCP metals in comparison with the FCC metals are of interest. 2. The analysis of the microstructure evolution as a result of SPD made it possible, in particular, to ascertain non-monotonous character of changing the size of the crystallites, elastic microdistortions of the crystal lattice, dislocation density, crystallographic texture with high pressure torsion on the example of Cu and Ni. 3. The study of the defect structure of nanomaterials by means of computer modeling of X-rays scattering taking into consideration various assembles of extrinsic grain boundary dislocations has been developed. 4. The X-ray analysis carried out revealed increased (especially large in grain-boundary areas) atomic displacements, a decreased Debye temperature and a changed character of atomic vibration spectrum which cause observed increase in the grain-boundary diffusion coefficient of Cu in nanostructured Ni processed by the SPD method.

9:50 AM Invited

Microstructure Characterisation by X-Ray Diffraction Profile Analysis: *Tamas Ungar*[1]; [1]Eotvos University, Dept. Gen. Phys., POB 32, Budapest H-1518 Hungary

Smallness of crystallites size and lattice distortions are diffraction order independent and dependent, respectively[1]. The g dependence of the mean square strain can be well accounted for by the anisotropic contrast effect of dislocations[2]. The average contrast factors of dislocations, have been compiled for cubic crystals according to a parameter q which depends on the screw or edge character of dislocations and the elastic constants of the crystal[3]. Ab-initio functions of size and strain profiles were constructed assuming log-normal size distribution and using ab-initio profile function for strain[4]. With these two theoretical functions and the hkl dependent factors a procedure has been developed to fit whole diffraction profiles[5]. The procedure will be illustrated by several case studies. [1]B. E. Warren, Progr. Metal Phys. 8, 147 (1959). [2]T. Ungár and A. Borbély, Appl. Phys. Letters, 69, 3173 (1996) [3]T. Ungár, I. Dragomir, Á. Révész and A. Borbély, J. Appl. Cryst. 32, 992 (1999) [4]M. Wilkens, phys. stat. sol. (a) 2, 359 (1970). [5]T. Ungár, J. Gubicza, G. Ribárik and A. Borbély, J. Appl. Cryst. 34, 298-310 (2001).

10:10 AM Invited

Size and Shape of Nano-Grains in Polycrystals Subjected to SPD: *Krzysztof Jan Kurzydlowski*[1]; [1]Warsaw University of Technology, Fac. of Matls. Sci. & Eng., 141 Woloska Str., Warsaw 02-507 Poland

SPD can be used to produce nano-polycrystalline materials. Size and shape of grains in such polycrystals are far different from those observed in conventionally processed materials. The paper describes modern methods of grain size quantification, which are based on principles of stereology, and computer aided image analysis. In particular the question of grain size homogeneity is discussed. Image analysis can also be used to quantify the shape of nano-grains. To this end, shape factors can be used which describe among other their elongation. Examples are given how the modern quantitative methods can be used to characterize and model processes taking place during annealing of SPD processed materials.

10:30 AM Break

10:40 AM Invited

Microstructure, Thermal Stability and Mechanical Behavior of Cryomilled Al Alloys: *E. J. Lavernia*[1]; F. Zhou[1]; V. L. Tellkamp[1]; R. Rodriguez[1]; R. W. Hayes[2]; [1]University of California, Dept. of Chem. & Biochem. Eng. & Matls. Sci., 916 Eng. Tower, Irvine, CA 92697-2575 USA; [2]Metal Technology, Inc., 19801 Nordhoff St., Northridge, CA 91324 USA

A mechanical attrition technique with low energy under liquid nitrogen environment (i.e., cryomilling) was used to produce nanostructured Al materials, including pure Al and Al-Mg alloys. The microstructural evolution during cryomilling was characterized in detail. The grain refinement appeared to be due to total internal strain. The nanostructures produced by cryomilling exhibited extremely high resistance against grain growth at elevated temperatures. As a typical example, the grain growth behavior of cryomilled Al was studied in detail. The high thermal stability was mainly attributed to the grain boundary pinning caused by impurities segregated along grain boundaries as well as dispersoids such as aluminum oxides and nitrides. The cryomilled nanostructured powders were consolidated into bulk samples with limited grain coarsening effects. Preliminary results on tensile and creep behavior of these ultrafine grained Al materials are presented.

11:00 AM Invited

Nanostructures of Titanium Processed by ECAP and Cold Rolling: *Jianyu Huang*[1]; Yuntian T. Zhu[1]; Ruslan Z. Valiev[2]; [1]Los Alamos National Laboratory, Matls. Sci. & Tech. Div., MS G755, Los Alamos, NM 87545 USA; [2]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Matls., K. Marksa 12, Ufa 450000 Russia

Nanostructured Ti was produced by equal channel angular pressing (ECAP) followed by cold rolling. Its defect structures, including grain and subgrain structures, dislocation cells, dislocation distributions, grain boundaries, and the hierarchy of these structural features, were studied using transmission electron microscopy (TEM). We found that large grains (>320 nm) may contain subgrains. Medium sized grains and subgrains (>130 nm) may contain dislocation cells. Small grains and subgrains (<150 nm) may contain random dislocations, or dislocation free if their sizes are <75 nm.

11:20 AM

Defect Characterisation of ECAP Deformed Cu by Selective Annealing Treatment: *Erhard Schafler*[1]; Carl Pilhatsch[1]; Anna Dubravina[2]; Zsolt Kovacs[3]; [1]Universitaet Wien, Inst. fuer Materialphysik, Strudlhofgasse 4, Wien A-1090 Austria; [2]Ufa State Aviation Technical University, Inst. of Phys. of Adv. Matls., 12 K. Marx St., Ufa 450000 Russia; [3]Eoetvoes University Budapest, Dept. for Gen. Phys., Pazmany Peter setany 1/A, Budapest H-1518 Hungary

Cu rods were deformed by Equal Channel Angular Pressing (ECAP) by applying two deformation paths, with (route B) and without (route A) rotation around the samples' axis between ECAP passes. Measurements of residual electrical resistivity and of X-ray Bragg profiles have been performed, in addition to mechanical properties. An isochronal annealing treatment has been carried out, during which the three investigation methods were performed, in order to differentiate between various deformation induced lattice defects and/or their arrangements. Samples of route A and B with the same number of ECAP passes show a different defect annealing behaviour, as concerns the height of the annealing steps as well as the annealing temperatures. The results have been analyzed in terms of rearrangement and annealing of deformation induced vacancies and dislocations. Thus, they allow to identify specific strengthening mechanisms in ECAP deformed Cu, also with respect to the two different paths of deformation applied.

11:35 AM

Effect of Pressure on the Final Grain Size after High Pressure Torsion: *Thomas Hebesberger*[1]; Reinhard Pippan[1]; Hein Peter Stüwe[1]; [1]Austrian Academy of Sciences, Erich Schmid Inst. of Matl. Sci., Jahnstraße 12, Leoben, Styria A-8700 Austria

It is now well established that severe plastic deformation is capable of reducing the grain size in metals to the submicrometer or even the nanometer level. Such ultra-fine grained materials show many promising properties such as increased strength and high strain rate superplasticity. High pressure torsion is one technique to produce this kind of material. The aim of this work was to study the effect of pressure on the development of the microstructure. It is obvious that high pressure influences the competition between deformation and fracture in such a way that far higher degrees of deformation compared with conventional deformation techniques are reachable. Apart from that it was recently reported that high pressure additionally may play an essential role in the refinement of the microstructure. In order to proof this assumption samples of recrystallized pure Copper were deformed by high pressure torsion at room temperature up to very large strains. The applied pressure has been varied in the range of 850MPa (minimum pressure to provide sufficient friction for applying the torsional momentum) to 8 GPa. After deformation the samples have been subjected to local orientation measurements by EBSD and OIM. The distribution of the crystallographic orientation has been investigated by scanning areas of 10 and 2µm in square with a step size of 200 and 100nm, respectively. These data were used to generate orientation maps providing information about the size of equally oriented areas. A new analysing method to treat the question for the average grain size and the misorientation distribution in a more quantitative manner, will also be presented. Independently of the pressure all samples show a very strong refinement of the microstructure reaching final grain sizes

of clearly below 500nm at a torsional strain of 250. Furthermore it will be clearly demonstrated that at comparable strains the microstructures of the samples deformed at higher pressures, have significantly smaller grain sizes.

11:50 AM
Microstructural Evolution of Cryomilled Nanocrystalline AL-TI-CU Alloy: *Zonghoon Lee*[1]; Rodolfo Rodriguez[2]; Enrique J. Lavernia[2]; Steven Nutt[1]; [1]University of Southern California, Dept. of Matls. Sci., Los Angeles, CA 90089-0241 USA; [2]University of California, Dept. of Chem. & Biochem. Eng. & Matls. Sci., Irvine, CA 92697-2575 USA

The microstructural evolution during processing of nanocrystalline powders and bulk of Al-Ti-Cu alloy was investigated using transmission electron microscopy and scanning electron microscopy. Grain refinement was achieved by cryomilling of elemental powders, and powders were consolidated by hot isostatic pressing (HIP) followed by extrusion to produce bulk nanocrystalline Al-Ti-Cu alloys. In an effort to enhance ductility and toughness, multi-scale structures were produced, which included nanocrystalline grains, elongated coarse grains of pure Al, and intermediate grains. Pure aluminum grains were elongated along the extrusion direction and formed coarse-grain bands comprised of sub-grains. Nanocrystalline second phases were distributed in the intermediate grains and nanocrystalline regions. The distribution and identity of these phases was achieved by analytical and high-resolution methods. Investigation of bulk tensile fracture specimens revealed unusual failure mechanisms and interactions between ductile coarse grains and nanocrystalline regions.

12:05 PM
The Microstructures and Compressive Deformation Behaviors of Nanocrystalline Al-5 at.% Ti Compacts Prepared by UHP-HP: *Kyoung Il Moon*[1]; Seung Chul Kim[1]; Kyung Sub Lee[1]; [1]Hanyang University, Matl. Sci. & Eng., Haengdang-dong, Seoungdong-ku, 17, Seoul 133-791 Korea

Two types of bulk nanocrystalline Al-5 at.% Ti alloys have been prepared by ultra high pressure hot pressing (UHP-HP) of mechanical alloyed powders and their mechanical properties have been investigated through compression tests. A full density was reached within 250 s at 120¡É under 4.8§è, in the specimen A120, and its microstructure was nanocomposite type with grain size less than 50 §¬. An abnormal grain growth was observed and some Al grains grew up over 500 §¬ in the specimens A300 prepared at 300¡É. The specimens A300 had a microstructure consisting of the large Al grains and nano-sized Al3Ti dispersoids. The compressive stress of the specimen A120 was 1010 §ç and that of the specimens A300 was 467 §ç at room temperature. The strength of the specimens A120 decreased greatly with increasing ductilities at 300-500¡É. The specimens A300 showed very small change in ductility and strength with temperature.

Shear Banding in Materials: Metallic Glasses

Sponsored by: ASM International: Materials Science Critical Technology Sector,
Program Organizers: Walter W. Milligan, Michigan Technological University, Metallurgical Engineering M, Houghton, MI 49931-1295 USA; Todd C. Hufnagel, Johns Hopkins University, Department of Materials Science and Engineering, Baltimore, MD 21218-2689 USA; Marc Andre Meyers, IMM, University of California, San Diego, CA 92093 USA; Hans Jorgen Roven, Norwegian University of Science and Technology, NTNU, Department of Metallurgy, Trondheim N-7034 Norway

Tuesday AM — Room: 303
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Walter Milligan, Michigan Technological University, Matls. Sci. & Eng., Houghton, MI 49931 USA; Hans Jorgen Roven, Norwegian University of Science and Technology (NTNU), Metall. Dept., Trondheim 7491 Norway

8:30 AM
Effects of Stress State on Shear Banding in Metallic Glass and Aluminum Alloys: *John J. Lewandowski*[1]; Peravudh Lowhaphandu[1]; [1]Case Western Reserve University, Dept. Matls. Sci. & Eng., Cleveland, OH 44106 USA

The effects of changes in stress state on the flow and fracture of a bulk metallic glass and a high strength aluminum alloy will be described. Both notched specimens as well as smooth specimens tested with superimposed pressure were used to vary the stress state in order to investigate the effects of such changes on the flow and fracture behavior. The flow behavior of the metallic glass will be compared to existing theories, while the effects of changes in stress state (i.e. notch root radius) on the magnitude of the toughness will also be presented for both materials.

8:50 AM
Shear Banding and Fracture Behavior of Zirconium Based Bulk Metallic Glasses and their Composites: *Katharine M. Flores*[1]; Reinhold Dauskardt[1]; [1]Stanford University, Dept. of Matls. Sci. & Eng., Stanford, CA 94305 USA

Plastic deformation of metallic glasses occurs by the formation of shear bands, however, the precise mechanisms of shear band formation and propagation remain uncertain. For example, the role of free volume magnitude and distribution, the effect of adiabatic heating, and the extent of local structural rearrangement, are under active investigation. In this presentation, we present direct evidence of free volume changes associated with plastic deformation of a zirconium based bulk metallic glass. Positron annihilation spectroscopy techniques have been utilized to study in detail the distribution and chemical environments of free volume sites, as well as the amount of free volume relative in shear bands compared to undeformed samples. In order for bulk metallic glasses to be used in structural applications, catastrophic failure due to the formation and propagation of a single dominant shear band must be prevented. A number of metallic glass matrix composites have been developed in an attempt to manage shear band formation and distribute plastic flow. The fracture and fatigue crack growth behavior of one such composite family utilizing selected ductile particle reinforcement phases has been examined and is compared with that of the monolithic alloy. The second phase blocks the propagation of shear bands and distributes the plastic deformation over a larger volume. This gives rise to extensive stable crack growth at stress intensities double the intrinsic toughness of the unreinforced bulk metallic glass. Implications for future composite microstructures are discussed.

9:10 AM
Nanoindentation Studies of Shear Banding in Fully Amorphous and Partially Devitrified Metallic Alloys: *A. L. Greer*[1]; S. V. Madge[1]; I. T. Walker[1]; J. R. Wilde[1]; [1]University of Cambridge, Dept. of Matls. Sci. & Metall., Pembroke St., Cambridge CB2 3QZ UK

Nanoindentation has been used to characterize shear banding in a wide variety of metallic glasses, ranging from bulk to melt-spun ribbon to sputtered thin films. The formation of shear bands is clearly evident as steps in the loading curve. Of particular interest is the effect of partial devitrification on the shear banding. When the crystallites are a few nm in diameter and smaller than the shear-band thickness, the shear banding is unaffected (although the hardness and modulus may increase significantly). On the other hand, with 100 nm crystallites (significantly larger than the shear-band thickness) the shear banding is no longer evident in the load displacement curves. The implications for deformation and hardening mechanisms in metallic glasses will be considered.

9:30 AM
Localized Free Volume Defects in Shear Bands in Metallic Glasses: Jing Li[1]; Zhong Lin Wang[2]; *Todd C. Hufnagel*[1]; [1]Johns Hopkins University, Matls. Sci. & Eng., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., E.J. Love Bldg., Rm. 163, Atlanta, GA 30332-0245 USA

Most recent theories of shear localization in metallic glasses use free volume as an order parameter. Direct experiment evidence of local variations in free volume has been lacking, however, due to the difficulty of adequately characterizing the structure of an amorphous material on length scales comparable to characteristic dimensions of shear bands (1-100 nm). In this work, we employ quantitative high resolution transmission electron microscopy to reveal the existence of increased free volume in plastically deformed regions of a Zr-based bulk metallic glass. The excess free volume exists as discrete subnanometer void-like defects throughout the metallic glass, with a much higher concentration of defects in plastically deformed regions than in undeformed material. The origins of these defects, and their influence on the mechanical behavior of metallic glasses, are discussed.

9:50 AM Break

10:10 AM
Mesoscopic Modeling of Deformation and Shear Localization in Metallic Glasses: *Mo Li*[1]; *Guang-Ping Zheng*[1]; [1]Johns Hopkins

University, Matls. Sci. & Eng., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

Deformation and shear localization in metallic glass is investigated using a newly developed method. This approach is an extension from the Landau theory, or phase field model, to include flow defects and dynamic process during the deformation process in metallic glass. In this talk, we describe the formulation of this new method and give examples from the modeling in crack initiation, propagation, and branching or shear localization in amorphous metals.

10:30 AM

Shear Band Nucleation and Evolution in Amorphous Metal Alloys: Effects of Crystallite Distributions: Christopher Mercer[1]; Patrick T. Anglin[1]; Rui H. Huang[1]; Zhigang Suo[1]; Anthony G. Evans[1]; *Winston O. Soboyejo*[1]; [1]Princeton University, Princeton Matls. Inst. & the Dept. of Mech. & Aeros. Eng., Eng. Quadrangle, Olden St., Princeton, NJ 08544 USA

This paper presents the results of a combined experimental and theoretical study of crack nucleation and growth in amorphous metal alloys. The paper compares the behavior of amorphous metal alloys with and without crystallite distributions. Scanning electron microscopy images of the crystallite distributions are presented before exploring the conditions for the nucleation and the propagation of shear bands in smooth and notched specimens. The experimentally-observed for crack nucleation have compared with those predicted by a theoretical model. The underlying mechanisms of crack nucleation and growth are also discussed for fracture under monotonic and cyclic loading. In particular, the paper examines the role that shear bands play in the nucleation and propagation of cracks in resistance-curve and fatigue experiments. The implications of the results are also discussed for the design of damage tolerant amorphous metal alloys.

10:50 AM

Characterization of Uniaxial Compressive Response of Bulk Amorphous Zr-Ti-Cu-Ni-Be Alloy: *Ghatu Subhash*[1]; Laszlo J. Kecskes[2]; Robert J. Dowding[2]; [1]Michigan Technological University, Mechl. Eng. & Eng. Mech., Houghton, MI 49931 USA; [2]US Army Research Laboratory, Aberdeen Proving Ground, MD 21005 USA

The uniaxial compressive response of bulk amorphous Zr-Ti-Cu-Ni-Be alloy, also called as Vitreloy-1, was investigated at quasistatic and high strain rates in the range of 10-3 s-1 and 103 s-1, respectively. The Vitreloy-1 specimens exhibited elastic response followed by catastrophic fracture along a narrow shear band. The ultimate strength of the specimens varied between 1,800 and 2,200 MPa irrespective of the strain rate and independent of the aspect ratio of the specimens. The quasistatically deformed specimens fractured into two or three large fragments. The fracture surfaces were relatively smooth and revealed well-developed and uniformly distributed veinal pattern. The dynamically loaded specimens, on the other hand, fractured into several fragments with relatively rough fracture surfaces containing non uniformly distributed and partially developed veinal patterns. Evidence of melting in the form of "liquid bubbles" was also observed along the cracks on the fracture surfaces of the specimens subjected to high strain-rate loading.

11:10 AM

A Study of Shear Band Propagation in Bulk Metallic Glasses using High-Speed Data Acquisition: *Wendelin Jane Wright*[1]; William D. Nix[1]; [1]Stanford University, Matls. Sci. & Eng., 416 Escondido Mall, Bldg. 550, Stanford, CA 94305-2205 USA

Plastic deformation in metallic glasses is caused by a dramatic viscosity reduction in single shear bands; however, it is still unclear whether the change in viscosity is caused by the creation of free volume or adiabatic shear band heating. Micrographs that support the adiabatic heating hypothesis have shown solidified droplets emanating from cracks adjacent to fracture surfaces; however, modeling of adiabatic shear band heating in metallic glasses suggests that the temperature rises caused by shear band propagation are not sufficient to cause flow localization. This modeling is highly sensitive to the time elapsed during the unloading segment of a single serration. Although previous measurements indicate that the time elapsed during shear band propagation is approximately 3 ms, the temporal resolution of the data is not sufficient to definitively measure the elapsed time. Current efforts utilizing strain gages and high-speed data acquisition will be discussed, and the data and implications for modeling will be presented.

Surface Engineering: Science & Technology II: Issues in Surface Engineering

Sponsored by: Materials Processing & Manufacturing Division, Surface Engineering Committee

Program Organizers: Ashok Kumar, University of South Florida, Department of Mechanical Engineering, Tampa, FL 33620 USA; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Gary L. Doll, The Timken Company, Canton, OH 44706 USA; D. S. Misra, Indian Institute of Technology-Bombay, Department of Physics, Powai, Mumbai 400076 India; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Kryoshi Yatsui, Nagoka University of Technology, Nagaoka, Niigata 840-2188 Japan

Tuesday AM — Room: 203
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Yip-Wah Chung, Northwestern University, Dept. of Matls. Sci. & Eng., Evanston, IL 60208 USA; J. S. Zabinski, Air Force Research Laboratory, WPAFB, Dayton, OH 45433 USA

8:30 AM Invited

Aspects of Surface Engineering in the Automotive Industry: *Yang-tse Cheng*[1]; [1]General Motors R&D Center, Matls. & Proc. Lab., MS 480-106-224, 30500 Mound Rd., Warren, MI 48090 USA

The manufacturing of an automobile is to a large extent the making of engineered surfaces on several length scales using a variety of processing techniques. In this presentation, we will summarize aspects of our recent work related to surface engineering. Examples include the preparation and characterization of nanocomposite thin films consisting of metal-metal and metal-ceramic materials; their tribological properties and potential application as low-friction and wear resistant coatings for electrical connectors will be discussed. The mechanical and tribological properties of thermal sprayed coatings for aluminum engine applications will also be discussed. Other examples include the investigation of thin films for sensors, battery electrodes, and catalysts. We will also review our work on modeling indentation measurements. Although significant progress has been made in surface engineering, it is still largely a "trial-and-error" process. We will illustrate, with examples, the needs for: (1) reducing the cost and improving the reproducibility of tribological coatings, (2) better characterization techniques for mechanical property measurements at the nano- and micrometer scales, and (3) design guidelines and models based on the fundamental understanding of the relationships between the structure, property, and performance of engineered surfaces.

8:55 AM

Micro to Nano-Small Research for Fuels and Combustion: *Christopher E. Bunker*[1]; Terrence R. Meyer[2]; Michael Brown[2]; Viswanath R. Katta[2]; Daniel A. Zweifel[3]; Ya-Ping Sun[3]; James R. Gord[1]; Barbara A. Harruff[1]; [1]Air Force Research Laboratory, Propulsion Direct., 1790 Loop Rd. N., Bldg. 490, Wright-Patterson AFB, OH 45433 USA; [2]Innovative Scientific Solutions, Inc., 2766 Indian Ripple Rd., Dayton, OH 45433 USA; [3]Clemson University, Chem. Dept., Howard L. Hunter Lab., Clemson, SC 29634 USA

The Fuels and Combustion Branches of the Air Force Research Laboratory have been actively investigating micro and nanoscopic materials as they pertain to fuel chemistry, sensor development, and MEMS devices. At these dimensions, surface interactions (e.g., surface chemistry, viscosity, and heat transfer) take on tremendous importance in understanding the macroscopic chemical and physical processes that occur. In some cases, current models used to predict chemical or physical properties are inadequate due to a lack of information concerning these surface effects. In this paper, we will detail some of our efforts to examine both physical and chemical processes occurring within and about micro and nanoscopic materials. Specifically, microfluidic results obtained using a LIF technique within a Si substrate Y-channel (~ 100 mm across and 30 mm deep), coupled with simple CFD calculations will be presented. In addition, spectroscopic results (steady-state and time-resolved) for a novel pyrene-dendrimer-modified carbon nanotube and model nanoparticles will be discussed.

9:10 AM

Preparation of Titanium Iron Thin Films by Ion-Beam Evaporation: T. Saikusa[1]; T. Suzuki[1]; H. Suematsu[1]; W. Jiang[1]; *K. Yatsui*[1]; [1]Nagaoka

University of Technology, Extreme Energy-Density Rsrch. Inst., 1603-1 Kamitomiokamachi, Nagaoka, Niigata 940-2188 Japan

Thin films of a titanium iron (TiFe) hydrogen-absorbing alloy were successfully prepared by ion-beam evaporation (IBE) for the first time. TiFe sintered pellets were bombarded by a proton beam with an energy of 1 MV (peak) and a current of 70 kA for 50 ns. The ablation plasma from the target deposited on both silica glass and silicon single crystal substrates. Results of X-ray diffraction revealed that neither titanium nor iron oxide phases were detected in the thin films on the glass substrate. Small amount of a $TiFe_2$ phase was found in the thin films. However, TiFe was a main phase in the thin films and essentially single phase TiFe thin films were successfully obtained. Although preparation of TiFe thin films have been attempted by many researchers, within the authors knowledge, no thin films consisting of crystallized TiFe phases were obtained.

9:25 AM

Nanomechanical and Nanotribological Characterization of Surface Engineered Materials: *Dehua Yang*[1]; Tony Anderson[1]; Rick Nay[1]; Thomas J. Wyrobek[1]; [1]Hysitron, Inc., Nanomech. Rsrch. Lab., 5251 W. 73rd St., Minneapolis, MN 55439 USA

Surface engineered materials have gained more and more attention and have undergone intensive studies because of their attractive low cost and ability to reduce the need for rare and noble materials. However, the shrinkage in dimension and size of materials has challenged the traditional mechanical and tribological testing approaches in many aspects. The lack of appropriate analysis and characterization methods has been a restraint to further breakthroughs in new material research and development. Further, the failure of materials, such as corrosion, oxidation and wear, originates from either the surface and/or the interface. Therefore, the appropriate characterization of surface and interface layers of materials is of significance to both processing control and failure analysis. In this paper, the current status and hot spots of nanomechanical and nanotribological characterization of surface engineered materials using nanoindentation based technology have been reviewed and discussed. Practical applications of different test methods and instruments are presented to backup the arguments.

9:40 AM

The Influence of Ion Energy Distributions on the Microstructure and Properties of Reactively Sputtered Titanium Oxide and Titanium Nitride Thin Films: *Chris Muratore*[1]; John J. Moore[1]; J. A. Rees[2]; Dan Carter[3]; Greg Roche[3]; [1]Colorado School of Mines, Adv. Coatings & Surface Eng. Lab. (ACSEL), Matls. Sci. Dept., 1500 Illinois St., Golden, CO 80401 USA; [2]Hiden Analytical, Ltd., 420 Europa Blvd., Warrington WA5 5UN UK; [3]Advanced Energy Industries, Inc., 1625 Sharp Point Dr., Ft. Collins, CO 80525 USA

Mass spectrometry and Langmuir probe techniques have been used to study inductively coupled plasma (ICP) enhanced reactive deposition and ion beam assisted deposition (IBAD) of titanium nitride and titanium dioxide. Both processes employ an unbalanced magnetron to generate the titanium flux, and a remote plasma source (ICP or ion beam) to excite the reactive gas before it enters the chamber. The use of either remote plasma source allows one to control the densities and energy distributions of the reactive species somewhat independently of the magnetron discharge. Experimental results reveal that measurements of intrinsic plasma parameters, coupled with a variety of materials characterization techniques, allow one to observe processing/structure relationships in thin film materials that would not otherwise be apparent.

9:55 AM Break

10:10 AM Invited

Interfacial Adhesion of Laser Clad Functionally Graded Materials: *J. T.M. De Hosson*[1]; Y. T. Pei[1]; V. Ocelík[1]; [1]University of Groningen, Dept. of Appl. Phys., Matls. Sci. Ctr. & The Netherlands Inst. for Metals Rsrch., Nijenborgh 4, Groningen 9747 AG The Netherlands

Performance of a coating depends on the adhesive strength of the coating to its substrate. In this paper samples of laser clad AlSi40 functionally graded materials (FGM) are described that are specifically designed for evaluating the interfacial adhesion of the FGM by tensile tests. In-situ microstructure observations in an FEG-ESEM (field emission gun-environmental scanning electron microscopy) during tensile tests reveal different failure modes of the FGMs and the substrates. Strain field mapping by the technique of digital imaging correlation demonstrates the gradient transition in tensile strain and related mechanical properties over the interface region and the softening effects in the heat-affected zones of the FGM tracks. The strengthening of the FGM is dominated by the size of the Al-halos around the particles, according to a dislocation pileup model.

10:35 AM

Optimization of Thermoelectric Properties in Boron Carbide Thin Films Prepared by Ion-Beam Evaporation: H. Suematsu[1]; I. Ruiz[1]; K. Kobayashi[2]; M. Takeda[2]; D. Shimbo[1]; T. Suzuki[1]; W. Jiang[1]; *K. Yatsui*[1]; [1]Nagaoka University of Technology, Extreme Energy-Density Rsrch. Inst., 1603-1 Kamitomiokamachi, Nagaoka, Niigata 940-2188 Japan; [2]Nagaoka University of Technology, Dept. of Mechl. Eng., 1603-1 Kamitomiokamachi, Nagaoka, Niigata 940-2188 Japan

Thin films of boron carbide ($B_{12+x}C_{3-x}$) with carbon content of $0<x<1$ have been successfully prepared by an pulsed ion-beam evaporation (IBE) method to optimize the thermoelectric properties. First, $B_{12+x}C_{3-x}$ bulks with nominal carbon contents of 3-x = 2.0 to 3.0 were synthesized by a spark plasma sintering. Secondly, the $B_{12+x}C_{3-x}$ thin films were prepared on glass substrates by the IBE without substrate heating or sample annealing. From X-ray diffraction results, the films consisted of a $B_{12+x}C_{3-x}$ phase and lattice parameters of the phase were comparable to that of the target. It was concluded that $B_{12+x}C_{3-x}$ thin films with various carbon contents were successfully prepared at room temperature. Finally, thermoelectric properties of the thin films were measured. The $B_{13.0}C_{2.0}$ thin film exhibits the highest power factor at room temperature among $B_{12+x}C_{3-x}$ samples reported.

10:50 AM

Formation of Gold Nanowires on MgO Surfaces: *Akira Ueda*[1]; Richard R. Mu[1]; Marvin H. Wu[1]; Don O. Henderson[1]; [1]Fisk University, Dept. of Phys., 1000 17th Ave. N., Nashville, TN 37208 USA

MgO has a cubic crystal structure with its easiest cleavage planes of (100). When we annealed a MgO single crystal at 1200°C, we observed steps running along two [110] directions on the (100) surface. By utilizing this property, the following attempt to fabricate gold nanowires was made: the MgO surface was tilt-polished toward one of two [110] directions with an angle of about 0.5 degree and then was annealed at a suitable temperature. Although the steps are not perfectly organized and there are some particulates on the surface, this surface was used as a substrate for gold deposition by the electron beam evaporation. Atomic force microscopy and optical absorption measurements were made for each annealing temperature to observe the gold nanowire formation. Polarized optical transmission spectra have been measured to detect the difference in absorption between parallel and perpendicular to the steps.

11:05 AM

Ion-Beam Synthesis of Superhard Materials by Dual Ion Implantation: Vladimir Vasilievich Uglov[1]; [1]Belarussian State University, Physl. Dept., pr.F.Scoriny 4, Minsk 220080 Belarus

The experimental results on formation of superhard materials (cubic boron nitride) after high dose subsequent ion implantation (SII) in iron of various combination of metalloids (N,B) are considered and discussed in work. The influence of SII by metalloids on the structure, element and phase composition, chemical bonds in Fe-B-N system is studied by methods of RBS, AES, CEMS, XPS and XRD. The formation of B-N chemical bond in Fe-B-N system is found by means of XPS and AES. These bonds are the nucleation centers of superhard c-BN phase as soon as incorporated boron and nitrogen concentration reaches 18 at.% and are stable at a temperature of up to 500°C. The threshold boron concentration of 28 at.% at which the transition of metastable $a\text{-}(Fe\text{-}B)_m$ amorphous phase into paramagnetic $a\text{-}(Fe\text{-}B)_p$ and boron clusterization take place was determined by CEMS and AES. The synthesis of superhard compound with covalent bonds is discussed from the point of view of formation of thermodynamically nonequilibrium solid solutions.

11:20 AM

A Hybrid Parameter for the Characterisation and Specification of Surface Texture: *P. Laxminarayana*[1]; V. S.R. Murti[1]; [1]Osmania University, Mechl. Eng., Univ. Col. of Tech.(Autonomous), Hyderabad, Andhara Pradesh 500 007 India

The inadequacy of specifying surface texture by the average height Ra of its roughness profile is universally acknowledged owing to its inability to identify the spatial characteristics.Auto correlation function (ACF) can discriminate between differing spatial structures by its decay properties. Treating the digitized values of ordinates from the roughness profile as a time series, its ACF can be computed. A combination of correlation length from the ACF and Ra, in the form of their ratio, leads to a hybrid parameter for the specification of the surface texture. Higher this index, denoted by Rx, better is the surface finish. This paper presents, such characterisation of two widely differing surfaces from turning and electrodischarge machining (EDM) and identifies the influence of dominant process parameters on the resultant surface texture. Charactically they are observed to be feedrate in turning and pulse current and frequency in EDM.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": New Smelter Projects and Optimization

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee, Copper, Nickel, Cobalt Committee, Lead and Zinc Committee, Non-Ferrous Metals Committee
Program Organizers: Robert L. Stephens, TeckCominco Metals, Ltd., Trail, British Columbia V1R 4L8 Canada; Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112 USA

Tuesday AM Room: 607
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: V. Ramachandran, 9650 E. Peregrine Place, Scottsdale, AZ 85262-1441 USA; Osamu Ishikawa, Ishikawa Consulting Inc., San Francisco, CA 94105-1895 USA

8:30 AM
Plant Commissioning and General Operations at Port Kembla Copper: *Robert West*[1]; [1]Port Kembla Copper Pty., Ltd., PO Box 42, Port Kembla, NSW 2505 Australia

Port Kembla Copper (PKC), a redeveloped copper smelting facility, was commissioned at the start of 2000. The site, located in Port Kembla, Australia, was a copper smelter from 1906 to 1995. PKC was formed in 1996 and the site was redeveloped from 1998 to 2000. The previous flow sheet consisted of a Noranda Reactor, Monsanto (MIC) acid plant, 2 Pierce Smith converters, 2 rotary anode furnaces, and a Wemec casting wheel. The redeveloped flow sheet replaced the Pierce Smith converters with a rotary holding furnace and Mitsubishi Converter. Additional capacity was added to the acid plant by the installation of another absorption tower and a Hitachi Zosen (HZ) unsteady state converter. Numerous difficulties were encountered during the commissioning such as multiple reactor tuyere line failures, absorbing acid tower refractory collapse, excessive acid plant SO_2 emissions, Noranda Reactor and Mitsubishi Converter process control problems, anode furnace gas handling and recycle material conveying problems. These problems are described along with the actions taken to overcome them. Process development is described particularly for the MIC and HZ converter. Plant operating data is included to show plant performance and process operating conditions such as HZ converter temperature profiles, furnace operating temperatures, grades, and minor element distributions.

9:05 AM
ISASMELT-Efficient & Cost Effective Smelting for MIM: *Chris Fountain*[1]; *Phil James Partington*[2]; [1]Mount Isa Mines, Ltd., Metall. Plants/Metall. Dept., PMB, Mount Isa, QLD 4825 Australia; [2]Mount Isa Mines Holdings, Ltd., MIM Proc. Tech., Level 2, 87 Wickham Terrace, Brisbane, QLD 4000 Australia

The Copper ISASMELT Furnace at Mount Isa, originally designed to treat concentrate containing 180,000 tonnes of copper per year, has over the past few months increased its yearly capacity to 270,000 tonnes. It offers a simple, robust and reliable process that is now operating with excellent results in Australia, USA, and India, with plants under construction in China and Germany. ISASMELT can also efficiently smelt other base metal concentrates and wastes. An ISASMELT furnace in Belgium treats mixed feeds, while secondary lead ISASMELT plants are operating in the UK and Malaysia. One of the most recent significant developments has been achieved in improved refractory life. The last refractory campaign at Mount Isa lasted two years, and the current campaign life prediction is considerably more. The ISASMELT process is ideally suited for existing plant upgrades and greenfield sites needing high production, good investment return and excellent environmental performance.

9:30 AM
The Noranda Process, the Technology of Choice for Emerging Economies in the 21st Century: *Cameron Harris*[1]; Phillip Mackey[2]; Yves Prevost[3]; Xainjian Guo[2]; [1]Norsmelt, 8475 ave. Christophe-Colomb, Bureau 2000, Montreal, Quebec H2M 2N9 Canada; [2]Noranda Technology Centre, 240 Hymus Blvd., Pointe-Claire, Quebec H9R 1G5 Canada; [3]Noranda, Inc., Horne Div., 101, ave. Portelance, Rouyn-Noranda, Quebec J9X 5B6 Canada

With the consolidation of the copper industry in the second half of the 20th century resulting in fewer plants treating ever increasing quantities and qualities of feed, the enormous flexibility of the Noranda Process gives it significant advantages over many competing technologies for the production of matte. Toll smelting, recycling, and secondaries treatment will become the cornerstone of profitable operations for all but the lucky few who have large captive mines associated with the smelter. Consequently, the ability to respond to changing market conditions without significant metallurgical or operational upsets will be of considerable commercial advantage. The inherent stability of this bath-smelting process leads to tight matte grade control, while the bath turbulence leads to rapid consumption of feeds with widely varying physical characteristics. Low capital and operating costs combined with a layout suitable for retrofit into existing converter aisles makes it a sensible choice for parts of the world still experiencing the aforementioned consolidation, which is essentially complete in fully developed economies such as the United States of America and the European Union. This paper will discuss features of the Noranda process compared with other technologies and its place in the future global copper production industry, which is set to become increasingly competitive and aggresive.

9:55 AM
From Autogenous Sulfide Smelting to the Production Network–Outokumpu Flash Technology as Trendsetter in Copper Production: *Ilkka V. Kojo*[1]; Pekka Hanniala[1]; [1]Outokumpu Technology Oy, PO Box 862, Riihitonuntie 7E, Espoo FIN-02201 Finland

The falling real price of copper metal and changes in treatment and refining charges have been characteristic to the copper industry during the previous decades. Outokumpu's continuous development of Flash Smelting Technology and increased productivity of the smelters based on these improvements may be one of the basic causes for these trends. From the early beginning of Flash Smelting Technology, it has been characterized by low operating costs and low emissions. The vision has been to develop the technology towards Direct Blister Flash Smelting, in which both smelting and converting are carried out in one furnace thereby making it possible to reduce the number of unit operations and thus further lower both operating and investment costs. The merger of large copper production companies that has taken place in the last few years is a sign of a new age in the copper production business. Now the aim is to increase the productivity of copper producton not just by utilizing economies of scale but also by increasing integration and optimization of the whole production chain from mine to refinery. This also means increasing win-win based cooperation between companies. The Production Network concept based on the Flash Converting process enables existing smelters to decrease operating costs and expand smelting capacity with minimum investment and at the same time decrease emissions. The new structure in ownership and partnership between copper production companies makes it easier to adopt the Production Network concept to both new and existing plants, where there exists a lot of built-in capacity. This paper presents Outokumpu's prediction on how the new options for the smelter will contribute to further changes in the structure of copper production. The visions are illustrated by giving examples of how the combination of matte production, Flash Converting, and Direct-to-Blister Flash smelting could be adapted in existing and new smelters.

10:20 AM Break

10:35 AM Invited
Chagres Smelter 1995-2000: From the Modernization to the Optimization: *Roberto M. Parada*[1]; Ricardo Bonifaz[1]; [1]Compañía Minera Disputada de Las Condes, Chagres Smelter, Pedro de Valdivia 291, Santiago, Chile

Modernization of Chagres Smelter shows its central issue in the Reverberatory furnace replacement by the Flash Outokumpu Smelting Technology. Nowadays, this smelting unity has achieved a 40% over design performance. The landmarks of this program of this update program described: a) Start Up (1995-1996): The main restrictions that didn't allow to reach the desired production curve are showed, in front of the implemented solutions. b) Reaching the design performance (1997-1998). Bottlenecks for smelting and production rates, restrictions for plant availability and the undertaken measures to overcome those difficulties are examined. c) Optimization (1999-2000): The main factors that led to the achievement of a historical annual throughput of 148.000 tons of blister, at 97.8% recovery in 1999 are analyzed. The migration from blister to anode copper production, obtaining a 98% approval for four different anode shapes, in parallel production, during 2000, is also presented.

11:00 AM
Inco's New Anode Furnaces and Anode Casting Shop at the Copper Cliff Smelter: *Bruce Bichel*[1]; [1]Inco, Ltd., Ontario Div., Copper Cliff Smelter, Copper Cliff, ON P0M 1N0 Canada

For the last 70 years, molten blister copper has been transferred by rail in hot metal cars from the Copper Cliff Smelter to the Copper Cliff Copper Refinery (ex Ontario Refining Company) where it was processed in two reverberatory anode furnaces by poling with logs, cast on four wheels, and electrorefined. This practice was modernized in 2001 with the installation of two rotary anode furnaces at the Copper Cliff Smelter which employ steam and natural gas for reducing and which provide a significant reduction in emissions. A new casting shop with a single anode casting wheel is used to cast 'regular' and 'stripper' constant weight anodes which are transported by truck to the Copper Cliff Copper Refinery for anode preparation and electrorefining. This paper presents an overview of the copper circuit at the Copper Cliff Smelter and a description of the CDN$30MM Capital project.

11:25 AM
An Update on Peirce-Smith Converter Operations at Sterlite Copper: *V. K. Handa*[1]; K. Anand[1]; D. K. Pagar[1]; V. Muthupandi[1]; [1]Sterlite Copper, SIPCOT Industrial Complex, Tuticorin 628002 India

Sterlite Industries (I) Ltd. started its copper smelter in 1996 and worked through the common problems associated with smelter start-ups. Specific issues in the converter area included converter blower IGV problems, punching machine reliability, converter spitting, back collar burning, and short converter campaign life. With a very young and dedicated team of engineers and technicians, smelter management assigned teams of three to four members to work scientifically and systematically through each major problem area to recommend and implement appropriate solutions. The converter mouth was redesigned with respect to tuyere and metal height to eliminate spitting and back collar burning. The air line was changed in places to reduce the pressure drop. A new back-up compressed air line and vessel was installed to prevent tuyere blockage in the event of a blower trip or power failure. The converter mouth liners and bricking around the mouth was modified to eliminate metal leaking through the pouring lip. Modifications to the punching machines were made to address specific problems and preventative maintenance practices were further streamlined and reinforced to ensure 100% availability of hot metal cranes, converter blowers, punching machines, and ID fans. Matte availability and matte temperatures were improved by streamlining the upstream ISA and Rotary Holding furnace areas. Implementation of a detailed training program for the converter operators significantly improved the operation and morale. Water-cooled double hoods and a third converter were installed as part of the smelter modernization plan. The new hoods dramatically improved hygiene in the crane aisle while the addition of the third converter posed new challenges for the same motivated group of crews with respect to flip flop operation to keep the idle converter hot and to maximize instack time, managing crane/ladle logisitics, and managing revert generation. This paper describes the converter operation's journey from the initial days of long blowing times and large revert accumulations to present operations with two converter flip flop operation, and also describes the team work, constant focus on increasing productivity and reducing costs, and benchmarking.

Water Vapor Effects on Oxidation of High-Temperature Materials: Alloys - II

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee
Program Organizers: Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Karren L. More, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6064 USA; Elizabeth J. Opila, NASA Glenn Research Center, Cleveland, OH 44135 USA

Tuesday AM — Room: 305
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: M. Schütze, Dechema e.v., Karl-Winnacker-Inst., Theodor-Heuss-Allee 25, Frankfort am Main 60486 Germany; D. J. Young, University of New South Wales 2052 Australia

8:30 AM
Combined Effects of Silica and Steam on Oxidation Behavior of Carbon Steels: *B. Gleeson*[1]; R. K. Singh Raman[2]; D. J. Young[3]; [1]Iowa State University, Matls. Sci. & Eng., 3157 Gilman Hall, Ames, IA 50011 USA; [2]Monash University, Sch. of Phys. & Matls. Eng., Clayton Australia; [3]The University of New South Wales, Sch. of Matls. Sci. & Eng., Sydney Australia

The oxidation behaviors of two Si-containing steels (Si contents: 0.51 and 2.2 wt.%) and a Si-free steel were studied in air and steam-containing environments at 730, 850, 935 and 1050°C. The oxidation kinetics of the silicon-free steel were parabolic and similar in the air and air + steam environments, with the scale products also being similar. By contrast, the oxidation kinetics of the Si-bearing steels in the steam + air environment were greater than in air alone. This behavior was attributed to increased scale adherence in the presence of steam. The oxidation kinetics of all the steels were much slower in a pure steam environment.

9:00 AM
Inner Scale Formation and its Effect on Oxidation Behavior of Fe-Based Si, Cr, or Al Alloys in Atmosphere Containing Water Vapor: *T. Narita*[1]; M. Fukumoto[1]; S. Hayashi[1]; [1]Hokkaido University, Grad. Sch. of Eng., Kita-Ku Kita-13 Nishi-8, Sappora 060-8628 Japan

Oxidation behavior of Fe-based alloys containing Si, Cr, or Al was investigated in atmospheres with various contents of N_2, O_2, and H_2O. The oxidation kinetics and scale structure were determined using TGA, EPMA, XRD, and TEM observations. There is no effect of water vapor on oxidation of pure iron, whereas the Fe-based alloys showed an s-shaped corrosion versus time curve. The rapid oxidation was triggered by a formation of inner scale formation, which generally consisted of a porous mixture of wustite and spinel oxides of Fe, Si, Cr, or Al. A scale structure similar to those in water vapor atmospheres was found to be formed in air for the Fe-3mass%SiO_2 alloy. In this paper, inner scale formation and its effect on an acceralated corrosion in water vapor atmospheres is discussed and analyzed in terms of internal oxidation phenomena.

9:30 AM
Effect of Water Vapor on Oxide Scale Microstructure: *B. A. Pint*[1]; K. L. More[1]; P. F. Tortorelli[1]; I. G. Wright[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., Bldg. 4500S, MS 6156, Oak Ridge, TN 37831-6156 USA

The presence of water vapor in the environment can have a substantial effect on the high temperature oxidation behavior of many alloys. However, the mechanistic role of water vapor is poorly understood. In order to provide additional microstructural information on the effect of water vapor, the scales formed on alumina- and chromia-forming alloys were examined after exposure to air and air plus 10vol.% water vapor. Characterization included cross-sectional metallography, scanning electron microscopy and cross-sectional transmission electron microscopy. In general, the scales examined after short oxidation times (100-500 h) were not significantly different with and without water vapor. On a type-321 austenitic stainless steel specimen, regions that formed a thin Cr-rich oxide were thinner after exposure to water vapor at 700°C. The microstructure of alumina scales formed on PM2000 (FeCrAl) and Haynes 214 (NiCrAl) showed only minor differences with the addition of water vapor at 1000° and 1100°C, respectively.

10:00 AM
Degradation of Fe-Cr-Al-RE Foils in Air and Combustion Gas Atmospheres: *J. E. Oakey*[1]; N. J. Simms[1]; J. R. Nicholls[2]; R. Newton[2]; J. Wilber[1]; A. Encinas-Oropesa[1]; J. F. Norton[1]; [1]Cranfield University, Power Generation Tech. Ctr., Cranfield, Bedfordshire MK 43 0AL UK; [2]Cranfield University, Sch. of Indl. & Mfg. Sci., Cranfield, Bedfordshire MK43 0AL UK

The degradation of thin foils of a group of nominally Fe-20wt%Cr-5wt%Al-RE alloys has been investigated in dry air (20% oxygen-balance nitrogen) and simulated natural gas combustion gases in the temperature range 950-1300°C for periods up to 2500 hours.

10:30 AM
The Effect of Water Vapor on the Passive Layer Breakdown of Fe-Al Based Alloys: J. R. Regina[1]; J. N. DuPont[1]; A. R. Marder[1]; [1]Lehigh University, Matls. Sci. & Eng., Whitaker Lab., 5 E. Packer Ave., Bethlehem, PA 18015 USA

Wastage of waterwall boiler tubes due to high temperature corrosion has been a serious issue for fossil fueled power companies for several years. The gas atmospheres in these boilers have been shown to contain water vapor. Although various authors have reported that additions of water vapor to high temperature corrosion environments accelerate corrosion, the role of water vapor on increasing the corrosion rate of boiler alloys and coatings is not very well understood. In order to better understand these effects, multiple Fe-Al based alloys with and without chromium additions were tested in three different multi-component corrosive gases with and without water vapor. The three gases were a sulfidizing gas, a mixed oxidizing/sulfidizing gas, and

an oxidizing gas and were used for corrosion tests at 500°C for 100 hours. Thermogravimetric testing showed that the corrosion kinetics increased when water vapor up to 6% was added to the atmospheres. The surfaces of the exposed samples were considered carefully to determine if the addition of water vapor changed the morphology of the corrosion products and more importantly affected the passive layer. It has previously been shown that the formation of nodules can be caused by the inability of the passive layer to re-heal itself after a defect forms allowing fast growing non-protective corrosion products to externally grow from the surface. In this study, the amount of external nodules that formed on the surface of the alloys was seen to increase with the addition of water vapor. An increase in the amount of external nodules present due to additions of water vapor gives an indication that water vapor increases the passive layer breakdown. The water vapor effect may be due to passive layer nucleation and growth that results in an increase in the passive layer defect structure producing fast diffusion paths for corrosion species to attack the underlying substrate.

11:00 AM

The Surprising Reduction of Cu2O by Water Vapor: *Judith C. Yang*[1]; Mridula Dixit Bharadwaj[1]; Guangwen Zhou[1]; [1]University of Pittsburgh, Matls. Sci. & Eng., 848 Benedum Hall, Pittsburgh, PA 15261 USA

We are investigating the effect of moisture on the initial oxidation stages of copper by in situ ultra-high vacuum transmission electron microscopy (UHV-TEM). Single crystal 99.999% pure 600-1000 Å Cu films were cleaned in situ by annealing in methanol vapor that reduces the copper oxides to copper. Water vapor (99.9% purity) was introduced directly into the TEM, at a partial pressure of 4X10-4 torr and 350°C. We have observed an unexpected surface phenomenon where H2O reduces Cu2O, which is contrary to the bulk thermochemical data. We have examined the possible artifacts for Cu2O reduction in wet oxidizing conditions by considering e-beam effect, carbon contamination and residual gases and conclude that it is unlikely that these play a role in the surprising reduction of copper oxide by water vapor. We speculate the interfacial and surface energies explain the discrepancy between bulk thermochemical data and our experimental observations.

11:30 AM

The Effect of Water Vapor on the Oxidation Resistance Behavior of Iron Aluminide CVD Coatings: Y. Zhang[1]; *B. A. Pint*[2]; J. A. Haynes[2]; P. F. Tortorelli[2]; I. G. Wright[2]; [1]Tennessee Technological University, Ctr. for Mfg. Rsrch., 115 W. 10th St., Brown Hall, Rm. 231, Cookeville, TN 38505-0001 USA; [2]Oak Ridge National Laboratory, Metals & Cer. Div., Bethel Valley Rd., PO Box 2008, Oak Ridge, TN 37831-6156 USA

The oxidation behavior of iron aluminide coatings produced by chemical vapor deposition (CVD) is being studied in the temperature range of 700-800°C with 10vol% of H2O vapor. By using a well-controlled laboratory CVD procedure, the coatings are uniform in composition, purity and microstructure. A typical ferritic steel, Fe-9Cr-1Mo, and an austenitic stainless steel, 304L (nominally Fe-18Cr-9Ni), were coated to examine differences in the two types of substrates. For both substrates, the as-deposited coating consisted of a thin (<5 microns), Al-rich layer above a thicker (30-50 microns), lower Al content layer. Besides the coated and uncoated Fe-9Cr-1Mo specimens, cast Fe-Al model alloys with similar Al content (13-20at%) to CVD coatings were included for comparison. The specimens were cycled to 500 1-h cycles at 800°C and 1000 1-h cycles at 700°C, respectively. The CVD coating specimens showed excellent performance in the water vapor environment at both temperatures, while the uncoated alloys were significantly attacked. Detailed microstructure characterization was conducted. These results suggest that a thin aluminized coating can substantially improve resistance to water vapor.

Advances in Metallic Glasses: Glass Crystallization - II

Sponsored by: Electronic, Magnetic & Photonic Materials Division, *Program Organizers:* K. F. Kelton, Washington University, Department of Physics, St. Louis, MO 63130 USA; A. L. Greer, University of Cambridge, Department of Materials Science & Metallurgy, Cambridge CB2 3QZ UK; Krishna Rajan, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering, Troy, NY 12180-3590 USA

Tuesday PM — Room: 212
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: John H. Perepezko, University of Wisconsin, Dept. of Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706; Takeshi Egami, University of Pennsylvania, Dept. of Matls. Sci. & Eng., LRSM, 3231 Walnut St., Philadelphia, PA 19104-6272 USA

2:00 PM Invited

Quasicrystals from Glass Devitrification: *Akihisa Inoue*[1]; Junji Saida[2]; [1]Tohoku University, Inst. for Matls. Rsrch., 2-1-1 Katahira, Aoba-ku, Sendai 980-8577 Japan; [2]Japan Science and Technology Corporation, Inoue Superliquid Glass Proj., 2-1-1 Yagiyamaminami, Taihaku-ku, Sendai 982-0807 Japan

It is well known that the icosahedral phase in the Zr-based system precipitates in the devitrification stage of bulk glassy Zr-Al-Ni-Cu alloys with a large supercooled liquid region before crystallization, though the precipitation is dominated by the existence of oxygen element. More recently, we have noticed that the icosahedral quasicrystal is formed during devitrification of Ti-, Zr- and Hf-based bulk glassy alloys containing additional elements which are immiscible against the other constituent elements. Consequently, the clarification of the precipitation behavior is important for understanding the origin for high thermal stability of the supercooled liquid leading to the formation of bulk glassy alloys in their special alloy systems. In this paper, we aim to review glassy alloys where the icosahedral quasicrystal is formed in the devitrified and as-solidified states, and microstructure, precipitation behavior, mechanical strength and electrical properties of the quasicrystalline base alloys in Ti-, Zr- and Hf-based systems.

2:30 PM

Phase Transformations and Mechanical Behavior of (Hf, Zr)-Ti-Cu-Ni-Al Bulk Metallic Glasses: *Xiaofeng Gu*[1]; Cang Fan[1]; L. J. Kecskes[2]; S. T. Szewczyk[2]; Todd C. Hufnagel[1]; [1]Johns Hopkins University, Dept. of Matls. Sci. & Eng., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA; [2]Army Research Laboratory, Weapons & Matls. Direct.

A series of bulk glass-forming alloys of composition $(Hf_x,Zr_{1-x})_{52.5}Ti_5Cu_{17.9}Ni_{14.6}Al_{10}$ and $(Hf_x, Zr_{1-x})_{57}Ti_5Cu_{15.4}Ni_{12.6}Al_{10}$ with x=0-1 have been prepared. The substitution of Hf for Zr was observed to generally reduce the glass-forming ability of these alloys. The alloys show similar crystallization behavior, including phase separation in the supercooled liquid state, although we observe an intermediate phase in the Hf-rich glasses ($Al_{16}Hf_6Ni_7$) that is not observed in the Zr-rich glasses. The flow strength under quasi-static uniaxial compression increases linearly with increasing Hf content, but the plastic strain to failure is maximized at intermediate compositions. Most of the differences in behavior between the Zr- and Hf-rich glasses can be rationalized in terms of the higher melting point of Hf, which causes the glass transition and crystallization temperatures to be higher in the Hf-rich glasses. The effect of Hf on the mechanical behavior is also discussed.

2:50 PM

Atomic Motion and Crystallization in Bulk Metallic Glasses: *Xiaoping Tang*[1]; Yue Wu[1]; Jan Schroers[2]; William L. Johnson[2]; [1]University of North Carolina, Phys. & Astron., CB 3255, Chapel Hill, NC 27599 USA; [2]California Institute of Technology, Keck Lab. of Eng. Matls., MC 138-78, Pasadena, CA 91125 USA

Nuclear magnetic resonance was used to investigate the atomic transport in the glassy and supercooled liquid states for the Pd-Ni-Cu-P, Pd-Ni-P, Pd-Cu-P, and Zr-Ti-Ni-Cu-Be bulk metallic glass systems as well as the crystallization process and the related crystalline states. Especially, the atomic motion in the Pd-Ni-Cu-P and Pd-Ni-P glasses was measured from below Kauzman temperature to above the glass transition temperature. The rate of the measured atomic motion was compared with the glass formability and thermal stability for systems with similar chemical compositions. The study shed light on the nature of the atomic transport in both the glassy and supercooled liquid states of bulk metallic glasses.

3:10 PM
Polymorphous Crystallization in an Al-Riched Amorphous Al-Fe Alloy Produced by Mechanical Alloying: *F. Zhou*[1]; R. Lüeck[2]; K. Lu[3]; E. J. Lavernia[1]; M. Rühle[2]; [1]University of California-Irvine, Dept. Chem. & Biochem. Eng. & Matl. Sci., 916 Engineering Tower, Irvine, CA 92697-2575 USA; [2]Max-Planck-Institut für Metallforschung, Seestr. 92, Stuttgart D-70174 Germany; [3]Chinese Academy of Sciences, Inst. of Metal Rsrch., Shenyang 110015 China

A metastable $Al_{90}Fe_{10}$ (at.%) alloy that consisted of fcc Al nanocrystals in an amorphous matrix, was produced by mechanical alloying of a mixture of Al and Fe elemental blends. The thermal induced crystallization behavior in the alloy was characterized in detail. A polymorphous crystallization of the amorphous matrix to a metastable crystalline Al_6Fe was observed. Thermodynamic analysis in terms of a hypothetical free energy diagram revealed the nature of the polymorphous crystallization in the amorphous composite. The influence of the pre-existing crystals on crystallization was not morphologically distinctive. The observed linear kinetics law behavior in combination with an Avrami exponent value of 2.7±0.1 suggests that the growth of a fixed nuclei population at a constant rate in three dimensions governs crystallization. The activation energy for crystallization was found to be close to the activation energy for the diffusion of Fe atoms in Al.

3:30 PM Break

3:50 PM
Pressure Effects on Formation, Properties and Glass Transition of Zr-, Pd- and Nd-Based Bulk Metallic Glasses: *Wei Hua Wang*[1]; [1]Chinese Academy of Sciences, Inst. of Phys., PO Box 603, Beijing 100080 China

The acoustic, thermal and elastic properties and their pressure dependence of various Zr- and Pd-based bulk metallic glasses (BMGs) were studied. The equation of state of these BMGs were obtained for the first time and compared to that of other kinds of glasses and crystalline solids. The microstructural characteristics, phase transformation and glass transition of the best glass forming alloys $Zr_{41}Ti_{14}Cu_{12.5}Ni_{10}Be_{22.5}$ and Pd39Ni10Cu30P21 BMGs under high pressure were investigated by using high resolution transmission electron microscope (HRTEM), differential scanning calorimeter (DSC), X-ray diffraction (XRD), ultrasonic study and density measurements. High pressure annealing below glass transition temperature Tg, results in a microstructural transformation from short-range order to medium-range order in the BMGs, the BMG with medium-range order structure exhibits different structural, thermal and acoustic properties. High-pressure annealing can also induce phase separation and nanocrystallization below Tg and a composite with fine nanocrystallites in amorphous-matrix in the supercooled liquid region. The nanocrystallization is pressure-assisted and complete nanocrystallization can be obtained by annealing under pressure of 5 GPa at 723K. The mechanism for the pressure-assisted nanocrystallization is discussed.

4:10 PM
Nucleation Mechanisms in Al-Si Alloys: *Peter Schumacher*[1]; Brian McKay[1]; [1]University of Oxford, Dept. of Matls., Parks Rd., Oxford, Oxfordshire OX1 3PH UK

Heterogeneous nucleation on commercial grain refiner addition of Al3Ti and TiB2 particles was studied in an Al80Ni10Cu8Si2 glass. Melt-spun ribbons contained hexagonal TiB2 particles within a glassy matrix. On TiB2 particles copious nucleation of Al was on basal faces of TiB2 particles. A distinct orientation relationship between the boride and the Al was found with the close packed planes and directions being parallel. Intermetallic particles nucleated on prism faces of boride particles. At short holding times before melt-spinning nucleation of Al2Cu was observed while holding for 30 min resulted in the formation of TiSi2. No nucleation of Al was observed on basal faces of boride when TiSi" nucleated on prism faces. The implication of the observed nucleation mechanism are discussed with respect to industrial casting practice in particular to Si-poisoning.

4:30 PM
Crystallization of a Beryllium-Boron Metallic Glass: *Alan F. Jankowski*[1]; [1]Lawrence Livermore National Laboratory, CMS-MSTD, PO Box 808, MS L-352, Livermore, CA 94551-9900 USA

It's known from prior studies of evaporation and sputter deposition that the grain size of nominally pure beryllium can be dramatically refined through the incorporation of specific metal impurities like iron. Recently, we have shown that the addition of boron serves as a glassy phase former in beryllium. Metallic glass formation is observed for boron concentrations greater than 11%, that is, beyond the eutectic seen in the binary alloy phase diagram. The effects of boron additions on the structure of beryllium are characterized with diffraction, x-ray absorption spectroscopy, and transmission electron microscopy. We now report on the crystallization of the beryllium-boron glass during annealing as observed in-situ with electron microscopy. A polycrystalline structure appears to be formed with a grain size less than 100 nm. This work was performed under the auspices of the US Department of Energy by University of California Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

4:50 PM
Structure and Properties of Zr-Ta-Ci-Ni-Al Bulk Glass-Forming Alloys: *Ryan Ott*[1]; Cang Fan[1]; Jing Li[1]; *Todd C. Hufnagel*[1]; [1]Johns Hopkins University, Matls. Sci. & Eng., 102 Maryland Hall, 3400 N. Charles St., Baltimore, MD 21218 USA

The mechanical behavior of metallic glasses is quite sensitive to composition and preparation conditions. For instance, we have recently shown that bulk amorphous $Zr_{59}Ta_5Cu_{18}Ni_8Al_{10}$ shows significantly enhanced plastic strain to failure, relative to other closely related compositions. In the present work, we explore the crystallization behavior and mechanical properties of $(Zr_{59}Cu_{18}Ni_8)_{90-x}$ -Ta_xAl_{10} as a function of alloy content. The crystallization behavior is similar to other Zr-based alloys, with the precipitation of an icosahedral quasicrystalline phase followed by formation of $NiZr_2$ and $CuZr_2$. While the flow strength of the alloys under uniaxial compression is essentially independent of composition, the plastic strain to failure shows a maximum around 5% Ta. The enhanced plastic strain to failure appears to be associated with increased structural order on the 1-2 nm length scale.

Alumina and Bauxite: Bayer Process Chemistry

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Jacques M. Mordini, Aluminium Pechiney, Gardanne, Cedex 13541 France; Steve Rosenberg, Worsley Alumina Pty, Ltd., Process Chemistry Group, Collie, West Australia 6225 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday PM — Room: 609
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: TBA

2:00 PM
12 Years of Experience with Wet Oxidation: *André Lalla*[1]; Rolf Arpe[1]; [1]Aluminium Oxid Stade GmbH, Johann-Rathje-Köser-Strasse, Stade 21683 Germany

In 1980 AOS started the wet oxidation process in order to reduce the organic Carbon level in their liquors. After an explosion in 1982, AOS redesigned the safety system, oxygen analyzers and reactor system. In 1989 AOS restarted wet oxidation with satisfying results. This paper will present the development of the org. C level and the benefits of a low org. C content. This is illustrated by the higher throughput of filtering operations, precipitation yield, brightness of hydrate, etc.

2:25 PM Cancelled
Some Options for Removal of Sulphate from Bayer Liquors: *Wayne Tichbon*

2:50 PM Cancelled
Influence of K2O in Spent Liquor on the Bayer Process of Diasporic Bauxite: *Yin Zhonglin*

3:15 PM Break

3:35 PM
Boehmitic Reversion in a Double Digestion Process on a Bauxite Containing Trihydrate and Monohydrate: *Jean-Michel Lamérant*[1]; [1]Aluminium Pechiney, Alumina Tech., BP 54, Gardanne 13541 France

Gardanne's Bayer Plant has been modified to process Boké bauxite with a double digestion. This bauxite gives a ratio: soluble alumina in the first digestion (140-150°C) to soluble alumina in the second digestion (225-240°C) close to 6, but this ratio is lower in industrial exploitation. The first digestion dissolves the trihydrate but causes simultaneous precipitation of monohydrate due to high boehmite content of muds (about 30%.) This reaction continues during muds settling, without disturbing their separation. The effects of factors such as alumina super saturation, temperature and residence time have been studied in lab experiments. Above a given super saturation threshold, boehmitic

reversion accelerates quickly. Plant process parameters have been optimized according to this situation.

4:00 PM
Kinetic and Thermodynamic Measurements of Hydrocalumite Formation in Bayer Liquors: *Steven P. Rosenberg*[1]; Jarrod H.N. Buttery[2]; Vincent A. Patrick[2]; [1]Worsley Alumina Pty, Ltd., Proc. Chem. Grp., PO Box 344, Collie, Western Australia 6225 Australia; [2]Central Chemical Consulting Pty, Ltd., PO Box 40, Karrinyup, Western Australia 6921 Australia

Recent work at Worsley Alumina Pty Ltd has shown the importance of the C4A-type calcium aluminate species and their subsequent reactions in controlling causticisation performance and calcium solubility in the Bayer process. Many of these reactions tend to occur concurrently, making detailed investigations of the kinetics and thermodynamics of the system difficult. However, new causticisation technology developed at Worsley can be applied to inhibit some of these reactions, allowing fundamental thermodynamic information of the formation of intermediates in causticisation to be obtained. Calorimetric experiments showed that, after addition of slaked lime to Bayer liquor, the exothermic reaction to form hydrocalumite occurred within seconds and was fully complete within a few minutes. In this paper, a practical method to prepare essentially pure hydrocalumite and measurements of the speed of its formation are reported. Thermodynamic data for both hydrocalumite formation and its decomposition to pure $CaCO_3$ are given.

Aluminum Reduction Technology: Process Control

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Martin Segatz, VAW Aluminum AG, D-53117, Bonn Germany; Halvor Kvande, Norsk Hydro ASA, Oslo N-0240 Norway; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday PM — Room: 6B
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Geoffry Bearne, Comalco, Australia

2:00 PM
Development of a State Observer for an Aluminum Reduction Cell: *Philip L. Biedler*[1]; Larry E. Banta[2]; Congxia Dai[2]; Ric Love[3]; Chuck Tommey[3]; Jan Berkow[4]; [1]West Virginia University, Mechl. & Aeros. Eng. Dept., 401 Newton Ave., Apt. 102, Morgantown, WV 26506 USA; [2]West Virginia University, Mechl. & Aeros. Eng. Dept., PO Box 6106, Morgantown, WV 26505 USA; [3]Century Aluminum, Ravenswood, WV USA; [4]Applied Industrial Solutions, Morgantown, WV USA

Developing a controller that will maintain an aluminum reduction cell near its optimal state is a difficult process. This is a result of the nonlinearity of the system, the multiple time scales that must be accounted for, and the lack of real time measurements for important process variables, such as alumina concentration, bath temperature, and bath ratio. A nonlinear mathematical model has been developed that simulates the electrical, thermal, fluid, and chemical processes occurring within the cell. This model can be used as a type of state observer to estimate unmeasured process variables from the measurement of available parameters, such as the cell voltage and line current. The state observer will be incorporated into a control scheme that would maintain the reduction cell closer to its optimal state. This paper will outline the development of the mathematical model and its ability to track the measured outputs of a reduction cell.

2:25 PM
Decomposition of Aluminum Cell Voltage Signals: *Larry E. Banta*[1]; Philip L. Biedler[1]; Congxia Dai[1]; Ric Love[2]; Chuck Tommey[2]; Jan Berkow[3]; [1]West Virginia University, Mechl. & Aeros. Eng. Dept., PO Box 6106, Morgantown, WV 26505 USA; [2]Century Aluminum, Ravenswood, WV USA; [3]Applied Industrial Solutions, Morgantown, WV USA

The voltage across an aluminum reduction cell is composed of signals from numerous concurrent processes, some of which are independent and some of which are coupled. Alumina concentration is a key process control variable, and is generally extracted from the voltage and current signals by extreme low-pass filtering, and by suspension of all anode movements and alumina feed operations during the measurement period. This strategy is fairly effective, but leads to suboptimal cell operation most of the time, since the controller constantly "hunts" back and forth across the optimal setpoint. Research at West Virginia University and Century Aluminum seeks to improve upon the traditional strategies by incorporating knowledge about the process and prior cell control actions in the data analysis operation. This paper discusses the methods being developed and their application to the control of Hall-Heroult reduction cells.

2:50 PM
Computer-Aided System for Pre-Set Voltage Control: *S. A. Shcherbinin*[1]; A. G. Barantsev[1]; V. Y. Buzunov[1]; V. I. Savinov[1]; [1]PSC Bratsk Aluminum Plant, Bratsk 665716 Russia

Bratsk Aluminum Plant has developed a computer-aided system for pre-set voltage control which includes data, calculation and estimation blocks. The voltage re-distribution between the potrooms is executed on the basis of data on the state of the cells in various potrooms (number of cells of a certain type, number of cells with collector bars of a certain type, number of cells of various age, stud ages, busbar types). The voltage re-distribution between the cells of each potroom is executed on the basis of comparative analysis of the calculation results (considering all aforementioned factors) and inspection measurements.

3:15 PM Break

3:25 PM
The Impact of Varying Conductivity on Control of Aluminum Electrolysis Cells: *Tormod Drengstig*[1]; Steinar Kolås[2]; Trond Støre[2]; [1]Stavanger University College, Sch. of Sci. & Tech., Dept. of Electl. & Comp. Eng., PO Box 2557, Ullandhaug, Stavanger 4019 Norway; [2]Hydro Aluminium, Tech. Ctr., Årdal 6807 Norway

In this paper we investigate from a theoretical point of view the impact of varying bath temperature, varying excess AlF_3 and varying alumina concentration on cell resistance and cell control. The results are interpreted using knowledge of the behavior of a general resistance controller to study the influence on the anode-cathode distance. This again leads to the proposal of a new control strategy for bath temperature and excess AlF_3. The theoretical results obtained are supported using real data.

3:50 PM
Dynamic Control of the Criolyte Ratio and the Bath Temperature of Aluminium Reduction Cell: *Vladimir Yourkov*[1]; Victor Mann[1]; Tatyana Piskazhova[1]; Konstantin Nikandrov[1]; Oleg Trebukh[1]; [1]PSC Krasnoyarsk Aluminium Smelter, Krasnoyarsk 660111 Russia

At the two previous TMS conferences the papers about a dynamic cell model, developed at Krasnoyarsk Aluminium Smelter and about a virtual reduction cell, based on this model, which imitates the operation of an industry cell, were presented. On basis of the above-mentioned model the system of criolyte ratio and bath temperature control of reduction cell was developed, tested and applied in several potrooms of KRAZ. The system is integrated by now into the potroom technologist workstation and uses the technological database of the smelter. The function of the system lies in following: On basis of technological data for previous 7 days, which are delivered continuously (voltage, current) or periodically (bath criolyte ratio and temperature, metal and bath levels etc.) at first the process in retrospective is imitated for obtaining predictions of all technological variables at the moment, and than the optimal three days values for aluminium fluoride feeding,the optimal voltage setpoint value for the next day are calculated with the optimization procedure. The calculation can be made daily or every 2 or 3 days. The calculated values are of recommendation character, but the technologists got persuaded, that following the system recommendations increases the stability of criolyte ratio and bath temperature. The first group of cells was connected to the system in May 2001. After two weeks the standard deviations from the criolyte ratio setpoint decreased on this cells from 0.07-0.11 to 0.04-0.05 and remain stabile low up to now.

4:15 PM
Analysis of the Phenomena at the Start-Up of an Al Electrolysis Cell by the Visualization of the Currents Distributions: *Aureliu Panaitescu*[1]; Augustin Moraru[1]; Ileana Panaitescu[2]; Gheorghe Dobra[3]; Marian Cilianu[3]; [1]University "Politehnica" of Bucharest, Electl. Eng. Dept., Splaiul Independentei 313, Bucharest 77206 Romania; [2]ISVOR, Torino Italy; [3]S.C. ALRO S.A., 116 Pitesti St., Slatina Romania

In the paper are presented the results of some experimental researches on the phenomena that appear at the start-up of an Aluminum electrolysis cell. The analyzed electrolysis cell has 92 kA and belongs to Hall no. 10 of the Aluminum electrolysis plant ALRO Slatina, Romania. The experimental data were obtained by acquisition

of the currents that flow through the 64 anode rods, for long intervals of time, starting with the period of cell warming, the period before the starting-up (shunt removal, cryolite feeding, flexible start-up elements removal), followed by the start-up sequence itself (feeding of the cell with liquid electrolyte, the first prolonged anode effect, change of the first anodes, introduction of the molten metal). It was analyzed the stability of the cell operation up to the third day after the start-up.

Automotive Alloys 2002 - III

Sponsored by: Light Metals Division, Aluminum Association, *Program Organizer:* Subodh K. Das, Secat, Inc., Lexington, KY 40511 USA

Tuesday PM Room: 611
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chair: Subodh K. Das, Secat Inc., 1505 Bull Lea Blvd., Lexington, KY 40511 USA

2:00 PM
Strain Development and Microstructure Evolution in Aluminum Tube Bending and Hydrofroming: *Alan A. Luo*[1]; Anil K. Sachdev[1]; [1]General Motors R&D Center, Matls. & Proc. Lab., MC 480-106-212, 30500 Mound Rd., Warren, MI 48090-9055 USA

This paper investigates the strain development and microstructure evolution of aluminum alloy 6061 extruded tubes during bending and hydroforming. The results show that aluminum tubes can spread the major strain over the bend longitudinally, similar to bending in steel tubes. The minor strains developed during bending are very small, indicating a plane-strain condition in rotary draw bending. However, localized minor strains in the tube corners can reach nearly 20% upon hydroforming. In press-quenched 6061-T4 tubes, the grain structure shows large and elongated grains along the extrusion direction. Such grains are further elongated longitudinally upon bending, and stretched circumferentially at hydroforming. The fracture surfaces of burst samples of the aluminum tubes were also analyzed. There is also significant amount of work-hardening, a 28% increase in yield strength, at the tube straight sections after hydroforming.

2:30 PM
Precipitation Hardening in Super Purity Al-0.8%Mg2Si Alloys Containing Cu: *Alok K. Gupta*[1]; David J. Lloyd[1]; [1]Alcan International, Ltd., Kingston R&D Ctr., PO 8400, Kingston, Ontario K7L 5L9 Canada

Commercial 6xxx automotive sheet alloys contain about 0.8 to 1 wt% Mg2Si together with excess Si and Cu each up to 0.8 wt%. In this study, the effect of Cu on 0.8 wt%Mg2Si alloy containing varying amount of excess Si has been studied with the help of tensile tests, differential scanning calorimetry and electron optical techniques. Seven alloys were cast, homogenized, hot and cold rolled to the final gauge. These alloy were then solutionized and quenched in forced air, cold water and boiling water. The properties were evaluated immediately after quenching and following one week of natural ageing. The results show that the properties of the alloys are a function of the solute level and processing conditions of the alloys. The presence of excess Si improves the paint bake response, although quenching conditions play an important role in determining the age hardenability of the alloys, especially those containing Cu.

2:55 PM
Spot Impact Welding of Sheet Aluminum: *Anthony J. Turner*[1]; *Peihui Zhang*[1]; *Vincent J. Vohnout*[1]; *Glenn S. Daehn*[1]; [1]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., 477 Watts Hall, Columbus, OH 43210 USA

Solid-state explosive welding was developed largely in the 1960's and is still in sustained use today. It allows one to combine radically dissimilar materials without the typical problems associated with heat affected zones and deleterious intermediate phases. The clear drawbacks of this technique include the need to store large amounts of explosive and the very large amounts of energy released upon discharge. We have recently found that similar solid state bonding can be developed using a projectile to strike two thicknesses of aluminum sheet to form them into a constrained shape. Metallurgical bonding is obtained in regions where the sheets slide significantly under large normal pressures, and strengths similar to those found in spot welds can be attained. We will show how dynamic finite element modeling can be used to understand and ultimately design for this process. The desired ultimate outcome is a technique that may compete with aluminum spot welding, which has significant drawbacks in joining aluminum.

3:20 PM
A Model for Eutectic Solidification in Sr-Modified Al-Si Foundry Alloys: Stuart D. McDonald[1]; Arne K. Dahle[1]; *John A. Taylor*[1]; David H. StJohn[1]; [1]CAST, Dept. Mining, Minls. & Matls. Eng., The University of Queensland, Brisbane, QLD 4072 Australia

A simple model for the nucleation and growth of Al-Si eutectic in Sr-modified hypoeutectic aluminium-silicon alloys solidified at low cooling rates is presented. The model was developed through a combination of cooling curve analysis and microstructural observations. Although it is confirmed that strontium promotes modification of the silicon phase through a flake-fibre transition, the degree of modification within a given casting often varies with location, with many areas displaying completely different eutectic morphologies. This lack of consistency in the silicon morphology is often found in modified castings and may arise due to variations in micro and macroscopic growth conditions, both thermal and compositional, during eutectic solidification. The model adequately predicts these changes and can also be used to estimate the eutectic grain size.

3:45 PM Break

4:00 PM
High Strength Casting Alloys for Automotive Applications: *Geoffrey K. Sigworth*[1]; [1]GKS Engineering Services, 1710 Douglas Ave., Dunedin, FL 34698 USA

A number of high strength aluminum casting alloys have superior properties. These materials could be used in a number of applications to reduce vehicle weight. Cost savings may also result, because less material would be required to provide the strength or fatigue resistance needed. In spite of their excellent properties, these materials are seldom used because of their propensity for hot cracking. This problem may be reduced by grain refinement. A brief historical review of grain refinement is presented, and then a new, improved grain refining process is presented. The new grain refinement process reduces the tendency for hot cracking in several casting alloys. Also, a new ultrasonic test procedure to detect cracks appears promising. Consequently, it appears to be time to reconsider the commercial feasibility of several high strength aluminum casting alloys. Control arms were produced from AA A206 alloy to establish the viability of this alloy for suspension components. The results from the first stage of production trials are given in this paper.

4:25 PM
Modelling Recovery and Recrystallization Kinetics during Continuous Annealing: *Johnson Go*[1]; Mary A. Wells[1]; Matthias Militzer[1]; Warren J. Poole[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, British Columbia V6T 1Z4 Canada

Over the past few decades, there has been an increased use of continuous annealing to manufacture advanced sheet metals for automotive applications. In an effort to develop process models to account for the highly non-isothermal conditions in continuous annealing, the recovery and recrystallization behaviour of the commercially cold-rolled aluminium alloys AA5754 and AA6111 was studied. As a result, a model for recovery and recrystallization kinetics has been developed which is applicable to typical annealing cycles involving rapid heating to a soak temperature. The model draws on the concept of internal state variables and adopts a rule of mixture to capture the time evolution of flow stress based on the dislocation density and the fraction recrystallized. Continuous annealing tests were performed simulating heating rates of industrial continuous annealing lines to validate the model predictions. For comparison, similar investigations have been conducted for a boron-containing interstitial free steel.

4:50 PM
Liquation in Al-Cu Alloys: Computational and Experimental Investigations: Briama Cooper[1]; Andre Wilson[1]; Joseph Harvey[1]; Paul Howell[1]; *Zi-Kui Liu*[1]; [1]The Pennsylvania State University, Dept. of Matls. Sci. & Eng., Steidle Bldg., University Park, PA 16802 USA

During welding, liquid may form in regions of the heat-affected-zone (HAZ) and often become associated with the grain boundaries, making the weldment susceptible to HAZ liquation cracking. In most instances, it is likely that several liquation mechanisms operate simultaneously. In the present work, the microstructurally-based liquation mechanism has been studied through experimental investigations and computational simulations in Al-Cu alloys. The regions of liquation and non-liquation are then evaluated with respect to heating rate and size of precipitates for given alloys. The procedure can be extended to multi-

component alloys and thus provide fundamental criteria on the susceptibility of commercial alloys to microstructurally-based liquation during heat-treatment and welding.

5:10 PM
The Influence of Homogenisation Cooling Rate, Billet Pre-Heating Temperature and Die Geometry on the T5-Properties for Three 6XXX-Alloys: *Jan Anders Sater*[1]; Elin Beate Bjoernbakk[1]; Ulf Tundal[1]; Oddvin Reiso[1]; [1]Hydro Aluminium, R&D Matls. Tech., Romsdalsveien 1, 6600 Sunndalsoera Norway

The use of aluminium for automotive applications has increased significantly the last years. Due to its beneficial strength-to-weight ratio, it is an attractive replacement for steel in automotive bodies and for use in automotive safety components. For taking full advantage of the strength potential in aluminium alloys the material needs to be processed in an optimum way. During extrusion of aluminium, several factors will influence on the strength of the final product. This can be alloy chemistry and microstructure or extrusion process conditions. In the present work, some of these factors have been investigated. Three different 6000-series alloys have been cast and homogenised with different cooling rates from the homogenisation temperature. The alloys were pre-heated in the temperature range 400°C-540°C before extrusion into different profile geometries. The profiles were aged to T5-condition and the mechanical properties were thereafter measured by tensile testing. It was found that the ultimate tensile strength and the flow stress increase with increasing pre-heating temperature. This is explained in terms of dissolution of Mg2Si-particles during billet pre-heating and the extrusion cycle. A low cooling rate after homogenisation gives lower mechanical properties after ageing, as compared to a higher homogenisation cooling rate. This effect is clearly visible at low pre-heating temperatures for the lean alloys 6060 and 6063.

Carbon Technology: Anode Baking and Process Improvements

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: Don T. Walton, Aluminum Company of America, Wenatchee Works, Malaga, WA 98828-9728 USA; Les Edwards, CII Carbon, Chalmette, LA 70044 USA; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday PM — Room: 602-603
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Stephen L. Whelan, Alcoa, Park Ave., E., PO Box 150, Massena, NY 13662 USA

2:00 PM Cancelled
Measurements of the Temperature Distribution in Anode Baking: Dido Rafael Cedeño

2:25 PM
Greenfield Carbon Baking Furnace: *F. Eric Knott*[1]; [1]Century Aluminum-Hawesville Operations, Techl., 1627 S. R. 271 N., PO Box 500, Hawesville, KY 42348 USA

A new carbon anode baking furnace was installed at Hawesville as part of the Fifth Potline expansion project in July 1999. The design of this furnace was based on computer modeling techniques, which looked at flows, pressures, temperature distributions, and species concentrations. The major design considerations were strength, flow, and draft aimed at anode quality, operating efficiency, and refractory maintenance costs. This paper discusses the furnace design, operating characteristics and performance over the first two years of operation.

2:50 PM
Advanced Cooling of Anodes in an Open Ring-Type Baking Furnace: *Detlef Maiwald*[1]; *Michael Schneider*[2]; *Christian Krupp*[2]; [1]Innovatherm, Automation, Roter Lohweg 22, Butzbach 35510 Germany; [2]Corus Aluminium Voerde GmbH, PO Box 101154, Voerde 46549 Germany

The production capacity of an open ring-type baking furnace is defined by the number of firing groups per furnace, the number of sections and pits and their dimensions and the time the anodes are heated up and cooled down. The cooling phase is a necessary, but non-productive process with a high potential to damage the baked anode and no potential to improve the quality. Therefore it is important to have a fast and well controlled cooling process. This paper will describe at first the complexity of the apparent simple cooling process. Secondly, experiences of an existing baking furnace will be described and finally tools and options to reduce the inefficiency of the cooling process will be given.

3:15 PM
Development and Test In Situ of a Heavy Oil Burner for Albras Bake Furnaces: *Ronaldo Raposo de Moura*[1]; [1]Albras Alumínio Brasileiro S/A, Carbon Plant, Rod. PA 483 Km 21, Vila do Murucupí, Barcarena, Pará 68447000 Brazil

The importance of fuel atomization in the combustion process and how it has been done in a specific application at Albras are discussed. A twin fluid atomizer (air/heavy oil) was proposed to substitute the original one in an oil-fired Open Top Horizontal Flue Ring Furnace for anode baking. The new heavy fuel oil burner, developed by Albras, is compared with the old one in a new baking furnace in the same process environment. Important aspects like: oil consumption, final anode temperature in the pit top layer and the thermal profile in the combustion chamber were analyzed to demonstrate the better efficiency of the twin fluid atomizer. Finally, the potential economic impact of the burner change in the anode production cost was studied.

3:40 PM
Improved Anode Baking Furnace Cover Material: *Fred Brunk*[1]; [1]HAW

The paper describes a new type of anode baking furnace cover material which replaces more traditional metallurgical or petroleum coke in closed baking furnace designs. The new granular material is a SiO2-CaO based ceramic which has been used routinely at the HAW smelter for the last 2 years with excellent results. The material is used as a cover layer on top of a thin coke layer and has resulted in the following improvements: lower packing coke consumption and reduced furnace operating costs, cleaner flue walls as a result of distinct lower slag formation and caking on the refractory brick lining, improved anode quality due to thermal insulating of the re-usable cover material. The paper discusses the composition and practical application of the new material.

Cast Shop Technology: Alloying

Sponsored by: Light Metals Division, Aluminum Committee
Program Organizers: David H. DeYoung, Alcoa Technical Center, Alcoa Center, PA 15069 USA; John F. Grandfield, CSIRO Australia, Preston, Victoria 3072 Australia; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday PM — Room: 6A
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Clark Weaver, Alcan Inc., Casting Tech., 1955 Mellon Boul., Jonquierre, Quebec G7S 4L2 Canada; Jim Pearson, LSM, Fullerton Rd., Rotherham, S. Yorkshire S60 1DL UK

2:00 PM
Recent Studies of Compacted Additives Including Industrial Scale Tests: *Gregorio Borge*[1]; Paul S. Cooper[2]; [1]Bostlan, S.A., Techl. Dept., Polígono Industrial Trobika, Mungia E-48007 Spain; [2]London & Scandinavia Metallurgical Company, Ltd., Techl. Ctr., Fullerton Rd., Rotherham, S. Yorkshire S60 1DL England

There have been several studies in recent years aimed at improving the understanding of how compacted additives behave when added to molten aluminium in a furnace. Most of these have been on a laboratory scale, where it is impossible to reproduce some of the situations encountered in the cast house. They have also mostly concentrated on cast house practice. This paper reviews the previous studies and how they have helped generate a model of dissolution mechanisms. Results from new studies based on more realistic conditions are presented. An emphasis of the studies is on the characteristics of the compacts themselves. Conclusions are drawn on how good and poor performances are achieved with compacted additives.

2:25 PM
An Assessment of Manganese Addition Practice by MnAl Briquettes and Mn Metal Powder Injection into Aluminum Melt: *Young E. Lee*[1]; Dana L. LeMasters[1]; [1]Eramet Marietta, Inc., St. Rt. 7 S., Riverview Dr., Marietta, OH 45750 USA

The addition of Mn metal as an alloying element in aluminum is practiced by MnAl briquette or Mn powder injection at cast shops.

Both addition methods are competing against each other with conflicting claims. A critical assessment with the plant trials indicates that both additive methods do not deliver the same cost effective performance because they are constituted differently and require different operating conditions at cast shops. The addition of MnAl briquettes does not require auxiliary equipment and extra attention. As it effectively utilizes the exothermic heat from the reaction between the contained ingredients, the dissolution reaction completes with a full recovery of Mn within a reasonable processing time at a relatively low operating temperature. The performance is relatively insensitive to the particle size distribution if the top particle size is limited to less than 40 mesh. Mn metal powder injection fluidizes particles with carrier gases and requires auxiliary equipment. Because of its dependency on particle size for its effective performance, it results in an inconsistent and poor performance when Mn powder includes particles smaller or larger than the optimum size. Its operation time is longer because of limited Mn feed rate, and its operation temperature higher because of high thermal losses.

2:50 PM
The Behaviour of Selenium Impurities during the Alloying of Aluminum with Manganese Additions: *Roderick Ian Lawrence Guthrie*[1]; Mihaiela Isac[1]; Sayed A. Sajjadi[1]; [1]McGill University, McGill Metals Procg. Ctr., M.H. Wong Engineering Bldg., 3610 University St., Montreal, Quebec H3A 2B2 Canada

Manganese is an important alloying element for both aluminum and steel melts. In the case of aluminum cast house practices, the manganese is either added as briquets, or is injected pneumatically into the melt through submerged lances. Some of the electrolytic grade manganese alloys used by the Light Metals Industries can be heavily contaminated with selenium as a result of the electrolytic processing route adopted for their production. Small scale laboratory tests were carried out in order to simulate the briquet method of alloying, and to determine how the selenium distributed itself between the melt, dross, and atmosphere. Similarly, since the chemical form of selenium is important from an environmental point of view, being hazardous to humans when present as a compound, chemical analyses of the vapors exhausted from the melt were made.

3:15 PM Break

3:30 PM
Silicon Granules for Aluminium Alloys: *Gerard Baluais*[1]; Johan Strydom[2]; *Michael Brown*[1]; [1]Pechiney Electrometallurgy, Invensil, Paris France; [2]Invensil, Pietersburg S. Africa

The time of dissolution of the additional components into aluminium bath to provide specific alloys is often the key of the productivity of the step of melting in the aluminium foundries. The faster is the operation the better is the cost. The saving of time allows to increase the number of melts and to decrease the oxidation of the elements, aluminium and magnesium above all. Because of your position of producer of silicon metal we have studied this question and we have made some industrial trials with especial metallurgical silicon grade. The paper presents the characterization of the products and gives the results in term of % recovery, dissolution rate, gassing and inclusions of aluminium alloys. Tentatives of explanations and mechanisms will be given and particularly why the dissolution of silicon granules is very fast, not only because its granulometric features. Others advantages of silicon granules in term of storage and chemical quality will be improve.

3:55 PM
10% Strontium Master Alloy Modification Performance in A356 as a Function of Al4Sr Size and Temperature: *Stephanie Sebacher*[1]; Robert W. Hyland[2]; [1]KB Alloys, Inc., Tech., 3293 McDonald Rd., Robards, KY 42452 USA; [2]KB Alloys, Inc., Tech., Box 53, 220 Old W. Penn Ave., Robesonia, PA 19551 USA

Master alloy performance requirements in cast shop and foundry applications continue to focus upon improved value generation for the end user, commonly in the form of lower operating costs and improved product cycle times. A prototypical example may be found in the microstructure modification of Al-Si A3XX casting alloys, in which rapid Al4Sr dissolution and Al-Si eutectic microstructure modification reactions are required of Al-Sr master alloys. In the present work we investigate the hypothesis that there exists a strong correlation between Al4Sr strontium aluminide size and size class distribution in the master alloy and the associated rate of dissolution and hence level of structural modification in the end product. Here we report on the results of bench scale, continuous cooling experiments that assess the phenomenological relationship between strontium aluminide size and rate of eutectic silicon modification in A356 alloy. These are compared with theoretical estimates of the rates of dissolution based up on long range volume diffusion controlled (VDC) dissolution kinetics. For the relative thermodynamic driving forces used in these experiments, these estimates provide support for the observed and comparatively weak dependence of Al-Si microstructure refinement upon initial Al4Sr size.

4:20 PM
Possibility of Substituting Mn for Cr in 6061 Aluminum Alloy Judged from the Point of View of Toughness and Tensile Properties: *Mitsuo Niinomi*[1]; Toshikazu Akahori[1]; Kei-ichi Fukunaga[1]; Sinji Kumai[2]; Mikihiro Kanno[3]; [1]Toyohashi University of Technology, Production Sys. Eng., 1-1 Hibarigaoka, Tempaku-cho, Toyohashi 441-8580 Japan; [2]Tokyo Institute of Technology, Matls. Sci. & Eng., 4259 Nagatsuta-cho, Midori-ku, Yokohama 226-8502 Japan; [3]The University of Tokyo, Grad. Sch. of Eng., 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8654 Japan

The amount of 6000 series aluminum alloys fabricated is around 400 ton per year and are used widely up to now in Japan. Recently, these alloys are getting much attention for automobile applications and are expected to use in place of other series aluminum alloys. 6061 aluminum alloy, Al-1.0 mass % Mg-0.6 mass % Si-0.25 mass % Cu-0.25 mass % Cr, is a representative aluminum alloy among other 6000 series aluminum alloys with excellent mechanical properties. However, additive transition elements in 6061 should be simplified for recycling of this alloy. Therefore, modified T-6 treated 6061 aluminum alloys where manganese, Mn, is substituted for chromium, Cr, and cooling rate after homogenization was changed between water quenching and air cooling were fabricated and their tensile properties and toughness were investigated in order to judge the possibility for substituting Mn for Cr.

Charles J. McMahon Interfacial Segregation and Embrittlement Symposium: New Methods for Study of Segregation and Fracture I

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, Jt. Mechanical Behavior of Materials, Jt. Computational Materials Science & Engineering, Texture & Anisotropy Committee
Program Organizers: Vaclav Vitek, University of Pennsylvania, Department of Materials Science and Engineering, Philadelphia, PA 19104 USA; Clyde Briant, Brown University, Division of Engineering, Providence, RI 02912 USA; Harvey D. Solomon, General Electric Company, Research & Development Center, Schenectady, NY 12309 USA

Tuesday PM — Room: 307-308
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Gregory M. Olson, Northwestern University, Evanston, IL 60208 USA; John L. Bassani, University of Pennsylvania, Dept. of Mechl. Eng., Philadelphia, PA 19104 USA

2:00 PM Invited
Advanced Analytical Electron Microscopy Studies of Segregation in Metals and Ceramics: *Manfred Rühle*[1]; [1]Max-Planck-Institut f. Metallforschung, Seestr. 92, Stuttgart 70174 Germany

Advanced analytical microscopy allows the analysis of materials down (nearly) to the atomic level. The composition of atomic columns can be determined. With different techniques (spatial difference technique) the net amount of segregation can be detected with high sensitivity which is comparable or even better than the sensitivity of Auger electron microscopy. Results for metals will be described, however, emphasis will be on investigations at grain boundaries in oxides such as α-Al_2O_3 and $SrTiO_3$. Extensive studies were done for different α-Al_2O_3 ceramics containing a well-defined amount of impurities. The segregation at different grain boundaries was studied using a dedicated STEM with expected high spatial resolution. The results observed so far suggest that abnormal grain growth starts if a certain level of specific impurities is segregated at grain boundaries. Those observations are supported by quantitative studies of the correlation between segregation at grain boundaries, grain size and grain size distribution. The implication of the different microstructures with respect to microstructural stability are discussed and compared to results described in the literature. A model will be presented for the cause of abnormal grain growth.

2:35 PM Invited

Nanoscale Studies of Segregation at Heterophase Interfaces: *David N. Seidman*[1]; Dieter Isheim[1]; Jason T. Sebastian[1]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Matls. & Life Scis. Bldg., Evanston, IL 60208-3108 USA

Quantitative experimental results of segregation at heterophase interfaces, ceramic/metal and metal/metal, on the sub- to nanometer length scales are presented. We are studying the relationships between interfacial segregation and structure employing atom-probe (APFIM) and three-dimensional atom-probe microscopy (3DAP), Z-contrast microscopy, high-resolution electron microscopy (HREM), and electron energy loss spectroscopy. The specific systems are: MgO/Cu(Ag), MgO/Cu(Sb), CdO/Ag(Au), MnO/Ag(Sb), Fe(V)/Mo, and alpha-Fe(Sn)/molybdenum nitride, and alpha-Fe(Sb)/molybdenum nitride. The emphasis is on measuring quantitatively the Gibbsian interfacial excess of solute and finding its relation to the interfacial structure. APFIM and 3DAPM analyses of the last two heterophase interfaces show substantial segregation of Sn and Sb only at interfaces containing misfit dislocations as observed by HREM, with Gibbsian interfacial excesses of up to 7±3 nm-2, while coherent have no detectable segregation. For semicoherent interfaces the value of the Gibbsian interfacial excess depends on whether or not the interface contains its full complement of necessary misfit dislocations.

3:10 PM Invited

A New Method to Predict the Enthalpy and Entropy of Solute Segregation at Individual Grain Boundaries: *Pavel Lejcek*[1]; Siegfried Hofmann[2]; [1]Institute of Physics, Acad. Sci. Czech Rep., Na Slovance 2, Praha 8 182 21 Czech Republic; [2]Max-Planck-Institut fur Metallforschung, Seestrasse 92, Stuttgart 70174 Germany

Theoretical and experimental studies of the properties of crystallographically well defined grain boundaries disclosed various interrelations between characteristic parameters. Two of them are related to interfacial segregation and will be presented in this contribution: (i) the dependence between the enthalpy of solute segregation on solid solubility and on the grain boundary structure, resulting in the construction of grain boundary segregation diagrams, and (ii) the linear relationship between segregation entropy and enthalpy for all grain boundaries. Based on these two dependences, it is possible to predict enthalpy and entropy of segregation of any element at any grain boundary. The method of this prediction and its capability will be documented by the comparison of predicted interfacial composition in numerous alpha-iron base binary systems with the recently measured experimental data for the same alloys.

3:45 PM

Segregation of Impurities to the Sigma 5 (310)/[001] STGB and the Influence to the Grain Boundary Structure: *Juergen M. Plitzko*[1]; Geoffrey H. Campbell[1]; Wayne E. King[1]; Stephen M. Foiles[2]; [1]Lawrence Livermore National Lab, Chem. & Matl. Sci., 7000 E. Ave., MS L-370, Livermore, CA 94550 USA; [2]Sandia National Laboratories, Computl. Matls. Sci. Dept., Albuquerque, NM 87185-1411 USA

For our investigations we have chosen sigma 5 symmetric tilt grain boundaries (STGB) in two face-centered cubic (FCC) metals, aluminum and copper. Both metals were doped with only 1 at% of the impurity species (Cu and Ag). One of our major goals in this study was to investigate not only the grain boundary structure and the influence of a segregated impurity but also the size effect on segregation of an impurity to distinct sites. Therefore we have selected the Ag as an impurity in Cu and Cu as an impurity in Al. The latter one is of special interest for applications like interconnects in microcircuits, where one of the major controlling factors of electromigration is expected to be the diffusion or segregation of Cu atoms at Al grain boundaries. The atomic structures were modeled with atomistic potentials based on the Embedded Atom Method (EAM), where we used a mixed basis set within the Local Density Approximation (LDA). For the determination of the atomic structure and the investigation of the size effect of the segregant species at this specific grain boundary, high resolution electron transmission microscopy (HRTEM) was used in combination with focal-series reconstruction. The interpretation of HRTEM images obtained with field emission technology can be somehow complicated. Therefore, we applied focal-series reconstruction of the exit wave function to exclude imaging artefacts and to validate the predictions of the theoretical grain boundary models. For the Al-1at%Cu bicrystal, we have discovered a structure, which was previously not considered. However, the possibility of an substitutional interstitial has been mentioned in literature but our experimental results validate for the first time the ab initio calculation. Additionally, we will present first results for the Cu-1at%Ag bicrystal obtained with HRTEM and analytical electron microscopy including electron energy loss spectroscopy and x-ray energy dispersive spectrometry. In both cases the amount of the segregated species at the interface was quantified and compared to the predictions of the EAM model. We will present and compare our findings regarding the two different metals with the modeled systems in detail and we will discuss our observations with respect to earlier presented results. This work was performed under the auspices of the US Department of Energy Office of Basic Energy Sciences by the University of California, Lawrence Livermore National Laboratory under contract number W-7405-Eng-48.

4:05 PM

Effects of Segregation on the Interfacial Fracture Energy: *W. W. Gerberich*[1]; J. M. Jungk[1]; J. W. Hoehn[2]; [1]University of Minnesota, Matls. Sci. & Eng., Minneapolis, MN USA; [2]Seagate Technology, Bloomington, MN USA

Superlayer indentation fracture tests on metallic thin films have shown that thin layers of deposited or segregated species can reduce the true surface energy by a factor on the order of two. It is shown by a series of tests with increasing film thickness that this can have nearly order of magnitude effects on the effective surface energy of thicker films because of the reduced plastic energy dissipation truncated due to a lowered debond strength. Effects of carbon, hydrogen, silver, copper and nickel between thin metallic films of niobium, aluminum, gold and copper are discussed. For example, several monolayers of Ni deposited on a substrate can degrade the interfacial toughness of subsequently deposited 100 nm Au film from about 2 N/m to 0.3N/m.

4:25 PM

Crystal Orientation Examination of Crack Propagation of the Haz of 1 1/4 Cr-1/2 Mo Steel: *Shig Saimoto*[1]; Charles H.J. Orchard[1]; Shaotang Cao[1]; [1]Queen's University at Kingston, Matls. & Metall. Eng., Nicol Hall, Union St., Kingston, Ontario K7L 3N6 Canada

Service life prediction of steam pipe correlates to the integral heat treatment of the steel. With the advent of micro-textural examination using orientation image microscopy, the examination of cracking in the steamline weldment was re-examined. The failure occurred in service after approximately 88,000 operating hours with a steam pressure of 16.2 MPa and an operating temperature of 538C. Both the components and weld metal were 1 1/4 Cr-1/2 Mo steel. The circumferential crack occurred between a forced Tee piece and the cast main steam stop valve. A sample was sectioned nearly perpendicular to the crack and polished. After electro-polishing, the microstructure was examined by electron channelling contrast and various segments along and in front of the crack was surveyed using orientation imaging. The details of this examination will be discussed with respect to the role of solute segregation in the grain boundaries depending on its misorientation.

4:45 PM

Structural Studies on Segregation of Na in Sigma 3 Boundries of Si Found in Na-Modified Al-Si Eutectic Alloy: *Mohammad Shamsuzzoha*[1]; [1]University of Alabama, SOMED, Tuscaloosa, AL 35487 USA

The crystal of Si phase in a Na-modified Al-Si alloy has been investigated by conventional and high-resolution electron microscopy. The Si crystals of this phase possesses a high-density of symmetric and asymmetric Sigma 3 boundaries. HERM images taken from some of the symmetric boundaries show segments primarily composed of a structural unit different than that found in a normal Sigma 3 of Si boundary. This variation in the content of the Sigma 3 structural unit is attributed to the local ordering of Na in the boundary resulted by the preferential segregation of Na atoms occurring during the solidification of the alloy.

5:05 PM

Nonequilibrium Grain Boundary Segregation in Austenitic Stainless Steels Induced by Vacancy Flow and Chemical Binding: *Edward P. Simonen*[1]; Dan J. Edwards[1]; Stephen M. Bruemmer[1]; [1]PNNL, Matls., PO Box 999, Richland, WA 99352 USA

A measurement and modeling approach has been developed to quantify approaches for controlling nonequilibrium grain boundary segregation during heat treatment and irradiation. Specifically, change in Cr concentration at grain boundaries in austenitic stainless steel is elucidated. This research has unexpectedly demonstrated that chemical binding can be critical during nonequilibrium segregation processes. Solute segregation during cooling is shown to require vacancy/solute binding energies that are only consistent with chemical binding. The determined binding energies are unreasonably large for conventional elastic binding. The slow disappearance of segregation during irradiation also supports the conclusion that chemical binding is important during nonequilibrium enrichment of Cr caused by cooling transients.

Quantification of the interaction and migration energies has allowed predictions of grain boundary compositions as they depend on heat treatment or irradiation parameters. This work was supported by the Materials Sciences Branch, BES, US Department of Energy, under Contract DE-AC06-76RLO 1830.

5:25 PM

Crack Tip Plasticity in Copper Bicrystals: *Jin Yu*[1]; J. W. Cho[1]; [1]Korea Advanced Institute of Science and Technology, Ctr. for Elect. Pkgg. Matls. (CEPM), Korea

Due to the limited number of slip systems and crystal anisotropy, characteristics of plasticity in single crystals are quite different from those of polycrystals. Previous work showed that the crack tip plastic zone develops as fan shaped sectors in single crystals, as predicted by theories, but details of slip traces and sector positions observed on specimen surfaces differ markedly from predictions and could be reasonably explained by the exclusive latent hardening model [1]. Plastic sectors observed on the specimen surface are reasonably valid on the specimen interior as well when the plane strain deformation prevails and the amount of necking near the crack tip is small. Only limited studies of the crack tip plasticity in bicrystals with interfacial crack have been made so far. In the present work, copper bicrystals with symmetric tilt boundaries of well defined coincidence site density, were grown by the Bridgeman method, and the crack tip plasticity was studied using the CT specimens notched along the interface. Then, bicrystal interfaces were embrittled by introducing Bi on the interface, and the effects of impurity induced embrittlement on the crack tip plasticity were investigated by studying slip traces and through thickness profiles on specimen surfaces. Finally, the exclusive latent hardening model, developed for single crystals, is used to explain slip traces observed on bicrystal surfaces, and measured fracture toughness of the interfaces was compared with that of other workers. [1]. J. Yu and J.W. Cho; p.311, Multiscale deformation and fracture, J.R. Rice 60th anniversary volume, ed. T. J. Chuang and J. W. Rudnicki, Kluver, Dordrecht, Netherlands (2000).

Commercial Mini-Sessions

Sponsored by: Light Metals Division
Program Organizers: David V. Neff, Metaullics Systems Company, Solon, OH 44139 USA; Wolfgang Schneider, VAW Aluminium AG, Research & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday PM — Room: 401
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: David V. Neff, Metaullics Systems Company, 31935 Aurora Rd., Solon, OH 44139 USA

Computational Modeling of Materials, Minerals & Metals Processing: Tuesday PM Plenary Session 2:00 PM – 2:45 PM

Sponsored by: Materials Processing & Manufacturing Division,
Program Organizer: Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Tuesday PM — Room: 619-620
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Koulis Pericleous, University of Greenwich, Greenwich Maritime Campus, Queen Mary Ct., Rm. 361 Greenwich, London SE10 9LS UK

Keynote

Computational Modelling of Metals Reduction Processes: *Phil Schwarz*[1]; [1]CSIRO Minerals, Box 312, Clayton S., Victoria 3163 Australia

This paper reviews the status of computational modelling of a variety of common metals reduction processes, namely the blast furnace, rotary kiln and fluidised bed, and one new process not yet commercialised, namely smelting-reduction as in the HIsmelt® Process. In each case, the last decade has seen the emergence of the capability to simulate the processes using multi-phase reacting computational fluid dynamics techniques. In some cases this capability is still in the process of being developed, and the next few years will see the maturing of the modelling techniques. As they become established, it will be possible to apply them to further refine the older technologies such as blast furnaces and rotary kilns, and assist in the optimisation, commercialisation and acceptance of the newer technologies such as fluidised bed and molten bath reduction. The development of a computational fluid dynamic model of bath smelting-reduction is described in some detail to illustrate how a large number of complex and interacting phenomena can be successfully simulated within a CFD framework.

Computational Modeling of Materials, Minerals & Metals Processing: Track A - CFD Modeling - II

Sponsored by: Materials Processing & Manufacturing Division,
Program Organizer: Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Tuesday PM — Room: 619
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Phil Schwarz, CSIRO Minerals, Box 312, Clayton S., Victoria 3163 Australia; Pascal Gardin, IRSID, Maizieres-les-Metz 57283 France

2:45 PM

Modelling of Raceway Hysteresis: Govind S. Gupta[1]; *S. Sarkar*[1]; M. G. Basavaraj[1]; P. D. Patel[2]; [1]Indian Institute of Science, Dept. of Metall., Bangalore 560 012 India; [2]Indian Institute of Science, Dept. of Cheml. Eng., Bangalore 560 012 India

Previous experimental study on raceway size hysteresis on two-dimensional cold model showed that the interparticle and wall-particle friction had a very large effect on the raceway size. Existing literature correlations for raceway size ignore the frictional effects. It has also been shown in the present study that their applicability to the ironmaking blast furnace is questionable. To take into account the effect of friction on the raceway size a stress analysis has been done for the raceway region. The partial differential equations for the stresses have been developed and solved computationally. The frictional forces were obtained in terms of stresses. To predict the raceway size a force balance was done for the raceway zone considering the pressure force, the frictional force and the bed weight. The resulting equations from the force balance are able to describe the raceway hysteresis phenomena correctly along with the raceway size. A two dimensional experimental set up has been fabricated in order to validate the computer predictions. Predicted values agree well with the experimental values. A correlation has been developed to predict the raceway size.

3:10 PM

Lifetime Prediction of Pneumatic Conveyor Bends with the Aid of Computational Models: *Mayur K. Patel*[1]; Robert Hanson[1]; [1]University of Greenwich, Ctr. for Numl. Modlg. & Proc. Analy., 30 Park Row, Greenwich SE10 9LS UK

The puncture of pneumatic conveyor bends in industry causes several problems. Two important factors are: (1) Escape of the conveyed product causing health and dust hazard and (2) Repairing and cleaning up after punctures necessitates shutting down conveyors, which will affect the operation of the plant, thus reducing profitability. Bends in pneumatic conveying systems tend to wear out and puncture first since particles generally strike the bend walls with larger intensity than straight pipe sections. Current models for bend lifetime prediction are inaccurate as they fail to account for key parameters that are of fundamental importance to the progression of the wear. The provision of an accurate predictive method would lead to improvements in the structure of the planned maintenance programmes, thus reducing unplanned shutdowns. The paper reports the first phase of a study undertaken to develop and implement a CFD based engineering tool to predict the lifetime of conveyor bends for two- and three-dimensional test cases. The model used is based on Eulerian and Lagrangian methods. It is unique in that the erosion due to the particle impacts is accounted for within a CFD framework, thus taking into account angle of attack and impact velocity, ensuring more realistic predictions of the wear profile and their grid dependency and sensitivity to the inlet particle distributions.

3:35 PM Break

3:50 PM

A Parametric Study of Oxy-Fuel Burners in Secondary Aluminum Melting: *Madhu Huggahalli*[1]; Neeraj Saxena[1]; Ken Greishaber[2]; Jerry Bednarski[2]; David Stoffel[2]; [1]BOC Gases Technology, 100 Mountain Ave., Murray Hill, NJ 07974 USA; [2]BOC Gases, 575 Mountain Ave., Murray Hill, NJ 07974 USA

The use of oxy-fuel burners in secondary aluminum melting applications offers several advantages over air-fuel burners including reduced fuel consumption, faster charge to tap times and lower NOx emissions. Their successful, safe and economical use in a furnace depends on several factors and considerations such as burner and flue placement, metal circulation, charge practices and the type of refractory used in the furnace. These factors required in the successful conversion of furnaces from air-fuel to oxy-fuel based burners are discussed in this paper. Critical parameters are identified and examined using computational fluid dynamics (CFD) simulations. Parameters are estimated via laboratory testing and validated through trials performed at commercial installations. Guidelines and a simplified approach to estimate A priori the economic impact of converting from air-fuel to oxy-fuel are presented The final products of this research are simplified and validated tools for modeling aluminum furnaces. These include improved heat and energy balance models and parameterized CFD solutions which provide rapid customization to individual furnace configurations. These tools provide field engineers with immediate and accurate predictions of performance, allowing for repeated precise scenario analyses.

4:15 PM

Computational Modelling of Vortex Formation in the Lead Refining Kettle: Suman Kumar[1]; *Chris Bailey*[1]; Mayur Patel[1]; A. W. Piper[2]; M. Cowling[2]; R. A. Forsdick[2]; [1]University of Greenwich, 30 Park Row, Greenwich, SE10 9LS UK; [2]Britannia Refined Metals, Ltd., Northfleet UK

The refining of lead bullion takes place in hemispherical vessels (known as kettles) of various sizes. It is normal practice to remove impurity elements (i.e. copper, silver, bismuth, antimony, etc.) sequentially, by the addition of reagents. This process has been in operation for many years in refineries all over the world. Unfortunately very little is understood about the actual mixing and refining process taking place in these kettles. This paper will present a detailed modelling analysis of this process, where computational and physical modelling techniques have been used. The computational fluid dynamics (CFD) techniques used to model fluid mixing by impellers will be discussed and results will be presented comparing CFD data with plant data. Also discussed will be the modelling techniques used to simulate the reactions taking place in the vessel during impurity removal.

4:40 PM

CFD Modeling of Solids Suspensions in Stirred Tanks: *Lanre Oshinowo*[1]; André Bakker[2]; [1]Hatch, 2800 Speakman Dr., Mississauga, Ontario L5K 2R7 Canada; [2]Fluent, Inc., 10 Cavendish Ct., Lebanon, NH 03766 USA

Mechanical agitation is widely used in process industry operations involving solid-liquid flows. The typical process requirement is for the solid phase to be suspended for the purpose of dissolution, reaction, or to provide feed uniformity. If these vessels are not functioning properly, by inadequately maintaining suspension, the quality of the products being generated can suffer. Associated with the operation of these units is a need to maintain the suspension at the lowest possible cost. The challenge is in understanding the fluid dynamics in the vessel and relating this knowledge to design. CFD modeling can provide insight to both the multiphase transport and the design parameters. An understanding of the parameters that govern the just-suspended impeller speed, Njs, and the distribution of solids, is critical. Recent advances in computational fluid dynamics allow for the modeling of multiphase systems, such as the liquid-solid mixtures discussed here. Of particular interest is the Eulerian multiphase model, which uses separate sets of Navier-Stokes equations for the liquid and solids (or granular) phases. Incorporating moving impeller modeling techniques, such as, the sliding mesh method, provides a rigorous estimate of the solids suspension behavior. This paper will assess the current design parameter Njs in the context of scale-up and compare it to the quality of solids dispersion as a means of assessing correct scale-up in suspension tank design. The work presented includes a study of a 45° pitched-blade turbine and a hydrofoil impeller. Both single and dual impeller operation have been evaluated. For a given impeller style in a fixed vessel, D/T and C/T are varied to explore suspension flow patterns at Njs. The settled solids fraction for speeds below Njs, and the cloud height for impeller speeds above Njs were studied. The CFD results correspond well with experimental literature data on velocity distribution and cloud height.

5:05 PM

Water Model and Numerical Study on the Spout Height in a Gas Stirred Vessel: *Diancai Guo*[1]; G. A. Irons[1]; [1]McMaster University, Steel Rsrch. Ctr., 1280 Main St., Hamilton, Ontario L8S 4L7 Canada

The average spout height and width produced by bottom gas injection in a water model of a steel ladle were measured with an image processing technique. It was revealed that the spout height could be described by a Gaussian curve. A combined SIMPLE-VOF model was developed to simulate the liquid surface and flow. The results showed that, though the model produced reasonable velocity distributions and free surface positions, the spout height due to the dynamic head of the rising liquid was substantially lower than observed, indicating that the bubble dynamics at the bath surface play an important role in spout height.

Computational Modeling of Materials, Minerals & Metals Processing: Track B - Thermo-Mechanical Modeling - I

Sponsored by: Materials Processing & Manufacturing Division,
Program Organizer: Mark Cross, The University of Greenwich, Centre for Numerical Modelling and Process Analysis, Old Royal Naval College, 30 Park Row, Greenwich, London SE10 9LS UK

Tuesday PM — Room: 620
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Brian G. Thomas, University of Illinois-Urbana, Dept. of Mechl. & Indl. Eng., Urbana, IL 61801 USA; Danny Wheeler, NIST, 100 Bureau Dr., MS 8555, Gaithersburg, MD 20899-8555 USA

2:45 PM

Thick Yield Surface: An Approach to the Processing of Computer Experiments on Polycrystalline Deformation: *Yan Beygelzimer*[1]; Alexander V. Spuskanyuk[1]; Victor Varyukhin[1]; [1]Donetsk Physical & Technical Institute of the NAS of Ukraine, High Press. Phys. & Adv. Tech. Dept., 72 R. Luxembourg St., Donetsk 83114 Ukraine

The main idea of the report is that the yield surface can be presented as thick, “foamed surface” with dimensionality exceeding two. By other words, perhaps, the yield surface is fractal, i.e. it belongs to the geometric objects with fractional dimensionality. Apparently, fractal structure of the yield surface is determined by the fractal structure of natural materials. Besides, the “cloud of internal stresses” term is introduced to describe the stress distribution in RVE. Its plastic flow is determined by the interaction of this cloud with the thick yield surface. Thick yield surface and internal stress cloud concepts allow to determine the additional correlation between micromechanical models of polycrystals and phenomenological theory of plasticity.

3:10 PM

Model for Stress, Temperature and Phase Transformation Behaviour of Steels on Run-Out Table in Hot Strip Mill: *Heung Nam Han*[1]; Jae Kon Lee[1]; Hong Jun Kim[1]; Young-Sool Jin[1]; [1]Pohang Iron & Steel Co., Ltd. (POSCO), Sheet Prod. & Rsrch. Grp., Techl. Rsrch. Labs., Pohang PO Box 36, 1 Koedong-dong, Nam-ku, Pohang-shi, Kyungbuk 790-785 Korea

A mathematical model was developed considering non-symmetric cooling and stress distribution in both thickness and width direction of strip on a run-out table of hot strip mill. In order to solve a transient heat transfer equation including the heat evolved from phase transformation, a finite difference method coupled with thermodynamic and kinetic analyses was applied. The heat capacity of each phase and heat evolution due to phase transformation were obtained from the thermodynamic analysis of the Fe-C-Mn-Si system based on a sublattice model. The phase transformation kinetics of the steels was derived by using continuous cooling experiments and the thermodynamic analysis. Heat transfer coefficients of strips on the run-out table were, by applying an inverse method, determined from actual mill data under various cooling conditions. As for the stress analysis, the density change of strip due to cooling and phase transformation and the transformation induced plasticity were considered. A constitutive equation for the transformation induced plasticity, which is related to the phase transformation kinetics and the applied stress, was newly suggested. A finite element method was adopted to calculate the deformation behaviour of strip on run-out table.

3:35 PM Break

3:50 PM
Thermo-Mechanical Coupling Finite Element Analysis of Sheet Metal Extrusion Process: *Zhanghua Chen*[1]; C. Y. Tang[1]; T. C. Lee[1]; [1]The Hong Kong Polytechnic University, Dept. of Mfg. Eng., Hung Hom, Hong Kong, China

In sheet metal forming process, the forming limit and strain distribution are governed by plastic instability and fracture following strain localization. It has been proved that the temperature gradient caused by plastic deformation, heat transfer, and friction between sheet and tools is one of crucial factors to induce the strain localization in high speed metal forming processes. In this paper, a numerical simulation of the sheet metal extrusion process has been conducted by using thermal-mechanical coupling finite element method. In the investigation, the sheet metal extrusion is assumed to be a non-isothermal and elasto-plastic process. The material of workpiece is SS400 steel which is the same as that used in experiment. The boundary energy dissipation due to heat convection has been taken into account. Bishop's step-wise decoupled strategy is adopted to handle coupling between mechanical deformation and the temperature variation. This technique has been proven to be robust and efficient for large thermal-plastic deformation computation. By adopting this approach only the pure mechanical parts of the weak forms have to be consistently linearized since the coupling terms are held constant during the iteration. In order to avoid locking deficiency that frequently exhibited in classical displace-based finite element method, an improved large deformation mixed finite element method has been used to solve this near-incompressible metal forming problem. The standard Newton-Raphson iteration method together with the corresponding consistent tangent operator has been adopted to solve nonlinear algorithmic equations. In thermal phase, the transient heat transfer finite element method together with the Crank-Nicholson algorithm has been employed to determine the temperature field. The total time for extrusion process is specified to be 0.4 second. Using the numerical result, the effect of temperature distribution on forming limit is discussed. By comparing with the experimental result, it has been revealed that the temperature gradient plays an important role to induce the strain localization and lead to fracture failure in metal material.

4:15 PM
A Model for Calculating the Lankford Value in Sheet Steels: *Sihai Jiao*[1]; *C. Isaac Garcia*[2]; *Anthony J. DeArdo*[2]; [1]Shanghai Baosteel Group Co., Shanghai Baosteel Rsrch. Inst. (R&D Ctr.), 1 Kedong Rd., Baoshan Dist., Shanghai 201900 China; [2]University of Pittsburgh, Basic Metals Proc. Rsrch. Inst., Matls. Sci. & Eng. Dept., 848 Benedum Hall, Pittsburgh, PA 15261 USA

The deep drawing capacity of sheet steels is generally indicated by the Lankford value, which is mainly determined by the texture in steels. Traditionally, various models derived from the Taylor model are employed to calculate the Lankford value from the texture. Due to the complexity of the texture description, the current models could be quite difficult to use and, moreover, a considerable error may be caused by the incompletion of including some trivial texture components. The present work proposes a practical model based on the Hill Equation to calculate the Lankford Value of interstitial free (IF) steels, by considering only a few selected texture components. In IF steels, a new parameter describing texture, the Texture Intensity Ratio (TIR), i.e. (111)/(100), not only affects the average Lankford value, but also has a strong influence on its distribution. By using the new TIR parameter, the texture based prediction models can be used more easily to calculate the Lankford value. The distribution of the Lankford value as function of the angle with respect to the rolling direction seems to be influenced by Mn and P additions. The practical significance of the results from this work will be presented and discussed.

4:40 PM
Interfacial Modelling of Hot Rolling: A Probabilistic Approach: *Sumitesh K. Das*[1]; Eric J. Palmiere[1]; Ian C. Howard[2]; [1]The University of Sheffield, Dept. of Eng. Matls., Sir Robert Hadfield Bldg., Mappin St., Sheffield, S. Yorkshire S1 2JD UK; [2]The University of Sheffield, Dept. of Mechl. Eng., Sir Frederick Mappin Bldg., Mappin St., Sheffield, S. Yorkshire S1 2JD UK

Heat transfer and friction in hot flat rolling have traditionally been characterised by an average heat transfer coefficient (h) and an average friction coefficient (μ). However, the presence of oxide scales and asperities at the interface question the use of such averages. The apparent irregularity and lack of pattern of interactions at the interface has prompted this present investigation to move away from averaged h and μ values, and to study their local evolution based on probabilistic rules. The approach is based on the probability of finding the workpiece surface in a specific configuration at a particular instance of time in the roll gap. These rules are based on observations of surface phenomena such as scale behaviour during hot rolling. The model is implemented using user-subroutines integrated with the commercially available finite element program ABAQUS. The results show the strong dependence of h and μ on the local scale interactions. In particular, the effect of scale thickness, material behaviour and processing conditions on the evolution of h and μ are discussed. Finally, the development of a Cellular Automata based Finite Element (CAFE) model is proposed to model the combined interfacial phenomenon of friction and heat transfer at the tool-work piece interface.

5:05 PM Cancelled
Computational Experiment in the Mechanics of Materials: *Leon Mishnaevsky*

Computational Phase Transformations: Phase Transformations in Multicomponent Systems

Sponsored by: ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Chemistry & Physics of Materials Committee, Jt. Computational Materials Science & Engineering, Thermodynamics & Phase Equilibria Committee
Program Organizers: Long-Qing Chen, Pennsylvania State University, Materials Science and Engineering Department, University Park, PA 16802-5005 USA; Mark Asta, Northwestern University, Department of Materials Science and Engineering, Evanston, IL 60208-3108 USA; Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; James Aaron Warren, NIST, CTCMS and Metallurgy Division, Gaithersburg, MD 20899-8554 USA

Tuesday PM — Room: 201
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Carelyn E. Campbell, NIST, Metall. Div., 100 Bureau Dr. Stop 8555, Gaithersburg, MD 20899-8555 USA

2:00 PM
Application of 3-D Visualization and CALPHAD Techniques on Metastable Pearlite in Fe-C-Mn Steels: *Gary J. Shiflet*[1]; [1]University of Virginia, Matls. Sci. & Eng., Charlottesville, VA 22903 USA

This talk will discuss the metastable formation of parallel plate and rod ferrite/cementite aggregates which form in high Mn steels. One of the unusual aspects of this phase transformation is that the pearlite forms in the austenite + cementite phase field at temperatures well above the Ae1 temperature. Examination of the growth and dissolution kinetics, manganese partitioning and microstructure stability will be reported and analyzed with the CALPHAD technique. Visualization of the three-dimensional microstructure will be presented employing a technique developed at UVA which includes recreating the region of interest as a solid body in the computer. Comparisons will then be made between the similarities of rod and plate pearlite that can occur in the same austenite grain. This work is supported by NSF-DMR.

2:30 PM
Zigzag Diffusion Paths and the "Horns of a Dilemma": K. Wu[2]; Y. Wang[2]; *J. E. Morral*[1]; [1]University of Connecticut, Metall. & Matls. Eng., 97 N. Eagleville Rd., U-136, Storrs, CT 06269-3136 USA; [2]Ohio State University, Matls. Sci. & Eng., 2041 College Rd., Columbus, OH 43210 USA

Diffusion paths are drawn on phase diagrams to characterize the interdiffusion zone of a diffusion couple. In 1994 a theoretical treatment of diffusion paths in two-phase regions of ternary diffusion couples predicted that the paths would follow a linear zigzag course in the vicinity of the initial interface. Major assumptions of the theory were that an "effective diffusivity" could be defined that is constant. This prediction has been confirmed many times by finite difference and phase field calculations, except when the effective diffusivity varies with composition. Then dramatic deviations from the linear zigzag path occur. These deviations appear as opposing "horns" near the initial interface. In recent work it was predicted using perturbation theory that the horns could point in the same direction instead of opposite directions, even though such deviations had never been seen before in experiments or other calculations. These "horns" have created a dilemma, the resolution of which will be discussed.

2:50 PM
Looking for Deep Eutectics in Multicomponent Systems: *Didier*

deFontaine[1]; Nathan Speed[1]; [1]University of California, Matls. Sci. Dept., 577 Evans Hall, Berkeley, CA 94720-1760 USA

The CVM in the pair approximation is used to investigate the influence of interaction parameters in the liquid and solid phases for the purpose of obtaining deep eutectics. For CVM thermodynamic purposes, the liquid is regarded as an n-fold coordinated aggregate with small clustering or ordering short-range order. Phase separation is allowed in the solid, but no "ordering" long range order. Several classes of 2-, 3-, and 4-phase multicomponent equilibria will be presented.

3:10 PM

Effect of Alloying Elements on the γ to α Transformation in Steels: *Joakim Odqvist*[1]; John Ågren[1]; [1]Royal Institute of Technology (KTH), Matls. Sci. & Eng., Stockholm SE-100 44 Sweden

A generalised solute drag model has been applied to the γ to α transformation in Fe-C, Fe-X and Fe-X-C alloys where X represent a substitutional alloy element. In the model thermodynamic and kinetic data available in commercial databanks are used. For the Fe-C the limit for massive transformation is calculated and the result is compared with similar calculations made with a phase-field model. In the case of a binary substitutional alloy, with X=Ni, calculation of the limit for massive transformation is compared with recent experimental results. Furthermore, the effect of alloying elements on the transition between para- quasipara- and ortho-equilibrium in Fe-X-C is discussed.

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Prediction of Precipitation Kinetics in Nb Added Steels: Seung Ho Lee[1]; Joo Ahn Cha[1]; Nam Soo Kim[1]; *Kyung Jong Lee*[1]; [1]Hanyang University, Div. of Matls. Sci. & Eng., HaengDang 17, Seoul 133-791 S. Korea

In Nb, V and Ti added steels, carbo-nitrides are formed due to their strong interaction with C and N. The quantitative analysis of distribution of precipitates and the effect of precipitates on the phase transformation and mechanical properties are still far from satisfactory. In this study, the analysis of precipitates in austenite was investigated using the fact that the formation of precipitates accelerates austenite/ferrite transformation. The formation of precipitates was controlled by adjusting holding temperature and time. Volume fractions transformed were measured by dilatometer. Iso-precipitation kinetics were determined by comparing 5% and 50% volumes transformed at various conditions respectively. The thermodynamic and kinetic models for precipitation in austenite and ferrite as well as austenite/ferrite transformation was formulated. The thermodynamic and kinetic models were based on the sublattice model and on the nucleation and growth model. The result was compared with the calculated.

3:50 PM Break

4:05 PM

Modelling the Austenite Decomposition Kinetics in Low Carbon Steels: *Fateh Fazeli*[1]; Matthias Militzer[1]; [1]UBC, The Ctr. for Metlgcl. Proc. Eng., #309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

A model is developed to describe the overall austenite decomposition kinetics to ferrite and bainite in low carbon steels. The model considers both interface and carbon diffusion-controlled ferrite formation in a mixed-mode approach. Solute drag effects of substitutional elements are considered employing the modified Purdy-Brechet theory. The relevance of solute drag parameters and their quantification are evaluated for Fe-C-Mn alloys. The challenges associated with modelling the bainite formation are delineated. Both diffusional and displacive model approaches are discussed for transformation to carbide-free bainite in Fe-C-Mn-Si steels.

4:25 PM

Application of Computational Thermodynamics and Kinetics to Control of High Mn Ductile Iron Austempering: *M. Nili Ahmadabadi*[1]; [1]University of Tehran, Dept. of Metall. & Matls. Sci., Fac. of Eng., PO Box 11365-4563, Tehran Iran

To provide sufficient hardenability for austempering of ductile iron, alloying elements like Mn, Ni, Cu or Mo are frequently added. But the segregation of these alloying elements, occurring during solidification, changes the kinetics of austenitisation and austempering reaction. In this work the solidification and thermodynamics relations is used to calculate the concentration of alloying elements as a function of intergraphite distance. Using the calculated alloying elements segregation profile, the related thermodynamics equations were used to calculate the Ae3 and A1 phase boundaries in the different regions of matrix. The results show the variation of austenitisation kinetics as a function of alloying elements segregation. Needles to say that as a consequence of alloying elements segregation and variation of austenitisation kinetics, kinetics of austempering should be a function of alloying elements local concentration. To verify the austempering kinetics, three high carbon Si-Mn steel compositions corresponding to three different parts of ductile iron matrix (i.e. near graphite nodules, intercellular regions and the area between these two regions) were prepared. The iron and steel samples were all subjected to different austempering cycles in dilatometry equipment. In addition, to predict time-temperature-transformation diagrams the classical nucleation theory and related equations were used to calculate the time of bainitic start transformation.

4:45 PM

Thermodynamic Modeling of the CaO-MgO-Al2O3-SiO2 System: *In-Ho Jung*[1]; Sergei Degterov[1]; Arthur D. Pelton[1]; [1]Ecole Polytechnique, Matls. Eng., PO Box 6079, Sta. "Downtown", Montreal, Quebec H3C 3A7 Canada

Liquid and solid phases in the CaO-MgO-Al2O3-SiO2 system are important in metallurgy, ceramics and geology. A complete critical evaluation and thermodynamic modeling of the phase diagrams and thermodynamic properties are presented. The modified quasichemical model is used for the molten slag. Extensive solid solutions like pyroxenes, olivine, melilite and spinel are modeled with the Compound Energy Formalism. Optimized equations for the properties of all phases are obtained which reproduce all available thermodynamic and phase equilibrium data within experimental error limits at all temperatures and compositions. The optimized properties and phase diagrams are believed to be the best estimates presently available. The database of the model parameters, along with software for Gibbs energy minimization, can be used to calculate any type of phase diagram section.

5:05 PM

Optimization of the CaO-MnO-SiO_2-Al_2O_3 Quaternary System and its Application to Ferroalloy and Steelmaking Processes: *Youn-Bae Kang*[1]; In-Ho Jung[2]; Sergei Degterov[2]; Hae-Geon Lee[1]; Arthur D. Pelton[2]; [1]Pohang University of Science and Technology, Matls. Sci. & Metlgcl. Eng., San 31, Hyojadong, Namgu, Pohang, Kyungbuk 790-784 Korea; [2]Ecole Polytechnique de Montreal, Centre de Recherche en Calcul Thermochimique, PO Box 6079, Sta. "Downtown", Montreal, Quebec H3C 3A7 Canada

Available thermodynamic and phase diagram data have been critically assessed for the solid and liquid phases of the quaternary system of CaO, MnO, SiO_2 and Al_2O_3. All reliable data have been simultaneously optimized to obtain one set of model parameter for the Gibbs energy of the liquid slag and all solid phases as functions of composition and temperature. The compound energy model was used for the solid phases, and the modified quasi-chemical model was used for the liquid slag. Calculations using FactSage have been carried out to apply this optimization to manganese ferroalloy production and steelmaking processes.

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Simulation of Paraequilibrium Growth in Multicomponent Systems: *Gautam Ghosh*[1]; [1]Northwestern University, Dept. of Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-3108 USA

Computational thermodynamic and kinetic tools, Thermo-Calc and DICTRA (DIffusion Controlled TRAnsformation) softwares, respectively, are used to model the growth kinetics under paraequilibrium condition in several multicomponent alloys. A methodology to simulate the paraequilibrium growth in multicomponent systems using the DICTRA (DIffusion Controlled TRAnsformation) software is proposed. For any given multicomponent system containing substitutional and interstitial elements, the basic approach is to define a hypothetical element Z whose thermodynamic and mobility parameters are expressed in terms of the weighted average (with respect to site fraction) of the thermodynamic parameters and mobilities of the substitutional alloying elements. This procedure facilitates the calculation of paraequilibrium phase diagrams and the paraequilibrium growth simulations directly in Thermo-Calc and DICTRA softwares, respectively. The results of two distinct case studies in multicomponent alloys will be presented. In the first example, we simulate the growth of paraequilibrium cementite. In the second example, we will present the results of paraequilibrium ferrite growth during continuous cooling from an intercritical temperature in an Fe-Al-C-Mn-Si low alloy steel. The results of both simulations are in good accord with experimental results.

Creep Deformation: Fundamentals and Applications: Composites and Engineering Applications

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Materials Processing & Manufacturing Division, Jt. Mechanical Behavior of Materials, Powder Materials Committee
Program Organizers: Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; James C. Earthman, University of California, Department of Chemical and Materials Science, Irvine, CA 92697-2575 USA; Sai V. Raj, NASA Glenn Research Center at Lewis Fields, Cleveland, OH 44135 USA

Tuesday PM Room: 214
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chair: J. Roesler, Technical University, Inst. for Matls., Langer Kamp 8, Braunschweig 38106 Germany

2:00 PM Invited
Dual Scale Particle Strengthened Alloys for High Temperature Applications: Creep Behavior and Fundamentals of Design: *Joachim Roesler*[1]; Cristina Tiziani[1]; Martin Baeker[1]; [1]Technical University, Inst. for Matls., Langer Kamp 8, Braunschweig 38106 Germany

The creep behavior of dual scale particle strengthened alloys containing particles on two different length scales, namely nanometer-size dispersoids and discontinuous reinforcements with meso- or macroscopic dimension, is discussed. Based on existing constitutive equations for dispersion and reinforcement strengthened matrices, a new steady state creep equation for this advanced material class is derived and analyzed. Provided certain design principles, which will be discussed in detail, are obeyed, a synergisitic strengthening effect and a creep strength level superior to today's best particle strengthened alloys is predicted, opening an avenue for the development of future high temperature materials. As practical example, design of dual scale particle strengthened copper is discussed and potential applications are outlined.

2:25 PM Invited
Creep of Ceramic Fibers: Modeling, Mechanisms, and Implications: *James Anthony DiCarlo*[1]; Hee Mann Yun[1]; [1]NASA Glenn Research Center, Matls. Div., 21000 Brookpark Rd., MS 106-5, Cleveland, OH 44135 USA

At the high temperatures of interest for ceramic matrix composites (CMC), current ceramic fibers display time-dependent deformation and fracture, i.e., creep and creep-rupture. Because this behavior can strongly affect CMC life for structural applications, NASA Glenn has performed a variety of studies over recent years to model, mechanistically analyze, and predict the technical implications of ceramic fiber creep. This paper briefly reviews the key results of these studies, with focus on SiC-based small-diameter fibers. It is shown that although the effects of time, temperature, stress, and microstructure can be modeled for creep and rupture of single fibers under simple test conditions, complications can arise due to such factors as fiber thermostructural history, fiber environmental sensitivity, and fiber loading when combined to form multifilament tows within uncracked and cracked ceramic matrices. Nevertheless, correlations have been found between single-fiber and CMC time-dependent behavior, thereby allowing the development of fiber-creep guidelines for CMC structural optimization.

2:50 PM
Elevated Temperature Mechanical Behavior of Diffusion Aluminide Bond Coat Layers: Deng Pan[1]; Michael L. Glynn[1]; Mingwei Chen[1]; *K. J. Hemker*[1]; [1]Johns Hopkins University, Dept. Mechl. Eng., Baltimore, MD 21218 USA

The bond coat layer is a key component of thermal barrier coatings (TBC) because it provides oxidation resistance and a foundation for the ceramic top coat. Plastic deformation of the bond coat governs the development of stresses during thermal cycling and in doing so plays an important role in determining TBC life. The use of standard tensile testing techniques for characterizing diffusion aluminide bond coats has been inhibited by their limited thickness (<60 microns), and a high temperature microsample tensile testing technique has been developed and used to characterize PtAl/NiAl bond coats in the temperature range from 25 to 1150C. The tensile strength and stress relaxation behavior have been measured for both as-deposited and thermally cycled coatings, and the intermediate temperature strength has been found to increase with thermal cycling. TEM observations have linked this increase to the formation of a Ni-rich martensitic microstructure. In situ TEM has also been used to document the loss of the mart ensitic structure at the highest service temperatures, and the influence of microstructure on the elevated temperature behavior of the bond coat will be emphasized. The results from this study provide material inputs for a finite element (FE) analysis of the development of stresses in the multi-layered TBC, and the results of this FE analysis will be used to explain the role of bond coat properties in determining TBC reliability.

3:10 PM
Is Transition-Based Approach Better for Interpretation of Dislocation Creep Mechanisms in Dispersion-Strengthened Materials?: *Rajiv S. Mishra*[1]; Zong-Yi Ma[1]; [1]University of Missouri, Dept. of Metlgcl. Eng., Rolla, MO 65409 USA

Dispersion strengthening is the best way to achieve high creep strength. It is used for almost all high temperature alloys. While the engineering aspects of dispersion-strengthened creep-resistant materials have developed significantly, the fundamental aspects are still not clear. Interpretations of creep mechanisms in dispersion-strengthened materials are frequently debated in literature. For pure metals and alloys, a number of transitions in creep mechanisms have been proposed, experimentally verified and quite well accepted. On the other hand, no theoretical framework has developed for transitions in dislocation creep mechanisms in dispersion-strengthened materials. In this paper, we highlight different experimental trends and microstructural features in dispersion strengthened aluminum alloys and composites. A need for transition-based approach is discussed.

3:30 PM
Creep Deformation and Dislocation Strengthening Mechanisms in a Nb-Al-Ti Base Alloy: *S. M. Allameh*[1]; R. W. Hayes[2]; E. A. Loria[3]; Winston O. Soboyejo[1]; [1]Princeton University, Princeton Matls. Inst. & the Dept. of Mechl. & Aeros. Eng., Engineering Quadrangle, Olden St., Princeton, NJ 08544 USA; [2]Metals Technology, Inc., 19801 Nordhoff St., Northridge, CA 91324 USA; [3]Reference Metals Company, Inc., 1000 Old Pond Rd., Bridgeville, PA 15017 USA

This paper presents the results of a study of the creep behavior of a multicomponent precipitation-hardened body-centered-cubic 44Nb-35Ti-6Al-5Cr-8V-1W-0.5Mo-0.5Hf-0.3C alloy (compositions quoted in atomic % unless stated otherwise). The alloy is shown to exhibit inverted creep in the primary creep regime, and an extended tertiary regime at 704°C. Furthermore, two-stage secondary creep behavior is also observed at 704°C. The dislocation substructures associated with creep deformation are evaluated by transmission electron microscopy. The observed dislocation/particle interactions are then discussed within the context of dislocation strengthening.

3:50 PM
Creep Cavitation and Growth Behavior of Bridged Crack: *S. B. Biner*[1]; [1]Ames Laboratory, Iowa State University, 208 Metals Development, Ames, IA 50011 USA

In this study growth behavior of bridged cracks, resulting from the growth of pre-nucleated creep cavities with diffusional and dislocation assisted mechanisms, is investigated numerically. The results indicate that the bridging traction significantly relaxes even with the creep deformation of the composite alone. The rate of this relaxation is not influenced by the rate of crack growth. However, the rate of change in the bridging zone length or the density of the bridging elements in the bridging zone strongly affects both the maximum value and the distribution of the traction in the bridging zone. A much weaker stress singularity than the ones described by K or C* was found ahead of the bridged cracks in the creep regime. In this weak singularity region the cavities grow at comparably high rates to each other. This work was performed for the United States Department of Energy by Iowa State University under contract W-7405-Eng-82.

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Mechanical and Microstructural Differences Between Uniaxial and Multiaxial Creep Loading of Short Fibre Reinforced Aluminium Alloys: *Alejandro Andres Yawny*[1]; Gregor Kausträter[1]; Birgit Skrotzki[1]; Gunther Eggeler[1]; [1]Ruhr-Universität Bochum, Institut für Werkstoffe, Bochum 44801 Germany

In the present work a short fibre reinforced aluminium alloy is subjected to uniaxial creep (tension and compression) and to shear creep loading. The creep behaviour was analysed in terms of the shape of individual creep curves and in terms of the stress and the temperature dependence of the secondary creep rate. Fibre reinforcing results in a decrease of creep rate under all loading conditions as compared to the unreinforced matrix material. Fibres are shown to strongly influence

the overall stress and temperature dependence of the creep process. And there are cases where the preferential orientation of fibres affects creep deformation. These findings are discussed in terms of the kinetics of the underlying elementary deformation and damage processes. Special emphasis is placed on the breakage of fibres which occurs under all creep conditions. Fibre length distributions shift to smaller values as creep strain accumulates (mean values: initial state - 110, after creep - 45 micro meter).

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Creep of Silicon Nitride/Silicon Carbide Ceramic Nanocomposites: *Matt J. Gasch*[1]; *Julin Wan*[1]; Kenneth C. Liu[2]; Edgar Lara-Curzio[2]; Amiya K. Mukherjee[1]; [1]University of California-Davis, Dept. of Cheml. Eng. & Matls. Sci., One Shields Ave., Davis, CA 95616 USA; [2]Oak Ridge National Laboratory, Bldg. 4515, MS-6069, 1 Bethel Valley Rd., Oak Ridge, TN 37831-6069 USA

In comparison to conventional ceramic sintering techniques, polymer precursors offer new methods for making silicon nitride/silicon carbide ceramic composites with microstructural features not attainable by hot pressing. Pyrolysis-derived amorphous powders prepared either by Fe or WC ball milling were Electric Field Assisted Sintered (EFAS), with oxide additives, in 10 minutes at 1600C. Sintering of such powders results in microstructures with a matrix of 200-300nm silicon nitride grains amongst nanometric silicon carbide grains and a small amount of residual amorphous phase. Depending on powder processing method, high temperature mechanical testing of consolidated specimens illustrates creep rates down to 3.5x10-9 at 1400C and 100MPa. The rate parameters for creep were established from mechanical tests. Microstructures, prior to and post creep testing, were examined with TEM in order to shed light on the rate controlling creep mechanisms. This research is sponsored by US Office of Naval Research Grant #N00014-00-1-0186.

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A Hierarchical Approach for the Creep Modeling of 9-12% Cr Steels: *P. Weinert*[1]; B. Buchmayr[1]; [1]Institute for Materials Science, Welding & Forming, Graz Univ. of Tech., Kopernikusgasse 24, A-8010 Graz Austria

In this contribution a hierarchical creep model for the description of the creep behaviour of complex ferritic/martensitic 9-12%Cr steels over all creep regimes is introduced. For the microstructural induced creep the model of Ghoniem et al. has been refined and extended, considering the essential changes in the microstructure and the influence of different second phases. To model the microstructural changes and the related strain-rate behaviour during the uniaxial testing of those materials, accepted concepts and physical formulations for the processes have been used. Due to the general character of the microstructure model related phenomena like recovery can be modelled too. To extend the modelling to the tertiary creep regime damage formulations have been incorporated with special focus on the continuous nucleation of intragranular pores and the growth in the constrained mode according to the observed kinetic of damage under typical test conditions. For the elasto-mechanical influence of damage, a Kachanov type formulation has been used. To consider instability phenomena and effects on a creep specimen, iterative elasto/visco-plastic finite element formulations have been used for the description of the thereby altered elastic stresses and deformations. Verification calculations on Cr steels are shown. Finally a critical review of the model is performed in order to evaluate the applicability.

5:20 PM

Properties of Matching Filler Metals for P91, E911, and P92: *Russel Fuchs*[1]; Herbert Heuser[2]; Claus Jochum[2]; [1]Bohler Thyssen Welding USA, Inc., PO Box 721678, Houston, TX 77272-1678 USA; [2]Thyssen Schweisstechnik GmbH, Wilhelmstrasse 2, Hamm 59067 Germany

It is the endeavour of operators of fossil fuel fired power stations to increase the degree of efficiency of their installations through an increase in steam pressure and temperature. For this reason new creep resistant steels have been developed in Europe and Japan, which allow the use of operating temperatures from 600-625°C. The steel developed in Europe is E 911 with 9% chrome, 1% molybdenum and 1% tungsten. The Japanese steel is known under the name Nf 616 or under the American name P 92 (9% chrome, 2% tungsten). At the same time as the development of these materials, matching weld metals were also developed. The new steels are already being used in new power stations (Alvedøre and Niederaußem). In this paper the results of a research project for the developing of matching filler metals are being presented. The starting point of the investigations were the ranges of chemical composition and minimum requirements for mechanical properties of both the parent metals E 911 and P 92. At the time of reporting, trial results for long-term investigations up to 20,000 hours for the all weld metal and 30,000 hours for the weld joints were available. With these investigations the highest level of certainty regarding the estimated life of creep resistant steel welded components in power stations has been gained.

David L. Davidson Symposium on Fatigue: Material Design for Fatigue Performance

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Jt. Mechanical Behavior of Materials, High Temperature Alloys Committee
Program Organizers: Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Richard S. Bellows, Solar Turbines, Inc., Materials and Process Engineering, San Diego, CA 92186-5376 USA; Peter K. Liaw, University of Tennessee, Department of Materials Science and Engineering, Knoxville, TN 37996-2200 USA; Winston O. Soboyejo, Princeton University, Department of Mechanical Aerospace Engineering, Princeton, NJ 08544 USA; Thomas Zogas, Carpenter Technology Corporation, Reading, PA 19612-4662 USA

Tuesday PM — Room: 208
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Tom Zogas, Carpenter Technology Corporation, PO Box 14662, Reading, PA 19612 USA

2:00 PM Invited

Fatigue in AeroPropulsion Engines: *Howard F. Merrick*[1]; Youri N. Lenets[1]; Krish Krishnamurthy[1]; [1]Honeywell Engines, Systems & Services, 111 S. 34th St., Phoenix, AZ 85034 USA

Of the possible failure modes in gas turbine engine components, fatigue is one that is of particular concern to designers. Fatigue induced failure may be due to engine mission cycles (LCF) or instabilities in the flow of air through the various components in the engine (HCF). As temperatures rise in the engine, issues of fatigue/creep interaction begin to emerge. While good design can minimize the effects of fatigue by keeping stresses and temperatures low, the continuing drive for better performance and efficiency in aeropropulsion engines requires materials that can operate at higher temperatures under large cyclical stresses. Thus, the ability of the materials engineer to optimize a component's microstructure to meet fatigue requirements is of paramount importance. In this paper we will review the nature of fatigue in gas turbine engine components and examples of materials commonly used. The role of microstructure will be examined in relation to fatigue crack initiation and growth. The application of surface treatment to impart compressive residual stresses to mitigate fatigue will also be examined.

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Low Cycle Fatigue Behavior of Cast GTD222 Weld Joint: *Ganjiang Feng*[1]; Dan Nowak[1]; [1]General Electric, 1 River Rd., 55-127, Schenectady, NY 12345 USA

GTD222 is a precipitation-hardenable nickel-base superalloy developed by GE for large power generation gas turbine applications. While the alloy possesses good creep and fatigue strength at high temperature, it is essentially weldable by various welding processes. In the present study, low cycle fatigue (LCF) behavior of cast GTD222 weld joints generated by electron beam (EB) welding process and tungsten inert gas (TIG) welding process were studied. The effect of filler materials, post weld heat treatment and hot isostatic pressing (hipping) on the LCF lives of cast GTD222 weld joints were discussed. While LCF lives are strong function of welding processes and welding defect, post weld heat-treat and the resulting microstructures play important role to achieve an optimized weld joint properties.

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Microstructure Effects on Fatigue and Dwell-Fatigue Crack Growth in Three Alpha/Beta Ti-6242 Alloys: *Weimin Shen*[1]; *Wole Soboyejo*[1]; Alfred B. Soboyejo[2]; [1]Princeton University, Dept. of Mechl. & Aeros. Eng., Princeton, NJ 08540 USA; [2]The Ohio State University, Dept. of Food, Agricultl. & Biologl. Eng., Columbus, OH 43210 USA

This paper presents the results of a combined experimental and analytical study of fatigue crack growth and dwell-fatigue crack growth in three forged alpha/beta Ti-6242 alloys. Following an initial characterization of microstructures and basic mechanical properties, the micromechanisms of long fatigue crack growth are presented for three microstructures. These include: a duplex structure, an elongated struc-

ture, and a colony microstructure. The colony microstructure is shown to have the best resistance to fatigue crack growth. The elongated structure has intermediate resistance, while the equiaxed structure exhibits the fastest fatigue crack growth rates. The fatigue crack growth rates in the near-threshold, Paris and high regimes are then characterized with empirical crack growth laws that relate the crack growth rates to the stress intensity factor range and key parameters on the fatigue crack growth curve. Finally, the results of dwell-fatigue crack growth experiments are presented for the three microstructures. The dwell-fatigue crack growth rates are shown to be almost identical to the fatigue crack growth rates in the intermediate regime. However, the fatigue crack growth rates are faster at higher stress intensity factor ranges. The underlying mechanisms of dwell crack growth are compared with the mechanisms of fatigue crack growth before discussing the implications of the work for the prediction of dwell or fatigue crack growth in Ti-6242. The effects of different frequencies and crack closure on dwell fatigue are also explored.

3:10 PM
Improvement of Creep-Fatigue Resistance by the Modification of Grain Boundary Carbides in an AISI 304 Stainless Steel: *Hyun Uk Hong*[1]; *Soo Woo Nam*[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., 373-1 Kusong-dong, Yusong-gu, Taejon 305-701 S. Korea

The modification of carbides through grain boundary serration and its subsequent effect on the creep-fatigue property at 873K have been investigated in an AISI 304 stainless steel. It was found that the grain boundaries are considerably serrated when a specimen is furnace-cooled. The grain boundary serration leads to a change of the grain boundary carbide characteristics as well as grain boundary configuration, i.e. the carbide morphology from an acute triangular to a planar form, the lowered density and an array of carbide particles from consistent to zigzag pattern when carbides select one grain between two neighboring grains to share the coherency. Planar carbides on serrated grain boundaries have a lower interfacial energy than that of triangular carbides on straight grain boundaries. From the result of tests, it is suggested that the modification of grain boundary carbides has a remarkable influence on the improvement of creep-fatigue resistance.

3:30 PM Break

3:45 PM
Predicting Fatigue Properties of Cast Aluminum by Characterizing the Behavior of Small Fatigue Cracks: *Michael J. Caton*[1]; John E. Allison[2]; J. Wayne Jones[3]; [1]US Air Force Research Laboratory, AFRL/MLLM, Wright-Patterson AFB, OH 45433 USA; [2]Ford Motor Company, Ford Research Lab., Dearborn, MI 48124-2053 USA; [3]University of Michigan, Matls. Sci. & Eng., 2300 Hayward, Ann Arbor, MI 48109-2136 USA

The increased use of cast aluminum in structural components necessitates a deeper understanding of the mechanisms controlling fatigue properties in order to enable improved predictive capabilities. Of particular interest is the ability to model the influence of processing variables on the fatigue performance of alloys used in automotive applications such as engine blocks and cylinder heads. This presentation will outline a study conducted on cast W319 aluminum, a commercial Al-Si-Cu alloy used in automotive engine components. The initiation and propagation of small fatigue cracks (~5 μm to 2 mm) has been studied in this alloy and a model has been developed to predict fatigue properties as a function of critical processing parameters. An ultrasonic testing method was used to determine the fatigue behavior in the very high cycle regime (~10^9 cycles). Fatigue cracks were observed to initiate almost immediately even at stresses where failure did not occur within 10^8-10^9 cycles. Endurance limits were observed for all solidification conditions of the W319 alloy resulting from the arrest of small cracks. A modeling approach for predicting the fatigue properties of cast aluminum based upon a small-crack threshold and small-crack growth correlation is presented.

4:05 PM
Effects of Microstructure and Micro-Texture on Dwell-Fatigue Susceptibility of a Near-Alpha Titanium Alloy: *V. Sinha*[1]; B. Rollins[1]; M. J. Mills[1]; R. B. Schwarz[2]; J. C. Williams[1]; [1]The Ohio State University, Dept. of Matls. Sci. & Eng., 477 Watts Hall, 2041 College Rd., Columbus, OH 43210 USA; [2]Los Alamos National Laboratory, Structure/Properties Relations Grp., MST-8, MS G755, Los Alamos, NM 87545 USA

Ti-6Al-2Sn-4Zr-2Mo-0.1Si (Ti-6242) alloy is quite extensively used in the compressor section as multi-stage rotors or spools of aeroengines. A significant reduction in the room-temperature, high-stress fatigue life of this class of alloys has been reported in the literature under dwell-fatigue conditions when compared with that under continuous cycling (normal-fatigue) conditions. We have investigated the effects of microstructure on the fatigue and dwell-fatigue response of Ti-6242. Micro-texture is shown to be a key variable in dwell-fatigue susceptibility of these near-α titanium alloys. Micro-texture analysis of three different α/β forged pancakes has been performed using electron backscatter diffraction techniques. The failure modes and associated fractographic features under normal-fatigue and dwell-fatigue conditions are distinct. Some preliminary results on the influence of hydrogen on dwell-fatigue susceptibility of these alloys will be presented. (This work is being supported by The Federal Aviation Administration).

Deformation and Stresses in Small Volumes: Deformation and Testing

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials
Program Organizers: David F. Bahr, Washington State University, Department of Mechanical & Materials Engineering, Pullman, WA 99164-2920 USA; Eric Kvam, Purdue University, School of Materials Engineering, West Lafayette, IN 47907-1289 USA; Scott X. Mao, University of Pittsburgh, Department of Mechanical Engineering, Pittsburgh, PA 15261 USA; Neville R. Moody, Sandia National Laboratories, Livermore, CA 94551-0969 USA

Tuesday PM — Room: 303
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: David F. Bahr, Washington State University, Mechl. & Matls. Eng., PO Box 642920, Pullman, WA 99164-2920 USA

2:00 PM Invited
Nanoscale Surface Mechanical Property Measurement using Force Modulation Technique: *Asif Syed*[1]; K. J. Wahl[2]; [1]Hysitron, Inc., 5251 W. 73rd St., Minneapolis, MN 55439 USA; [2]Naval Research Laboratory, Code 6170, Washington, DC 20375 USA

Quantitative study of the mechanical properties of materials at the nanoscale has received much attention in recent years. For submicron scale mechanical property measurement, depth sensing nanoindentation techniques are very successful and gaining much attention. However, due to poor surface sensitivity, difficulty in characterizing the tip shape, unknown thermal drift and floor noise, measuring the quantitative mechanical properties below 10 nm-length scale is extremely difficult. In this presentation we show that combining force modulation with depth-sensing nanoindentation allows measurement of the mechanical properties of materials on the nanometer scale. The stiffness sensitivity of the technique is ~0.1 N/m, which is sufficient to detect long-range surface forces and locate the surface of compliant materials. The tip-surface interaction during approach to contact, asperity deformation during contact and time-dependent deformation at the atomic scale can all be studied. Force modulation can be extended in the form of a dynamic nanoscale Johnson-Kendall-Roberts (JKR) test, to examine adhesive contacts in polymers and thin films. We also present a novel quantitative stiffness imaging technique, which can be used to map directly, the mechanical properties of materials with sub-micron lateral resolution.

2:30 PM
Indentation Technique to Investigate the Micro- and Nano-Scale Response of Small-Volume Structures: *T. A. Venkatesh*[1]; Subra Suresh[1]; [1]Massachusetts Institute of Technology, Matls. Sci. & Eng., 8-139, 77 Massachusetts Ave., Cambridge, MA 02139 USA

Small-volume structures such as thin films and patterned lines on substrates, and micro- and nano- electro-mechanical systems, collectively represent a growing field with a large potential for far-reaching technological impact. In this work, we present a classification of miniature structures based on their geometry (1-D, 2-D or 3-D), structural environment (unconfined, partially confined or fully confined), and coupling characteristics (pure mechanical, piezoelectric or magnetostrictive). This framework is developed with the objective of addressing the uncoupled and coupled mechanical response at the micro- or nano-scale in a unified fashion. In parallel with this classification, a broad and complementary classification of experimental techniques that enables the extraction of the fundamental mechanical properties of materials is also presented. Key results of the various methods for (elastic, plastic, fracture and fatigue) property extraction are reviewed, and the advantages and limitations of each method are analyzed. Particular emphasis is placed on recent advances in the use of instru-

mented indentation as a technique for the assessment of the micro- and nano-scale response of small volume structures.

2:50 PM
Stereoimaging as a Technique for Displacement Measurements with High Spatial Resolution: *David L. Davidson*[1]; [1]Southwest Research Institute, San Antonio, TX USA

The stereoimaging technique was developed specifically to measure displacements with high spacial resolution. The technique has been used for 20 years to measure material response at the tips of cracks, strain in fibers in composites and tensile samples, the deformation associated with twinning, and many other applications. The technique compares two photographs made within the SEM under two different states, such as two loads or two temperatures. The effect of loading can be visualized and measurements can be made. The in-plane displacements can be measured by machine vision or by photogrammetric techniques. Three elements of the symmetric strain tensor can be derived, and from these the principal strains and the maximum shear strain can be computed. The technique will be described and illustrations in fracture, bone mechanics, and microelectronics will be given.

3:10 PM
Characterization and Prediction of Elastic Moduli of Gold Bonding Wire for Fine Pitch Packaging Applications: *Kyongsub Kim*[1]; Soon Hyung Hong[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., 373-1 Kusung-dong, Yusung-gu, Taejon 305-701 Korea

The gold bonding wire for ULSI packaging applications having diameter ranged 20μm ~30μm was fabricated by cold drawing and annealing processes. The mechanical properties such as elastic modulus, ultimate tensile strength, yields strength and elongation were characterized using a micro-tensile test facility. The texture of gold bonding wire was characterized using the calculated orientation distribution function from the measurement of x-ray diffraction pole figure. The drawn gold wire showed very strong (111) deformation texture parallel to the drawing axis. The elastic constant of gold bonding wire was measured higher than that of common gold materials parallel to the drawing axis. Elastic moduli could be calculated from the integration of the orientation distribution function of gold bonding wire. The measured elastic moduli showed good agreement with the calculated elastic moduli from the theoretical models.

3:30 PM Break

4:00 PM
Study of the Yielding and Strain Hardening Behavior of CU Thin Films on SI Substrates using Microbeam Bending: *Jeffrey N. Florando*[1]; William D. Nix[1]; [1]Stanford University, Matls. Sci. & Eng., 416 Escondido Mall, Bldg. 550, Stanford, CA 94305 USA

There is a continuing need for the development of new techniques for studying the mechanical properties of thin films on substrates. Recently a new microbeam bending technique utilizing triangular beams was introduced. For this geometry, the film on top of the beam deforms uniformly when the beams are deflected, unlike the standard rectangular geometry in which the bending is concentrated at the support. This uniform strain allows for the prediction of the stress-strain relation for the film while attached to its substrate. Utilizing this technique, the yielding and strain hardening behavior of bare Cu thin films has been investigated. Specifically, since the Cu films have a dual texture, an analysis of the onset of yielding in the different orientations has been preformed. Also, the effect of microstructure and film thickness on the yield properties of the film has also been studied.

4:20 PM
Characterization of an Optical Sapphire Fiber Embedded in an Aluminum Cladding using Synchrotron X-Ray Micro-diffraction: *Jay C. Hanan*[1]; Ersan Üstündag[1]; C. Can Aydiner[1]; Steffen K. Kaldor[2]; I. Cevdet Noyan[2]; [1]Caltech, Matls. Sci., 138-78, Pasadena, CA 91125 USA; [2]IBM, Rsrch. Div., T. J. Watson Rsrch. Ctr., Yorktown Heights, NY 10598 USA

Analysis of strains on a microscopic scale using diffraction presumes a continuity in the diffracting medium rarely available in most engineering materials. As an alternative to powder diffraction techniques, single crystal diffraction allows strain measurements at scales less than a μm. Microbeam X-ray diffraction was used to topograph a 70 mm radius optical fiber of sapphire with and without an aluminum cladding. The measurements reveal a significant shear stress gradient along the fiber resulting from coefficient of thermal expansion mismatch between the fiber and cladding. Similarly, observed variations in the quality of the aluminum cladding resulted in discernible stress variations within the fiber.

4:40 PM
In Situ Deformation Study of Metallic Thin Films and Multilayers by X-Ray Diffraction: *Philippe Goudeau*[1]; Pascale Villain[1]; Pierre-Olivier Renault[1]; K. F. Badawi[1]; [1]Universite de Poitiers-CNRS, Lab. de Metallurgie Physique, SP2MI, Blvd. Marie et Pierre Curie, BP 30179, Futuroscope, Chasseneuil 86962 France

Polycrystalline thin films deposited by ion beam sputtering techniques on non epitaxial substrates often exhibit very small grain size which may induce particular mechanical properties. These thin films are characterized by high residual stress state which can be related to a large number of grain boundaries and high defect densities. Due this particular microstructure, the elastic constants of the films such as Young's modulus and Poisson's ratio may differ from the bulk material ones which are more often used for evaluating the x-ray stresses in thin films and for modeling thin film mechanical behavior. In the present study, in situ tensile testing have been performed in an x-ray diffractometer to analyze the elastic properties of supported thin films and multilayers with very low period thicknesses. Experiments have been done on the beam line H10 of the French synchrotron facility LURE-Orsay. The studied thin metallic layers have been deposited on polyimide substrates.

Fatigue and Creep of Metal Matrix Composites: Applications of Metal Matrix Composites

Sponsored by: Structural Materials Division, Jt. Composite Materials Committee
Program Organizers: Nikhilesh Chawla, Arizona State University, Department of Chemical and Materials Engineering, Materials Science and Engineering Program, Tempe, AZ 85287-6006 USA; John J. Lewandowski, Case Western Reserve University, Department of Materials Science and Engineering, Cleveland, OH 44106 USA

Tuesday PM — Room: 206
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Herve Deve, 3M, 3M Ctr., Bldg. 60-1N-01, St. Paul, MN USA; Martin Kearns, The Aluminium Powder Company Ltd., Forge Ln., Minworth, Sutton Coldfield, W. Midlands B76 1AH UK

2:00 PM Introductory Remarks

2:10 PM Keynote
Durability Considerations of Aluminum-Based Metal Matrix Composites for Automotive Applications: *Russell Chernenkoff*[1]; [1]Ford Research Laboratories, Mfg. Sys. Dept., MD3135, Rm. 2022B, 2101 Village Rd., Dearborn, MI 48124 USA

Cost effective aluminum-based metal matrix composites are being developed as a replacement material for ferrous-based components in automotive engine and transmission applications to meet the continued demand for lighter weight, more fuel-efficient vehicles. Particular attention is being given to optimizing material properties in terms of strength and durability. Recent advancements in material development, component manufacturing techniques, and post-treatment processes have been instrumental in improving component durability. This presentation provides an overview of aluminum-based metal matrix composites for automotive applications and addresses the durability issues of an engine and a transmission component currently under development.

2:50 PM Invited
Fatigue and Creep Behaviour of SiCp-Reinforced Powder Metallurgy Metal Matrix Composites: *Martin Anthony Kearns*[1]; Andrew Tarrant[2]; [1]The Aluminium Powder Company, Ltd., Forge Ln., Minworth, Sutton Coldfield, W. Midlands B76 1AH UK; [2]Aerospace Metal Composites, Ltd., RAE Rd., Farnborough, Hampshire GU14 6XE UK

Particulate MMCs offer a number of attractive properties for high performance structural applications where high strength and stiffness are required. In typical hot service environments it is also important to display good fatigue and creep properties to make durable parts. Examples are drawn from aluminium pMMC developments to illustrate the choice of matrix chemistry, particulate type and loading to achieve the best combination of properties for given applications. Precipitation hardening systems based on AlCuMgMn and dispersion hardening systems based on AlFeVSi systems are reviewed. Rotating bend test and fatigue crack growth data are presented and rationalised in terms of reinforcement type and level. Creep data are discussed in

relation to the inherent thermal stability of the parent matrix. It is shown that for a given concentration of stiff reinforcement to achieve primary property goals (stiffness, strength, ductility), the size and distribution of the reinforcing phase is critical in determining the level of fatigue and creep performance. Moreover, it is demonstrated that efficient dispersion by mechanical alloying, gives superior properties compared with simple blending.

3:20 PM Invited
Fiber Reinforced Aluminum Matrix Composites in Overhead Transmission Lines and Automotive Applications: *Herve E. Deve*[1]; [1]3M, 3M Ctr., Bldg. 60-1N-01, St Paul, MN USA

Fiber reinforced aluminum matrix composites are emerging as enabling materials for applications in power transmission lines and in the automotive industry. While a number of changes have taken place in the power utility industry in the last century, the conductors used to transmit power continue to be produced from essentially the same basic steel and aluminum constituents. At the same time, the needs of the industry have changed and this change has created new needs. A new generation of overhead conductors has been introduced based upon the fundamental property changes offered by aluminum matrix composites. Continuous aluminum composite wires have been specifically developed for use in high temperature overhead conductor applications. They offers a range of attractive properties including reduced thermal expansion characteristics, improved strength to weight ratio and conductivity improvements. The value associated with the use of composite conductors depend upon the end application and economic benefits. For example, aluminum composite conductors can be used to re-conductor existing lines to double or triple the capacity without having to rebuild or reinforced the towers. The strength and stiffness of fiber composites can also be utilized to reduce component weight in automotive applications. For example, a new aluminum metal-matrix composite brake caliper is being developed to replace the traditional cast-iron brake calipers used in vehicles. The selectively reinforced brake calipers enable up to 50% weight reduction while maintaining strength and stiffness and without increasing the overall dimensions. Traditional cast iron brake calipers can weight 2.7 kilograms each in a small car or up to 14 kilograms each in a truck. The new composite brake calipers decrease weight as well as vibration; the value is increased fuel efficiency and better handling performance.

3:50 PM Break

4:10 PM Keynote
Metal Matrix Composites for Aerospace Systems: *Daniel B. Miracle*[1]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., 2230 Tenth St., Wright-Patterson AFB, OH 45433 USA

Metal matrix composites (MMC's) have been used in the aerospace industry for over 20 years, in applications which range from simple secondary structures to fracture critical flight components. An overview of representative aerospace MMC applications will be provided in this presentation. Components produced from continuously- and discontinuously-reinforced MMC's in the space, aerostructures and aeropropulsion markets will be described. Potential future applications will be defined, and strategies for material development, certification and insertion will be highlighted. Concepts for improved MMC's which are required to achieve these next applications will be described.

4:50 PM Invited
Metal Matrix Composites for Thermal Management Applications: *Warren Hunt*[1]; [1]ThermAluminum Materials, LLC, 4530 William Penn Hwy., Ste. 3900, Murrysville, PA 15668-2002 USA

Metal matrix composites, particularly those based on aluminum matrices, have found increasing application in the electronics packaging field due to the combination of tailorable coefficient of thermal expansion along with light weight and good thermal conductivity that these materials exhibit. This overview presentation will describe the types of metal matrix composites available for commercial exploitation, their manufacturing processes, and properties.

Fundamentals of Advanced Materials for Energy Conversion: Batteries

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee
Program Organizers: Renato G. Bautista, University of Nevada-Reno, Department of Chemical and Metal Engineering, Reno, NV 89557-0136 USA; Dhanesh Chandra, University of Nevada-Reno, Metallurgical & Materials Engineering, Reno, NV 89557 USA

Tuesday PM — Room: 613
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Annick Percheron Guegan, CNRS, LCMTR/UPR209, 2 rue Henri Dunant, Thiais 94230 France; Nobuhiro Kuriyama, National Institute of Advanced Industrial Science and Technology, Special Div. of Green Life Tech., AIST Kansai, 1-8-31 Midorigaoka, Ikeda, Osaka 563-8577 Japan

2:00 PM Plenary
Influence of Stoichiometry and Substitution on the Structural and Electrochemical Properties of AB5 Alloys used as Negative Electrode for Ni-MH Battery: *Annick Percheron-Guegan*[1]; *Michel Latroche*[1]; Jean-Marc Joubert[1]; [1]CNRS, LCMTR/UPR209, 2 rue Henri Dunant, Thiais 94230 France

Nowadays Ni-MH batteries replace widely Ni-Cd ones in the worldwide market of portable tools. The negative electrodes of such batteries are made of LaNi5 type alloys and improved materials have been obtained from pseudo-binary phases such as MmNi3.55Mn0.4Al0.3Co0.75. Lanthanum was replaced by mishmetal(Mm) for cost reasons. Mn, Al, Co have substituted Nickel to improve thermodynamical properties and cycle life. We will present the influence of stoichiometry and composition on the electrode degradation behaviour. First, by in situ powder neutron diffraction the appearance of an intermediate gamma phase during the charge for some alloys was observed. This involves a two step process in the alpha to beta transformation allowing a significant reduction for the decrepitation and subsequent increased corrosion. Second, by high resolution X-ray powder diffraction, intense anisotropic line broadening was observed and interpreted in terms of dislocations whose nature and densities depend on the substituant and play also a role on the decrepitation.

2:30 PM Invited
Improvement of Electrochemical Properties of LiMn2O4 Cathode for a Rechargeable Lithium Battery by Surface Coating with LiNi1-XCoXO2: *Jai-Young Lee*[1]; Sung-Chul Park[1]; [1]Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., Kusong-dong 373-1, Yusung-gu, Taejon 305-701 S. Korea

The surface of LiMn2O4 was coated with LiNi1-XCoXO2(X = 0.2 and 1) by a chemical method in order to improve its high temperature performance and rate capability. The high temperature (65C) storage and cyclic properties of LiMn2O4 were notably improved by coating its surface with LiNi1-XCoXO2. The improvement of elevated temperature performances can be attributed to the suppression of electrolyte decomposition on the surface of LiMn2O4 and the restraint of Mn dissolution. LiNi1-XCoXO2 (X = 0.2 and 1)-coated LiMn2O4 has a better rate capability than as-received LiMn2O4. EIS analysis explains that the improvement of rate capability of LiNi1-XCoXO2 (X = 0.2 and 1) is due to the suppression of 1st arc and 2nd arc in EIS profile which results from the decrease of passivation layer that acts as an electronic insulating layer and higher electrical conductivity of LiNi1-XCoXO2.

3:00 PM Invited
Transmission Electron Microscopy Studies of the Structure and Bonding in $LiCoO_2$: *B. Fultz*[1]; J. Graetz[1]; H. Gabrisch[1]; R. Yazami[1]; C. C. Ahn[1]; [1]California Institute of Technology, Eng. & Appl. Sci., Mail 138-78, Pasadena, CA 91125 USA

Commercial cathodes for "Li-ion" rechargeable batteries use Li_xCoO_2 for their ability to insert and deinsert Li. As x changes between 0.5 and 1, these materials undergo significant changes in electronic and atomic structure. We are studying these changes by transmission electron microscopy. In samples with various amounts of Li, electron energy loss spectrometry was used to measure the K-edge of oxygen atoms, and the $L_{2,3}$-edge of cobalt. These measurements showed the largest changes in electronic structure around the oxygen atoms, consistent with electronic structure calculations on Li_xCoO_2. The cobalt $L_{2,3}$-edge showed changes that were small but measurable. In studies of defect structures of $LiCoO_2$, we have identified dislocations and partial dislo-

cations on the basal plane of the hexagonal structure. From the separation of the partial dislocations we can estimate the energy difference between the fcc and hexagonal forms of $LiCoO_2$. [This work sponsored by US DOE DE-FG03-00ER15035.]

3:25 PM Invited
Activation Behavior of AB2-Type Metal Hydride Electrodes: *Nobuhiro Kuriyama*[1]; Yasuko Endo[1]; Tetsuo Sakai[1]; Hiroyuki T. Takeshita[1]; Hideaki Tanaka[1]; Itsuki Uehara[1]; [1]National Institute of Advanced Industrial Science and Technology, Special Div. of Green Life Tech., AIST Kansai, 1-8-31 Midorigaoka, Ikeda, Osaka 563-8577 Japan

Electrochemical activation behavior of an AB2-type hydrogen storage alloy, Zr0.9Ti0.1Ni1.1Co0.1Mn0.6V0.2, was studied to make clear activation process of pellet-type metal hydride electrodes made of mixture of alloy and Cu powder. An alkaline treatment of the electrodes with heated alkaline solution gave alloy surface enriched with nickel, and the treated electrode was more easily activated. The treated electrode desorbed more hydrogen during the first charge-discharge cycle, and suggested larger surface area based on impedance spectroscopy. A cross sectional view of the activated electrode showed many cracks formed in the Cu matrix. Since the cracks formed in the electrode by expansion of alloy particles with hydrogenation are considered to improve contact between electrolyte and alloy particles, improvement of initial activity of the alloy surface by the alkaline treatment accelerates pulverization of the alloy particles in the electrode and increases utilization of them.

3:50 PM Break

4:05 PM
Mg-Si and Mg-Sn Anode Materials for Lithium-Ion Batteries: Gregory A. Roberts[1]; *Elton J. Cairns*[1]; Jeffrey A. Reimer[1]; [1]Lawrence Berkeley National Lab, Environmental Energy Tech. Div., One Cyclotron Rd., MS 70-108B, Berkeley, CA 94720 USA

Magnesium-based silicides and stannides are possible alternatives to graphite for use as negative electrode materials in lithium-ion batteries. Mechanical alloying was used to synthesize these alloys with fine particle sizes and nanocrystalline microstructures (20-30 nm crystallites). Three synthesis products, cubic Mg2Si, cubic Mg2Sn, and rhombohedral Mg2Sn, were characterized by x-ray diffraction and microscopy techniques. Cycling over wide voltage windows of 0-1 V vs. Li/Li+ at room temperature produces lithium capacities in excess of graphite's capacity, but the large capacities fade rapidly during cycling. Cycling over smaller voltage windows produces smaller discharge capacities that are stable during cycling. Mg2Sn electrodes with nanocrystalline microstructures have shown larger capacities and better kinetics than the annealed, crystalline electrodes. Electrochemical techniques and x-ray diffraction have been used to examine the structural changes that accompany lithium insertion and to investigate capacity-loss mechanisms during cycling.

4:25 PM
Charge-Discharge Processes of Misch Metal-Based Hydrogen Storage Alloys at Relatively Low Temperatures: *Hiroshi Senoh*[1]; Nobuhiro Kuriyama[2]; Kohji Morimoto[1]; Yasutaka Hara[1]; Hiroshi Inoue[1]; Chiaki Iwakura[1]; [1]Osaka Prefecture University, Dept. of Appl. Chem., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan; [2]National Institute of Advanced Industrial Science and Technology, Special Div. of Green Life Tech., 1-8-31 Midorigaoka, Ikeda, Osaka 563-8577 Japan

For the purpose of clarifying the reason caused a drastic decrease of discharge capacity for $MmNi_{3.6}Mn_{0.4}Al_{0.3}Co_{0.7}$ electrode at relatively low temperatures, various factors influencing the charge-discharge processes were investigated in the temperature range of 25°C to -40°C. The charge efficiency obviously decreased at low temperatures less than 0°C due to a decrease in the rate of the charge-transfer reaction on the electrode surface. Moreover, both the rate of the hydrogen diffusion from the alloy bulk to surface and the rate of the charge-transfer reaction significantly influenced the high-rate dischargeability. The addition of Mo to $MmNi_{3.6}Mn_{0.4}Al_{0.3}Co_{0.7}$ alloy was found to be effective for improving the electrocatalytic activity, leading to the improvements of the charge efficiency and the high-rate dischargeability at relatively low temperatures.

4:45 PM
Manganese Oxide Composites as Electrode Material for Electrochemical Capacitors: *Ravinder Reddy Nagireddy*[1]; Ramana G. Reddy[1]; [1]The University of Alabama, Metlgcl. & Matls. Eng., A129 Bevill Bldg., PO Box 870202, Tuscaloosa, AL 35487 USA

The Pseudo capacitance of layered type MnO2 electrode material for electrochemical capacitors (EC's) was investigated. MnO2 was synthesized in our laboratory. MnO2 was prepared from heating of KMnO4 at 300°C in air. Cyclic Voltammetry (CV) experiments were carried out using three electrode system, Saturated calomel electrode as reference electrode, platinum mesh as a counter electrode, and MnO2 composite material mounted in a platinum mesh used as a working electrode. Two kinds of composite working electrode materials were used, one was mixture of MnO2 and 25 wt% acetylene black, while the other was MnO2, 25 wt% acetylene black and 25 wt% carbon. All the experiments were carried out at 25°C. A maximum specific capacitance of 130 F/g and 118 F/g were observed at the scan rate of 2 mV/s and 20 mV/s respectively in 1M KCl for composite electrode made with carbon. Results obtained in this study are compared with that of other electrode materials.

5:05 PM
Measurement of Hydrogen by Magnetic and Electronic Techniques in Metallic Materials: Preecha Termsuksawad[1]; Saisamoirn Niyomsoan[1]; *Brajendra Mishra*[1]; David L. Olson[1]; Zamir Gavra[1]; Ron B. Goldfarb[2]; [1]Colorado School of Mines, Metlgcl. & Matls. Eng., Golden, CO 80401 USA; [2]National Institute of Standards and Technology, Electromagnetic Tech. Div., 814.05, Boulder, CO 80303-3328 USA

The ability of advanced hydrogen storage materials as well as nickel metal-hydride battery materials to absorb and desorb hydrogen has been measured using magnetic and electronic techniques. These techniques provide a unique sensing tool for available hydrogen in these materials. The ability of absorption and desorption of hydrogen has been correlated with the d-shell and f-shell electronic interactions with hydrogen as an electron donor. The tendency of the metallic materials, therefore, to dissolve hydrogen as well as to form hydride compounds has been established on the basis of donor and acceptor concepts. Experimental measurements include magnetic properties, electrical resistivity and thermoelectric [Seebeck] coefficient in different materials as a function of hydrogen content and temperature. Ingress and egress diffusivities have been measured along with the thermal differential analyses of the uncharged and charged samples. Hydrogen Charging through gas phase at various temperatures for different lengths of time and at different pressures was accomplished. Cathodic charging was also used for bulk materials. Gas-chromatography was used to determine the content of hydrogen in samples as well as the weight change. The as-received samples were baked in argon environment at 400°C for 3 hours, before any charging of hydrogen gas. High temperature charging was avoided in these samples to prevent any structural change in the material.

5:25 PM
Effects of Dissolution and Exsolution of Ni in YSZ: *Soren Linderoth*[1]; Nikos Bonanos[1]; [1]Risoe National Laboratory, Matls. Rsrch. Dept., DK-4000 Roskilde, Denmark

The effect of the dissolution of Ni in zirconia, doped with about 8 mol% yttria, has been investigated with respect to sinterability, grain growth, solubility and electric conductivity. The solubility is in the 1 at% range and the addition of such small amounts of NiO causes the zirconia to sinter at temperatures about 65°C lower than without NiO present. The grains become about twice as large. At higher dopant levels the NiO inhibits grain growth. The conductivity at low temperatures is lowered when adding NiO, while at 850°C the effect is found to be positive up to the solubility limit. Upon reduction the conductivity drops dramatically.

Fundamentals of Structural Intermetallics: Advanced Intermetallics & Oxidation

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Jt. Mechanical Behavior of Materials
Program Organizers: Young-Won Kim, UES, Inc., Materials & Processing Division, Dayton, OH 45432 USA; Kwai S. Chan, Southwest Research Institute, Department of Materials Science, San Antonio, TX 78284 USA; Vijay K. Vasudevan, University of Cincinnati, Department of Materials Science and Engineering, Cincinnati, OH 45221-0012 USA

Tuesday PM — Room: 615-616
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Joachim H. Schneibel, Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6115 USA; Mahesh C. Chaturvedi, University of Manitoba, Mechl. & Indl. Eng, 356 Engineering Bldg., 15 Gibson St., Winnipeg, Manitoba R3T 5V6 Canada

2:00 PM Invited
Mechanical Properties of Ir-Nb-Zr Alloys as Ultra-High Tem-

perature Materials: *Yoko Yamabe-Mitarai*[1]; Hiroshi Harada[1]; [1]National Institute for Materials Science, High Temp. Matls. Rsrch. Grp., Sengen 1-2-1, Tsukuba, Ibaraki 305-0047 Japan

The Ir-based alloys with the fcc and $L1_2$ two-phase structure has been developed as next-generation high-temperature materials under high temperature materials 21 project. Some of the binary alloys showed superior strength, however, the strength above 1200°C was not remarkable considering their melting temperatures. Creep properties of binary alloys up to 1500°C are also very promising because their minimum creep rate at 1500°C are between 10^{-7} and 10^{-6}/s and the tertiary creep was not observed until 300 hours. However, above 1600°C, the minimum creep rate increased 10^{-5}/s and the tertiary creep was observed after 10 hours. To improve high temperature strength above 1500°C and creep properties above 1600°C, third element addition has been tried for binary alloys. Recently we found the minimum creep rate of Ir-Nb-Zr alloy with the fcc and $L1_2$ two-phase structure at 1800°C was 10^{-6}/s. Deformation mechani sm of Ir-based alloys was investigated in binary Ir-Nb, Ir-Zr and ternary Ir-Nb-Zr alloys. The deformation mechanism will be discussed in terms of precipitate morphology, lattice misfit, and grain boundary strength. Then the way for Ir-based alloy design as high-temperature materials will be pointed out.

2:30 PM

Microstructures and Mechanical Properties of NiAl-Re Alloys: Ralf Rablbauer[1]; Johannes Deges[1]; *André Schneider*[1]; Georg Frommeyer[1]; [1]Max-Planck-Institut für Eisenforschung GmbH, Matls. Tech., Max-Planck-Str. 1, Duesseldorf 40237 Germany

The B2-ordered intermetallic NiAl phase forms with the refractory metal Re a quasi-binary eutectic system. The microstructures of as cast ingots with Re concentrations up to 1.8 at% consist of NiAl(Re) primary crystals in between a network of fibrous NiAl-Re eutectic. The as cast hypoeutectic $Ni_{49.5}Al_{49.5}Re_1$ alloy shows a remarkable high temperature strength of 120MPa at 1200°C due to an effective particle strengthening caused by finely dispersed Re-particles in the primary solidified NiAl(Re) solid solution. The microstructures of the alloys in the as cast condition and after annealing have been studied by atom probe field microscopy (APFIM) and transmission electron microscopy (TEM). A perfectly formed uniaxial eutectic microstructure with a Re concentration of 1.25 at% was achieved by unidirectional solidification using Bridgeman technique. This fibrous microstructure exhibits an high thermal stability up to 1300°C, and the creep stresses reveal high stress exponents of n=16.

2:50 PM

Microstructure and Deformation of an Ir-Rich Ir3Nb (L12) Alloy at Ultra-High Temperature: *Y. F. Gu*[1]; *Y. Yamabe-Mitarai*[1]; *S. Nakazawa*[1]; *H. Harada*[1]; [1]National Institute for Materials Science, 1-2-1 Sengen, Tsukuba-shi, Ibaraki 305-0047 Japan

Recently, we proposed a new class of superalloys, called refractory superalloy, based on platinum group metals (PGMs), which have coherent fcc-L12 two-phase structure, high melting temperature, and good potential as structural materials used at temperature up to 1800°C. In the refractory superalloys, the precipitation hardening caused by precipitates with L12 structure play an important role in keeping the alloy strength, such as Ni3Al phase in Ni-based superalloy. However, little information about mechanical properties and deformation structures for these precipitates at ultra-high temperature is known now. In this research, we investigated the deformation behaviours in one of these precipitates, Ir-rich Ir3Nb (L12) alloy, at temperatures from 1650°C to 1800°C. The dislocation structures due to compression deformation and creep deformation at the ultra-high temperatures will be compared. The deformation mechanisms for the alloy in different test conditions (compression and creep) will be discussed.

3:10 PM

Thermal Fatigue of NiAl and NiAl-Base Eutectic Alloys: M. T. Kush[2]; R. O. Urbance[3]; J. W. Holmes[4]; *R. Gibala*[1]; [1]University of Michigan, Dept. of Matls. Sci. & Eng., Ann Arbor, MI 48109-2136 USA; [2]Rolls-Royce Corporation, Indianapolis, IN 46206-0420 USA; [3]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., Cambridge, MA 02139 USA; [4]Pennsylvania State University, University Park, PA 16802 USA

Single crystals of [001]-oriented NiAl and directionally-solidified single-crystal eutectic composites of NiAl-Mo and NiAl-Cr alloys were subjected to thermal fatigue stress environments in an argon atmosphere. The specific technique employed circumferential induction heating, holding at temperature, and cooling of disk-shaped specimens such that substantially asymmetric thermoelastic stress-strain cycles could be imposed for thousands of repetitions. Several time-temperature heating and cooling profiles, mainly between 973 K and 1473 K, were used to produce different thermal strain histories. The thermal fatigue behaviors of these materials were found to be controlled by combinations of the following factors: (a) shape changes associated with the elastic anisotropy of the NiAl matrix; (b) differential cyclic strain accumulation (thermal ratcheting) associated with the basic test method; (c) internal stresses associated with the CTE mismatch between the NiAl matrix and the bcc metal fibers; (d) the details of the alloy microstructure produced by directional solidification. This research was supported by the AFOSR MURI Program, Grant No. F49620-93-1-0289.

3:30 PM

A Study of Alloying at the Electronic Level in TiAl and MoSi2 Base Alloys: *Panos Tsakiropoulos*[1]; Spyros Diplas[1]; Aristidis Arvanitis[1]; John Watts[1]; [1]University of Surrey, Matls. Sci. & Eng., Guildford, Surrey GU2 7XH England

Alloy phase formation at the atomic scale involves coexistence of atoms from two or more different elements in the same lattice accompanied with electronic changes in the valence configurations, which lead to the formation of new bonds or modification of the existing ones. Thus, the crystal structure may be modified or completely altered and these modifications may trigger microstructural changes with significant impact on the properties and performance of the alloys formed. This interplay between atomic structure « crystal structure « microstructure « material performance justifies the increasing necessity for a better understanding of alloying at the electronic level. Our contribution will be reporting and discussing results of our on going experimental research towards understanding the phenomena taking place at the atomic and sub-atomic level upon alloying via information obtained by electron spectroscopy and to relate these observations to the microstructures of alloys of the Ti-Al-V and Mo-Si-Al systems.

3:50 PM

The Effect of Ion-Implantation on Oxidation Resistance of TiAl: *Michiko Yoshihara*[1]; Shigeji Taniguchi[2]; Yao-Can Zhu[3]; [1]Yokohama National University, Mechl. Eng. & Matls. Sci., 79-5 Tokiwadai, Hodogaya-ku, Yokohama, Kanagawa 240-8501 Japan; [2]Osaka University, Matls. Sci. & Proc., Grad. Sch. of Eng., 2-1 Yamadaoka, Suita, Osaka 565-0871 Japan; [3]Ion Engineering Research Institute Corporation, 3rd Proj. Rsrch. Lab., 2-8-1, Tuda-yamate, Hirakata, Osaka 573-0128 Japan

The ion implantation was applied to Gamma TiAl alloys, because it is a useful method for surface modification without changing the properties of the matrix. The oxidation resistance of the alloys implanted with various species was investigated through cyclic oxidation test at 1200K in a flow of purified oxygen under atmospheric pressure. The implanted species were selected from wide range of the elements. Metallographic examinations were performed for implanted specimens and oxidized specimens using XRD, AES, SEM and EPMA. The implantation of Nb, Mo, Ta or W resulted in significant improvement of the oxidation resistance with the formation of protective alumina layer, whereas the implantation of B or V enhanced scale spallation resulted in deteriorated oxidation resistance. The results will be discussed in detail.

4:10 PM Invited

Fundamental Considerations for the Development of Oxidation Resistant Alloys and Coatings Based on Gamma-TiAl: *Lorenz Singheiser*[1]; Leszek Niewolak[1]; Willem Joseph Quadakkers[1]; Vladimir Shemet[1]; Udo Flesch[2]; [1]Research Centre Juelich, IWV 2, Juelich, NRW 52425 Germany; [2]RWTH Aachen, Inst. fuer Anorganische Chemie, Professor-Pirlet-Strasse 1, Aachen, NRW 52074 Germany

Alloys on the basis of gamma-TiAl are promising high-temperature materials that may replace conventional heat-resistant steels and superalloys in applications where high strength in combination with low density is required. However, an important hindrance for the use of gamma-TiAl alloys at high temperatures is their relatively poor oxidation resistance and sensitivity against environmentally induced embrittlement. This material degradation is related to the poor protective properties of the mixed TiO2/Al2O3 surface scales which form on the surface during high temperature exposure. The reasons, why gamma-TiAl alloys do generally not form protective alumina scales are two-fold: (i) in the sub-surface zone beneath the external oxide scale Al depletion results in formation of alpha2-Ti3Al. The high oxygen solubility (up to 20 At.-%) of this phase results in a rapid inward diffusion of oxygen into the alloy so that the Al tends to oxidize internally rather than to form an external scale. (ii) during air exposure Ti-rich nitrides tend to be formed at the scale/alloy interface thereby impeding the development of a continuous alumina layer. The oxidation resistance of gamma-TiAl alloys can be improved by adding alloying elements which either decrease the growth rate of the mixed

oxide or which promote the formation of a protective alumina scale. The alloying element most frequently used to reduce the growth rate of the mixed oxide is Nb. A substantial decrease in oxide growth rate can be obtained by suitable additions of this element, whereby the oxidation rate in air decreases with increasing Nb content. A similar positive effect has been reported for additions of W and Ta. It should be mentioned that oxygen induced embrittlement and sub-scale nitride formation are not prevented by the mentioned alloying additions. Recently it was shown that protective alumina scale formation on gamma-TiAl can be obtained by small additions of Ag. This effect was found to be related to formation of the so-called Z-Phase in the sub-scale depletion layer at the expense of alpha2-Ti3Al. It was however found, that the beneficial effect of Ag can be suppressed if the alloys contain additionally alpha2-stabilizing elements, such as Nb, as is the case for most (semi-)commercial, high strength alloys. Therefore, recent efforts concentrated on developing the Ag containing x-TiAl alloy as oxidation resistant coatings for high-strength titanium aluminides. First results using magnetron sputtering have shown, that due to the similarity in chemical and physical properties of coating and base material, the Ag containing material offers promising possibilities to be qualified as coating material for reducing the oxidation induced degradation of titanium aluminides and perhaps also to prevent the oxygen embrittlement of Ti and Ti alloys.

4:40 PM
Oxidation and TEM Characterization of Ti-44Al-xNb-2(Ta,Zr) Alloys: *John Woo*[1]; Rabindra N. Mahapatra[2]; Shailendra K. Varma[1]; [1]The University of Texas at El Paso, Dept. of Metlgcl. & Matls. Eng., El Paso, TX 79968 USA; [2]Naval Air Warfare Center, Aircraft Div., Patuxent River, MD 20650 USA

Ti-44Al-xNb-2(Ta, Zr), x=9 and 11, alloys have been subjected to oxidation treatment in air at 900 and 1000°C for a week. Results indicate an improvement in resistance to oxidation, over and above that was obtained by adding 9 or 11 atomic percent Nb to Ti-44Al, by the addition of Ta but it is degraded by Zr. Ellingham diagram (graph between free energy and temperature) confirms this observation. Identification of the nature of oxides formed in the scale has been characterized by EDX in the SEM. TEM analysis of the microstructures developed during the treatments will be compared with the Ti-44Al-xNb alloys in order to determine the effect of quaternary addition of Ta or Zr on the phase transformations taking place during the oxidation process.

5:00 PM
Computation and Validation of Weld Pool Temperature Profiles for Gamma TiAl: *Kirti Bisen*[1]; Mario Arenas[1]; Viola L. Acoff[1]; [1]The University of Alabama, Metlgcl. & Matls. Eng., Box 870202, Tuscaloosa, AL 35487-0202 USA

Previous research by the authors has shown that the welding current has a strong effect on the weld properties and microstructures of gamma TiAl. To calculate the temperature profiles associated with spot welding gamma TiAl, the governing equations describing conductive, convective and phase change processes in the solid, liquid and mushy regions of a metal were used. The computed temperature fields predicted that as the welding current is increased, the maximum temperature reached in the weld pool also increases. Experimental validation of the computed temperature fields was determined by placing thermocouples at different locations on the welded specimen to record the temperatures during welding using computer-based data acquisition hardware and software. The relationship between maximum weld pool temperature, microstructure, and mechanical properties will be established which should lead to better control of gamma TiAl weld pool properties.

5:20 PM
An Optimal Structure and High Mechanical Properties of Titanium Aluminides: *B. A. Greenberg*[1]; *V. V. Rybin*[2]; *N. V. Kazantseva*[1]; [1]Institute of Metal Physics Ural Division RAS, 18 S.Kovalevskaya, GSP-170, Ekaterinburg 620219 Russia; [2]Central Research Institute of Structural Materials «Prometey», 49, Shpalernya, St. Petersburg 193015 Russia

The structure of a lot of Ti-48at.%Al-1at.%V samples was analyzed. The samples were prepared by the method of pulsed forging under different conditions and did not have a texture or a dendrite structure characteristic of cast TiAl and Ti3Al. The alloy cast in a copper mold under a gas pressure had high strength characteristics: the microhardness 4000 MPa, the fracture stress 1088 MPa, and plasticity 32.6%. The alloy prepared by this method contained two ordered phases TiAl and Ti3Al, was in a polycrystalline state, and had a structure approaching an oriented lamellar structure. The alloy consisted of uniform equiaxial grains with an average size of about 40 mkm. The obtained data were compared with the strength characteristics of an orthorhombic Ti-22at.%Al-26.6at.%Nb alloy, which was melted in an arc furnace under an argon atmosphere. The last alloy had a two-dimensional modulated basket-like structure. The strength characteristics of the orthorhombic alloy were a maximum for the 3-phase composition, proof fracture stress 1319 MPa, and plasticity 26%. The strength characteristics of the alloy with the orthorhombic phases only were as follows: proof fracture stress 1381 MPa, and plasticity 19%.

High Performance Metallic Materials for Cost Sensitive Applications: Titanium Alloys - III

Sponsored by: Structural Materials Division, Structural Materials Committee, Titanium Committee
Program Organizers: Edward Y. Chen, TiTech International, Inc., Pomona, CA 91768 USA; Rod Boyer, Boeing Commercial Airplane Group, Seattle, WA 98124-2207 USA; F. H. (Sam) Froes, University of Idaho, Institute of Materials and Advanced Processes, Moscow, ID 83844-3026 USA; Eric M. Taleff, University of Texas, Mechanical Engineering Department, Austin, TX 78712-1063 USA

Tuesday PM — Room: 213
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Larry LaVoie, Titanium Products, Inc., Newport, OR 97365 USA; Steve J. Gerdemann, Albany Research Center-DOE, Albany, OR 97321 USA

2:00 PM Invited
Making Titanium Affordable for Combat Vehicles: *F. Robert Dax*[1]; Oscar Yu[2]; Steve Luckowski[3]; [1]Concurrent Technologies Corporation, 425 6th Ave., Regional Enterprise Tower, 28th Fl., Pittsburgh, PA 15219 USA; [2]RMI Titanium Company, 1000 Warren Ave., PO Box 269, Niles, OH 44446-0269 USA; [3]US Army, ARDEC AMSTA AR WEA, Bldg. 355, Picatinny Arsenal, NJ 07003 USA

Titanium alloys have not been widely used in combat vehicles due to their high cost. Recently, the U.S. Army and Marine Corps have been looking at ways to both lower the weight of combat vehicles and decrease the total cost of ownership. These goals have directed the Military to look increasingly at using titanium and its alloys for various applications. However, the affordability of titanium still limits its use. In order to address this issue, the military has targeted single melt practices as a means for reducing the procurement cost of titanium. This paper will present efforts of Army and Marine Corps regarding the use of Plasma Arc Melting (PAM) in place of double and triple melt Vacuum Arc Remelting (VAR) to increase the scrap usage.

2:30 PM Invited
Fabrication of Cost Affordable Components for US Army Systems: *V. S. Moxson*[1]; J. Qazi[2]; F. Sun[2]; F. H. (Sam) Froes[2]; J. Montgomery[3]; [1]ADMA Products, Inc., 8180 Boyle Pkwy., Twinsburg, OH 44087 USA; [2]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [3]Army Research Laboratory, Aberdeen Proving Ground, MD 21005-5069 USA

Titanium is an attractive replacement for steel in US Army Systems such as armored vehicles and engine components where weight is a concern. However, the high cost of titanium has slowed implementation. In the work to be presented, a powder metallurgy near net shape approach to fabrication of parts will be described. Various powder sources have been evaluated and compacts produced by a novel loose sintering technique, in some cases followed by a working operation. Microstructures and mechanical properties will be reported, included ballistic behavior of materials with various microstructures.

3:00 PM Invited
A Comparison of Commercial and Experimental Titanium Powders: *Steve J. Gerdemann*[1]; David E. Alman[1]; [1]Albany Research Center-DOE, 1450 Queen Ave. S.W., Albany, OR 97321 USA

Titanium powder can be made by a number of processes. These differences have a large impact on the powder's price and physical and chemical properties. Recently several new processes have been announced that purport to make less expensive titanium powder. This paper will compare of commercial atomized and titanium powders with titanium powders made by newer processes including Idaho Titanium Technologies, International Titanium Powder and hydride-dehydride of Titanium Chips (ADMA). The chemistry, shape and physical properties of vacuum sintered buttons and dilatometry of these

powders will be measured. Some thoughts on the potential of titanium powder metallurgy and other processes will also be offered.

3:30 PM
Cost Effective Synthesis of Ti-6Al-4V Alloy Components Produced via the P/M Approach: *O. M. Ivasishin*[1]; D. G. Savvakin[1]; F. H. (Sam) Froes[2]; V. S. Moxson[3]; K. A. Bondereva[1]; A. N. Demidik[1]; [1]National Academy of Science of Ukraine, Inst. for Metal Phys., 36 Vernadsky St., Kiev 03142 Ukraine; [2]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA; [3]ADMA Products, Inc., 8180 Boyle Pkwy., Twinsburg, OH 44087 USA

The near net shape powder metallurgy (P/M) approach to production of titanium components allows a substantial widening of the field of applications of titanium alloy, reducing the cost of parts compared to traditional processes. The most cost-effective P/M process is based on the use of blended elemental (BE) powders of titanium and masteralloys as input materials. In this paper, the P/M BE technique will be reviewed including shape making capabilities, mechanical properties and cost of parts. Examples of components, which have been produced for industries such as automobiles and aerospace, will be given.

4:00 PM Break

4:20 PM Invited
Titanium Powder Injection Molding (Part I): *John Lombardi*[1]; J. Fravel[2]; L. LaVoie[3]; M. Godfrey[2]; F. H. (Sam) Froes[4]; [1]Ventana Research Company, 831 N. Camino Miramonte, Tucson, AZ 85716 USA; [2]Titanium Manufacturing, Inc., 3019 W. Windsor Ave., Phoenix, AZ 85009 USA; [3]Titanium Products, Inc., 3503 S. Coast Hwy., Newport, OR 97365 USA; [4]University of Idaho, Inst. for Matls. & Adv. Proc., Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

An approach to reducing the cost of titanium components is by near net shape technologies. In this paper, a powder injection molding approach will be discussed. A major concern here is the binder, which is used as titanium and is extremely reactive and can easily be contaminated. A new source of powder will be discussed and approaches to compounding this with low contaminating binders will be presented.

4:40 PM Invited
Titanium Powder Injection Molding (Part II): *Larry LaVoie*[1]; J. Fravel[2]; J. Lombardi[3]; M. Godfrey[2]; F. H. (Sam) Froes[4]; [1]Titanium Products, Inc., 3503 S. Coast Hwy., Newport, OR 97365 USA; [2]Titanium Manufacturing, Inc., 3019 W. Windsor Ave., Phoenix, AZ 85009 USA; [3]Ventana Research Company, 831 N. Camino Miramonte, Tuscon, AZ 85716 USA; [4]University of Idaho, Inst. for Matls. & Adv. Proc. (IMAP), Mines Bldg., Rm. 321, Moscow, ID 83844-3026 USA

In the second part of this presentation, the results of producing complex shapes using a new titanium binder combination will be presented. This will include an analysis of the dimensional capabilities of the process, the basic microstructure and the resultant mechanical properties. Comparisons will be drawn between the product of this new process and results from the literature.

5:00 PM Invited
Cold Spray Process for Cost-Sensitive Applications: *Anatolii N. Papyrin*[1]; [1]Ktech Corporation, Eng. Dvlp. Dept., 2201 Buena Vista S.E., Ste. 400, Albuquerque, NM 87106-4265 USA

Cold Spray is a material deposition process in which a coating is formed by exposing a substrate to a high-velocity jet of solid-phase particles. This paper presents an overview of some results of recent studies in the field of Cold Spray. Principles of operation and basic features of Cold Spray as well as its advantages for cost-sensitive applications are discussed. Various technologies including technologies of spraying aluminum and titanium are described. It is shown that Cold Spray can be successfully used for direct fabrication of near net shape parts.

Hume-Rothery Award Symposium: CALPHAD and Alloy Thermodynamics: Applications of Computational Thermodynamics

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee
Program Organizers: Patrice E.A. Turchi, Lawrence Livermore National Laboratory, Materials Science and Technology Division, Livermore, CA 94551 USA; Antonios Gonis, Lawrence Livermore National Laboratory, Livermore, CA 94551-0808 USA; Robert D. Shull, NIST, Magnetic Materials, Boyds, MD 20841-9015 USA

Tuesday PM — Room: 204
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Robert D. Shull, National Institute of Standards and Technology, 100 Bureau Dr., Gaithersburg, MD 20899-8552; Bo Sundman, Royal Institute of Technology, Dept. of Matls. Sci. & Eng., Stockholm 10044 Sweden

2:00 PM Invited
An Industrial Perspective on the Use of Phase Diagrams: *Karin Frisk*[1]; Björn Uhrenius[2]; [1]Swedish Institute for Metals Research, Drottning Kristinas väg 48, Stockholm SE-11428 Sweden; [2]Sandvik Hard Materials, Stockholm S-12680 Sweden

The development of steels and alloys and the choice of their heat treatments has always been dependent on the access to reliable phase diagrams. Still in the 1970's phase diagrams were drawn by hand and were often based on experimental sources which were not easily assessed. During following years the more efficient computers made phase diagram calculations possible and opened up for the use of thermodynamic models to describe Gibbs energies of multi component systems. The CALPHAD approach to link experimental information and phase diagram evaluations created a tool which became powerful also in the industrial environment. Results of such phase diagram calculations are now widely spread. A few examples are given to show how these phase diagrams made it possible to describe areas for the heat treatments of stainless steel and how to improve the development work of new cemented carbides.

2:30 PM Invited
Understanding Magnesium Alloys: A Computational Approach: *Zi-Kui Liu*[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., 209 Steidle Bldg., University Park, PA 16802 USA

About thirty years ago, Dr. Kaufman pioneered the CALPHAD approach of computational thermodynamics. This approach has not only extended the horizon of classic thermodynamics, but also created the foundation for today's system materials design. In the present work, both aspects will be discussed in connection with magnesium alloys. As the lightest metallic structural material, magnesium has a great potential in weight-reduction of automotives. One of the significant challenges for a wider application of magnesium alloys is the low creep temperature, typically below 100°C. It is well known that rare-earth alloying elements can significantly improve the creep behavior of magnesium alloys, but their high costs prevent the application in massive production of automotives. In this presentation, the effects of alkaline-earth alloy elements are explored through the combination of the CALPHAD approach, combinatory/diffusion-triple investigations, and experimental investigation of individual alloys. This project is supported by the National Science Foundation under the grant DMR-9983532.

3:00 PM Invited
Thermodynamic Calculations Applied to Devitrified Al-Ni-Gd Metallic Glass: *Michael C. Gao*[1]; Gary J. Shiflet[1]; [1]University of Virginia, Dept. of Matls. Sci. & Eng., 116 Engineer's Way, Charlottesville, VA 22904 USA

Based on phase equilibria studies and thermal analysis results, a self-consistent thermodynamic database was developed for Al-Ni-Gd metallic glass system using the CALPHAD approach. The driving forces for nucleation of crystalline phases are calculated and compared with experimental results. The crystallization processes are extensively studied in this study, using XRD, DSC, TEM, analytical TEM and high-resolution TEM techniques. It was found that, depending on alloy composition, either fcc Al nanocrystalline or the ternary compound phase will be formed first during primary crystallization. Isothermal DSC study revealed that Al nanocrystalline precipitation may proceed via a growth stage only, while the formation of the ternary phase

occurs via a nucleation and growth process. Another interesting discovery was that the ternary phase changes its morphology from rods to irregular particles in different tie triangles. Topics concerning glass forming ability, nucleation driving forces and optimal control of the devitrification path will be addressed.

3:30 PM Break

4:00 PM Invited
The Role of Computational Thermodynamics in the Design of Duplex Stainless Steels: *Frederick Henry Hayes*[1]; [1]UMIST, Matls. Sci. Ctr., Grosvenor St., Manchester, England M1 7HS UK

Duplex stainless steels, developed over the past 20 years, are an important class of engineering materials, mainly due to their high resistance to stress corrosion cracking and their good weldability. These materials are based on the austenite + ferrite two-phase region of the Fe-Cr-Ni system, stable in the range 900-1400C. Overall compositions are selected to give a 50-50 phase balance at the solution annealing temperature prior to quenching. Further alloying elements e.g. Mo, W, Cu and N are added to enhance corrosion and mechanical properties. However, these additions can also result in the formation of stable intermetallic phases such as sigma, chi, Laves and nitrides, which can cause embrittlement and reduce pitting resistance. This paper shows how Calphad computations of phase equilibria for multicomponent duplex and superduplex stainless steels enable stable duplex composition ranges and processing windows to be derived.

4:30 PM Invited
Applications of Computational Thermodynamics to Welding: *John M. Vitek*[1]; Suresh S. Babu[1]; Stan A. David[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Bldg. 4508, MS 6096, Oak Ridge, TN 37831-6096 USA

Computational thermodynamics and the Calphad method, pioneered by Larry Kaufman, offer the potential of describing phase stability in multi-component systems as a function of alloy composition and temperature. Such information is critical for predicting weld microstructures, including the fusion zone, where base metal dilution with filler metals takes place, and the heat-affected zone, where high temperature exposure alters the base metal microstructure. When combined with diffusion-controlled kinetics analyses, computational thermodynamics can describe the evolution of microstructure in the entire weldment. This paper will review the numerous applications of computational thermodynamics in welding, including calculations of phase stability, solidification behavior, microsegregation effects, and inclusion formation. Examples of these calculations for predicting microstructure and properties of weldments in a wide range of materials, including steels and nickel-base superalloys, will be provided. This research was sponsored by the Division of Materials Science and Engineering, US Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle.

Imaging of Dynamic Processes - II

Sponsored by: Extraction & Processing Division, Materials Processing & Manufacturing Division, Jt. Processing Modeling Analysis & Control Committee
Program Organizer: Iver Anderson, Iowa State University, Ames Laboratory, Ames, IA 50011-3020 USA

Tuesday PM — Room: 310
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Ralph E. Napolitano, Ames Laboratory, Metall. & Cer., 104 Wilhelm, Ames, IA 50011 USA

2:00 PM Invited
Imaging Spatial Heat Flow and Dynamic Instabilities in Melt Spinning: *Matthew J. Kramer*[1]; Ralph E. Napolitano[1]; Halim Meco[1]; Matthew Sawka[1]; Kevin W. Dennis[1]; R. William McCallum[1]; [1]Iowa State University, Ames Lab., 37 Wilhelm, Ames, IA 50011 USA

Stop-action digital photography is being used to analyze the time-temperature evolution of the melt jet and melt pool of a single roller free jet melt spinner. Images were obtained using a 12 bit 640 x 480 pixel charged coupled device (CCD) camera with full frame rate of up to 50 frames/sec and a 200 µm shutter speed. From the images perpendicular to the wheel axis, the dynamic variability in length, height and area of the melt-pool are obtained as a function of time. A second CCD camera looking down on the surface of the wheel approximately 45° between the vertical crucible the flight path of the ribbon is used to measure temperature by a split beam two-color narrow band pass filter process and image reconstruction. The combinations of these images are being used to provide the boundary conditions necessary for thermal and solidification modeling.

2:30 PM
The Use of High Speed Imaging for Thermomechanical Characterization of Melt Pool Dynamics during Rapid Solidification: *H. Meco*[1]; M. J. Kramer[2]; R. E. Napolitano[1]; M. Sawka[2]; K. W. Dennis[2]; R. W. McCallum[2]; [1]Ames Laboratory, US DOE Metall. & Cer. Prog., 235 Wilhelm Hall, Ames, IA 50011 USA; [2]Iowa State University, Ames Lab, 37 Wilhelm, Ames, IA 50011 USA

For single roller free jet melt spinning, the geometry and temperature distributions within the melt-pool were measured dynamically using a high-speed charged coupled device (CCD) imaging system. Images were obtained using a 12 bit 640 x 480 pixel CCD camera with full frame rate of up to 50 frames/sec and a 200 µs shutter speed. The effects of process variables such as wheel speed, chamber atmosphere and quench media (i.e. wheel material) on melt-pool geometry and temperature profiles were investigated for three alloys, namely Fe-Si-B, Nd-Fe-B and Sm-Fe. The factors which influence melt pool stability and shape will be discussed in detail. Making use of the correlations between processing variables and measurements on melt-pool geometry and thermal profiles, fluid flow and heat transfer characteristics of the chill-block melt spinning process is discussed.

3:00 PM Invited
Schlieren Imaging in Materials Processing: *Steven P. Mates*[1]; [1]NIST, Metall. Div., 100 Bureau Dr., MS 8556, Gaithersburg, MD 20899-8556 USA

Even before Ernst Mach used it to visualize shock waves cast by a supersonic bullet in 1888, the schlieren optical technique has been a valuable diagnostic tool in fluid mechanics. It remains particularly useful in the fields of aerodynamics and heat convection, where strong density gradients in the flow are able to produce high-contrast schlieren images. Recent advances in high-speed, high resolution CCD imaging technology have facilitated high quality imaging of dynamic phenomena in materials processing operations that involve compressible fluid flow, such as inert gas atomization of molten metals and thermal spraying of protective coatings. This paper describes the use of three types of schlieren optical arrangements to visualize compressible flow patterns produced in these processes using commercially available CCD cameras. Factors affecting schlieren sensitivity and image contrast and quality associated with the choice of optical elements, cutoff filter, light source are discussed, as are issues arising from the operational characteristics of most commercial CCD cameras.

3:30 PM Break

3:45 PM
Increased Understanding of Gas Atomization from Gas Flow Imaging and High Speed Cinematography: *I. E. Anderson*[1]; R. L. Terpstra[1]; S. Rau[2]; R. Figliola[3]; [1]Iowa State University, Ames Lab., Ames, IA 50011 USA; [2]University of Bremem, Bremen Germany; [3]Clemson University, Clemson, SC 29634 USA

Visualization of gas-only flows and high speed photography of the atomization process have provided valuable insight to guide the development of a succession of high pressure gas atomization (HPGA) nozzles. HPGA is a close-coupled, discrete jet atomization method that has proved to be one of the most effective methods of producing rapidly solidified fine metal and alloy powders with high yields less than 20 microns using Ar, N2, or He gas. This presentation will compare the gas flow characteristics of convergent and convergent-divergent single jets and full gas jet ensembles used for HPGA. High speed photography and cinematography characterization of the HPGA process will also be presented, including brief high speed movie selections. This data, along with size and aspiration results, enabled an enhanced understanding of the melt disintegration mechanisms that operate during HPGA processing. This work was supported by USDOE-BES and the Process Science Initiative under contract no. W-7405-Eng-82.

4:15 PM
Imaging and Particle Image Velocimetry of Granular Flows: Daniel Steingart[1]; *James W. Evans*[1]; [1]University of California-Berkeley, Dept. of Matls. Sci. & Eng., Berkeley, CA 94720 USA

Granular flows are important in many processes encountered in the minerals/metallurgical/materials industries. Examples include the flow of ore particles in mineral processing, the flow of coke, ore and limestone in the iron blast furnace and the flow of particles in filling molds during ceramic processing. Unfortunately such flows are far less understood than the flow of fluids and there is need for additional theoretical

and experimental work to enhance processing technology. The paper describes an experimental investigation of the flow of dense, large particles from two dimensional hoppers. This flow displays a wide range of particle velocities from stationary particles in some regions of the hopper to fast falling particles just below the hopper exit. By using both a fast CCD camera (up to 270 frames/sec) and stroboscopic illumination it has been possible to image the particles throughout this range. Particle image velocimetry, which is normally a technique for measuring fluid velocities, has been applied to the images so that particle velocities have been determined as a function of position, hopper geometry and particle properties.

4:45 PM
High Speed Imaging in Rapid Solidification: *Andrew Martin Mullis*[1]; [1]University of Leeds, Dept. of Matls., Clarendon Rd., Leeds, W. Yorkshire LS2 9JT UK

Rapid solidification of deeply undercooled melts can give rise to many different types of microstructure. Normally these can only be characterised by performing optical or electron microscopy on the as-solidified samples, a time consuming process which requires mounting, polishing and etching of the samples to be analysed. Here we report on experiments to image the solidification front directly using high-speed (up to 42000 fps) digital video and the extent to which the likely solidification microstructure can be identified from the morphology of the solidification front. The technique will be illustrated with reference to the systems Cu-Sn and Cu-O. Depending on the level of undercooling applied three distinct types of microstructure can be identified in these systems, twinned dendritic, untwinned dendritic and grain refined.

International Symposium on Science and Technology of Interfaces in Honor of Dr. Bhakta Rath: Dislocations and Interfaces

Sponsored by: Structural Materials Division, ASM International: Materials Science Critical Technology Sector, Electronic, Magnetic & Photonic Materials Division, Physical Metallurgy Committee, Superconducting Materials Committee, Jt. Mechanical Behavior of Materials, Titanium Committee

Program Organizers: Sreeramamurthy Ankem, University of Maryland, Department of Material & Nuclear Engineering, College Park, MD 20742-2115 USA; I. Ovidko, Russian Academy of Sciences, Institute of Problems of Mechanical Engineering, Laboratory for Theory of Defects in Materials, St. Petersburg 199178 Russia; Chandra Pande, Naval Research Laboratory, Materials Science and Technology Division, Washington, DC 20375-5000 USA; S. Ranganathan, Indian Institute of Science, Department of Metallurgy, Bangalore 560 012 India

Tuesday PM — Room: 617
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Brian Cantor, University of Oxford, Dept. of Matls., Parks, Rd., Oxford OX1 3PH UK; Julia R. Weertman, Northwestern University, Matls. Sci. & Eng., Evanston, IL 60208 USA

2:00 PM Invited
Misfit Dislocations and Interfacial Strength: *Richard J. Arsenault*[1]; [1]University of Maryland, Matls. & Nucl. Eng., Bldg. 090, Stadium Dr., College Park, MD 20742-2115 USA

The occurrence of misfit dislocations at an interface is dependent upon numerous variables, and one of these variables is the strength of interface bonding. In other words, if there is no interfacial bonding, there will be no misfit dislocations. Vellinga has studied misfit dislocation cores form and it turns out that their structure depends on both misfit and bond strength. A trend from delocalized structures to localized structures that resemble VOLTERRA-type dislocations can be seen for decreasing misfit at constant strength of interaction, and for increasing strength of interaction at constant misfit. Results are compared with a description of misfit dislocation based on anisotropic linear elasticity. The conclusion is that the strength of the interfacial bond can be approximated by the dislocation core structure for a given misfit.

2:25 PM Invited
The Structure of Semicoherent Interfaces in Multilayers: *Peter M. Hazzledine*[1]; [1]UES, Inc, 4401 Dayton-Xenia Rd., Dayton, OH 45432 USA

Multilayered materials derive their strength from the abundance of interfaces which act either to restrict plasticity to single layers or to obstruct the passage of glissile dislocations across them. The key parameters are the obstacle strengths of the interfaces and the spacing of the layers. Macroscopic strength may be increased by strengthening the interfaces and by reducing their spacing, but not indefinitely: there is a 'strongest size'. The obstacle strength depends on the structure of the interfaces which is controlled by four mismatches across the interfaces, mismatches in lattice parameter, elastic modulus, fault energy and glide geometry. The first three mismatches depend strongly on the level of coherency and hence on the layer thicknesses. When the layers are thin they are fully coherent, there are no mismatch dislocations, the elastic moduli and effective fault energies are abnormal and coherency stresses are large. When the layers are thick, fault energies, elastic moduli (and the Koehler effect) are normal but the interfaces are populated with mismatch dislocations which may have a three-dimensional structure. The form, and hence the strength, of the interfaces varies continuously between these limits as the layer thickness is changed.

2:50 PM Invited
Utility of the Plasmon Theory of Surface and Interfacial Energies: *John J. Gilman*[1]; [1]UCLA, Matls. Sci. & Eng., 6532 Boelter Hall, Los Angeles 90095-1595 USA

Since both plasmon frequencies (energies) and cohesive energies depend primarily on the density of valence electrons in solids, they are intimately connected. This includes a connection between plasmon frequencies, bulk moduli, and surface energies. Then, since plasmons are electromagnetic disturbances, they obey some of the boundary conditions for electromagnetic fields that are independent of the details of a system. Therefore, it is convenient to use them to estimate the energies of simple interfaces in terms of well-known properties. The purpose of this paper is to review the plasmon theory of free-surfaces, and to show how plasmon energies, and/or free-surface energies can be used to estimate interfacial energies, and other cohesive properties.

3:15 PM Invited
The Nature of Interfacial Processes in Friction and Wear: *Doris Kuhlmann-Wilsdorf*[1]; [1]University of Virginia, Matls. Sci. & Eng., Charlottesville, VA 22904 USA

When two different materials slide on each other, significant intermixing of the two sides can occur, the more so, the stronger the local pressure. The mechanism of this intermixing has been studied in detail, especially for copper and silver, i.e. essentially mutually insoluble metals. The intermixing begins with the formation of fairly coarse "tongues". With continuing sliding these refine and develop into lamella of progressively decreasing thickness. In extreme cases, amorphization can occur which, however, is almost instantaneously followed by the nucleation of crystals of the same but thermodynamically unstable composition. Lubrication retards the process. The implications of this mechanisms for friction and wear behavior are profound.

3:40 PM Cancelled
Role of Planar Dislocation Boundaries in Metallic Deformation: *Robb M. Thomson*

4:05 PM Invited
Load Transfer at Imperfect Interfaces-Dislocation-Like Model: *H. Y. Yu*[1]; [1]Army Research Office–Far East, Unit 45002, APO AP 96337-5002, Tokyo Japan

Scientific and engineering studies of the physical and mechanical properties of macroscopic systems often require knowledge of the interface–the boundary region–separating bulk phases. On the macroscopic scale, interfaces are viewed as continuum entities with macroscopically defined parameters such as free energy per unit area, mobility, etc. From the point of view of classical elasticity, an interface manifests itself through interfacial boundary conditions imposed upon equations of elastic equilibrium. One of the most commonly used models is the linear spring-like model that a thin layer of interphase material is introduced near the interface. In the limit of vanishing layer-thickness, the interfacial tractions become continuous, but the displacements at either side of the interface layer become discontinuous, the jump in displacement being linearly proportional to the interfacial traction. The proportional constants are interface parameters called spring-like constant. In this work a new model (dislocation-like model) describing the boundary conditions of a partially debonded interface will be presented. The boundary conditions to be modeled are similar to the linear spring-like model except that the jump in displacement at the interface is assumed linearly proportional to the displacement at the interface of the constituent where the stress source is. The effect of the imperfect interface on the load transfer studied by

photoelastically measuring the elastic deformation in bimaterials due to an inclusion with dilatational misfit strain will also be reported. The maximum shear stress distributions measured from the isochromatic fringe patterns are in good agreement with the theoretical calculations. The results show that an imperfect interface could be viewed as a continuum entity with interface rigidity as proposed by the dislocation-like model.

4:30 PM Invited

Materials by Design: An Informatics Approach to Interface Engineering: *Krishna Rajan*[1]; [1]Rensselaer Polytechnic Institute, Matls. Sci. & Eng. Dept., MRC 110, 110 8th St., Troy, NY 12180 USA

It is of course well established that interfaces play a major role in controlling the properties of materials. The relationships between specific interface chemistries and/or structure and final properties is generally studied (experimentally and theoretically) from material to material. Yet there is a need to develop a more generalized approach which permits one to explore vast arrays of data on structure-property relationships based on interface characteristics. Such an approach can permit one search for correlations between structure and property across vastly different length scales. This type of "data mining" or informatics approach is a well established tool in the chemical and biochemical sciences in searching for structure-property relationships in a large combinatorial designs of molecular chemistries. This approach is not wide spread in the materials science community, however, we will outline the value of this approach in some specific cases in designing materials through controlling interfacial characteristics.

Lead-Free Solders and Materials Issues in Microelectronic Packaging: Electromigration, Processes and Emerging Technologies

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

Program Organizers: Srini Chada, Motorola, Department APTC, Fort Lauderdale, FL 33322 USA; Darrel R. Frear, Motorola, Tempe, AZ 85284 USA; Sung-Ho Jin, Lucent Technologies, Bell Laboratories, Murray Hill, NJ 07974 USA; Sung Kang, IBM, T. J. Watson Research Center, Yorktown Heights, NY 10598 USA; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Michael J. Pfeifer, Motorola, Northbrook, IL 60062 USA; Martin Weiser, Honeywell Electronics Materials, Plated and Discrete Products, Spokane, WA 99216 USA

Tuesday PM — Room: 612
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: C. Robert Kao, National Central University, Dept. of Cheml. Eng., Chungli City Taiwan; Gautam Ghosh, Northwestern University, Metall., Evanston, IL USA

2:00 PM Invited

Lead-Free Universal Solders for Optical and Electronic Devices: *Hareesh Mavoori*[1]; Ainissa G. Ramirez[1]; Sungho Jin[1]; [1]Bell Laboratories, Agere Systems/Lucent, Appl. Matls. Rsrch., 700 Mountain Ave., Murray Hill, NJ 07974 USA

There is a need to directly bond a wide variety of inorganic materials such as oxides, nitrides, carbides, fluorides, diamond, and semiconductors in electronic, optoelectronic and optical devices. Such applications involve active components, dielectric layers, diffusion barriers, wave guides, and heat sinks. These materials are known to be very difficult to wet and bond with low melting point solders. In this presentation, we will describe new Pb-free universal solders containing rare-earth elements which allow direct and powerful bonding onto the surfaces of various optical and electronic device materials. The microstructure, interface properties, and mechanical behavior of the solder bonds will be described, and the potential applications of these new solder materials for devices such as optical fiber gratings, MEMS, heat actuation or heat dissipation devices, and electronic packaging will be discussed.

2:25 PM

Lead-Free Active Solder Joining in Electronic Packaging: Ronald W. Smith[1]; *Ayman Salem*[2]; [1]Materials Resources International, 811 W. Fifth St., Unit 2, Lansdale, PA 19446 USA; [2]Drexel University, Matls. Eng., 32nd & Chestnut St., Philadelphia, PA 19104 USA

Lead-free, active solders have recently been developed that have show promise to be able to join most metals, many ceramics, and many metal matrix composites (MMC). Active solders have compositions derived from conventional solders, however, have the addition of reactive elements that are able to interact and/or react with directly with surfaces to be joined without the assistance of fluxes, reducing atmospheres and or plating. Electronic packaging is also getting more complex. With the increase in computing speeds both interconnects are being required to be more direct, more reliable and able to handle and transfer the increased powers and associated heating. As such, thermally conductive ceramics such as aluminum nitride and low CTE composites such as Al:SiC and Al:Gr are being used in electronic packages, combined with aluminum, copper and low CTE metals such as Kovar®. The paper presents the metallurgy of several active solders based in Sn-Ag-Ti and Zn-Ag-Al that have lanthanide elemental additions. Their joint structures with silicon, AlN, Al2O3, Gr-foam, aluminum and copper as well as other packaging candidates will be presented and selected joint properties will be discussed. Several new electronic packaging assembly concepts, enabled by active soldering will also be presented.

2:45 PM

Experimental Investigation of a New Pb-Free Solder Alloy for Power Die Attachment: *J. Nick Lalena*[1]; Nancy F. Dean[1]; Martin W. Weiser[2]; [1]Honeywell Electronic Materials, Pkgg. Sci. & New Matls., 15128 E. Euclid Ave., Spokane, WA 99216 USA; [2]Honeywell Electronic Materials, Plated & Discrete Products, 15128 E. Euclid Ave., Spokane, WA 99216 USA

It now seems certain that the electronics industry will eventually adopt a Sn-Ag-Cu solder alloy for board level (Level 2) assembly. These solders are reflowed between 235 to 270C, thus raising the minimum required solidus of the internal Level 1 material attaching the die to the leadframe in power devices. The solidus of this material must be high enough to prevent detachment of the die while the package is soldered to a PWB. Although current power die attach solders will survive these temperatures, they are either lead containing (e.g. Pb-5Sn) or expensive, while possessing less-than-ideal mechanical properties (e.g. Au-20Sn, Au-12Ge). Surprisingly, however, there has been little published research on possible alternatives. Because power die are often quite large (0.8 cm on a side is common) and generate a large amount of heat (> 100 W), die attach solder must have a high thermal conductivity and good resistance to thermomechanical fatigue. We have identified a low cost alloy with a melting point above 260C, a moderate thermal conductivity, and possessing a shear modulus and tensile strength significantly better than that of the gold alloys or Alloy J (Sn-25Ag-10Sb). Test assemblies containing Si die have survived over 2000 thermal cycles. The alloy can be soldered to copper, nickel-plated copper and silver-plated copper substrates. Furthermore, wire, ribbon and preform shapes can be fabricated by conventional metalworking techniques. We believe this alloy is thus a very attractive alternative Pb-free power die attach solder. The microstructure, thermal and mechanical properties, and physico-chemical behavior of the alloy will be discussed.

3:05 PM

Leadfree BGA-Balls–Production Method and Properties: *Bernd Kempf*[1]; Muriel Graff[1]; Oliver Hutin[1]; Markus Rettenmayr[2]; [1]OMG AG & Co. KG, Techl. Matls./R&D, Rodenbacher Chaussee 4, Hanau 63403 Germany; [2]Technische Universität Darmstadt, Fachbereich Materialwissenschaften, Darmstadt, Germany

BGA-balls are usually produced by remelting of preforms like cut wire pieces. An innovative production method is presented which allows the production of BGA solder balls directly from melt. The physical background of this production method is discussed and the situation for Sn-based leadfree BGA-balls is analyzed. The parameters sphere size distribution and roundness are similar for leadfree alloys and the standard alloy Pb37Sn63. Differences are encountered concerning the optical appearance, caused by a rather "rough" sphere surface. The reason is a difference in the solidification behavior which leads to a coarse grained structure in the case of Sn-based alloys, whereas PbSn-alloys show fine-grained solidification behavior and hence a shiny and smooth surface. The investigations base on scanning electron microscope and light microscope. Possible differences in the workability of leadfree and PbSn-BGA were discussed. One striking difference in this field is the "darkening" effect of PbSn-BGA. A "darkening" of the BGA-balls during handling can be observed. This is probably caused by oxidation effects as result of "mechanical grinding of the surface". This can cause problems by identifying the balls with optical sensors in the pick & place station. The "darkening behavior" was investigated in comparison with PbSn-balls. The results show a significant higher stability against "darkening" for leadfree BGA in comparison to PbSn-BGA.

3:25 PM
Microstructure of Lead-Free Composite Solders Produced by an In-Situ Process: Sung-Yong Hwang[1]; *Zin-Hyoung Lee*[1]; [1]KAIST, Dept of Matls. Sci. & Eng., Ctr. for Elect. Pkgg. Matls., Taejeon 305-701 Korea

Composite solders are being developed for the purpose of improving creep and thermal fatigue strength of monolithic solders. Particles such as Cu6Sn5, Ni3Sn4 in the solder matrix could be formed by an in-situ method. These intermetallics were solidified in forms of dendrites or platelets in the solder matrix after casting. It is found that plastic working such as cold rolling the intermetallics were crushed into particles and redistributed in the solder matrix. In this process, the cast microstructure which is strongly dependent on the cooling rate played an important role on the final structure. As the cast microstructure became finer, the intermetallic particles in the final solder alloy were also finer and more uniformly distributed. The solder was made into solder balls by disc-forming method. After reflow soldering, the sedimentation of the particles was observed. The sedimentation of the particles was reduced, as the size of the particles decreased.

3:45 PM Break

4:00 PM Invited
The Polarity Effect of Electromigration on Intermetallic Compound (IMC) Formation in Solder V-Groove Samples: *Hua Gan*[1]; King-Ning Tu[1]; [1]University of California-Los Angeles, Dept. of Matls. Sci. & Eng., Los Angeles, CA 90095 USA

The polarity effect of electromigration on intermetallic compound (IMC) formation at cathode and anode in the solder joints has been investigated. Lead free solder was reflowed into the V-grooved Silicon wafer and two copper wires were used as electrodes. The V-groove samples with width of about 100μm were used to study morphology and thickness changes of IMC in 95.5Sn3.8Ag0.7Cu/Cu system under different current density and temperature settings. The current densities used were 10E4 to 10E5 A/cm2 and the temperature settings were in the range of 120C to 180C. The growth of IMC has been enhanced by electric current at anode and inhibited at cathode comparing with the samples without applying current. We found that electromigration has different effect on morphology and thickness changes at cathode and anode sides, and this effect will be discussed.

4:25 PM
Electromigration of Flip Chip Solder Bump on Cu/Ni(V)/Al Thin Film under-Bump-Metallization: *Woojin Choi*[1]; *King-Ning Tu*[1]; Everette C.C. Yeh[2]; Peter Elenius[3]; Haluk Balkan[3]; [1]University of California-Los Angeles, Dept. of Matls. Sci. & Eng., 6532 Boelter Hall, Box 951595, Los Angeles, CA 90095-1595 USA; [2]Intel Corporation, Santa Clara, CA 95052 USA; [3]Flip Chip Techologies, Phoenix, AZ 85034 USA

The electromigration of flip chip solder bump (eutetic SnPb) has been studied at temperatures of 100, 125 and 150°C and current densities of 1.9 to 2.75 x 104 A/cm2. The under-bump-metallization on the chip side is thin film Al/Ni(V)/Cu and on the board side is thick Cu. By simulation, we found that current crowding occurs at the corner on the chip side where the electrons enter the solder ball. We are able to match this simulation to the real electromigration damage in the sample. The experimental result showed that voids initiated from the position of current crowding and propagated across the interface between UBM and the solder ball. The Cu-Sn intermetallic compounds formed during the reflow is known to adhere well to the thin film UBM, but they also detached from the UBM after current stressing. Therefore, the UBM itself could be part of the reliability problem of the flip chip solder joint under electromigration.

4:45 PM
The Effect of Flux Chemistry, Applied Voltage, Conductor Spacing and Temperature on Conductive Anodic Filament Formation: *Laura J. Turbini*[1]; W. Jud Ready[2]; [1]Centre for Microelectronics Assembly and Packaging, Univ. of Toronto, 184 College St., Rm. 150B, Toronto, ON M5S 3E4 Canada; [2]Microcoating Technologies, 5315 Peachtree Industrial Blvd., Atlanta, GA 30341 USA

Conductive Anodic Filament formation, a failure mode in printed circuit assemblies exposed to high humidity and high voltage gradient, has caused catastrophic field failures. This study quantified the effect of flux chemistry, applied voltage (V), spacing (L) and temperature on the failure rate. Test vehicles, which had hole-to-hole spacing of 0.5mm or 0.75mm, were processed with one of three fluxes, and a heated control was also evaluated. The samples were placed in a temperature humidity chamber at 85%RH, at one of three temperatures: 75°C, 85°C or 95°C. A voltage of 150V or 200V was applied to the test vehicle and periodically removed so that a measurement could be taken. A specially designed linear circuit was used to determine when the insulation resistance dropped significantly, indicating a failure. Activation energies were determined. The Mean Time to Failure was a function of L^4/V^2.

5:05 PM
Effect of Heat Treatment on the Electrical Resistivity of Near-Eutectic Sn-Ag-Cu: *Bruce Alan Cook*[1]; *Iver Eric Anderson*[1]; Joel Lee Harringa[1]; James C. Foley[1]; Robert L. Terpstra[1]; [1]Ames Laboratory, Metall. & Cer., 253 Spedding, IA State Univ., Ames, IA 50011-3020 USA

The electrical resistivity of Sn-Ag-Cu and Sn-Ag solder compositions was determined by a 4-point probe technique and interpreted in terms of microstructure and composition. The resistivity is reported of both drawn wire solder and of solder joints obtained by hand soldering to copper substrates. Measurements were obtained at -40C, 22C, and 50C on the drawn wire specimens, enabling determination of the temperature coefficient of resistivity (TCR) for each composition. The resistivity of solder joints was characterized over a temperature range from -40C to 150C, covering the entire operational range of interest for Pb-free solders. Selected joint specimens were measured before and after a 72 hour heat treated at 150C. Optical metallography was performed on the joints to characterize coarsening effects and relate these to changes in the resistivity. Effects of alloying, such as partial substitution of bismuth for tin, were also studied and compared with a baseline ternary near-eutectic Sn-Ag-Cu composition. Support received from USDOE-BES, Materials Science Division (contract no. W-7405-Eng-82).

Magnesium Technology 2002: High Temperature Alloy Development - Mechanical Properties - II

Sponsored by: Light Metals Division, Magnesium Committee, International Magnesium Association
Program Organizers: Howard I. Kaplan, Magnesium Corporation of America, Salt Lake City, UT 84116 USA; Menachem Bamberger, Technion, Israel Institute of Technology, Haifa 32000 Israel; Byron B. Clow, International Magnesium Association, McLean, VA 22101 USA; Gerald S. Cole, Ford Motor Company, Ford Research Laboratories, Dearborn, MI 48121 USA; Rod Esdale; John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Zi-Kui Liu, Pennsylvania State University, Materials Science and Engineering, University Park, PA 16082-5005 USA; John L. Mihelich, Metal Experts International, Winston, GA 30187 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. Pekguleryuz, Noranda, Noranda Technology Centre, Pointe-Claire, Quebec H9R 1G5 Canada; Bob R. Powell, General Motor Corporation, NAO Research and Development Center, Warren, MI 48090-9055 USA; Allen Schultz, Hatch, Mississauga, Ontario L5K 2R7 Canada

Tuesday PM — Room: 606
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Gerald S. Cole, Ford Motor Company, Ford Rsrch. Labs., MD 3135, Rm. 2170, Dearborn, MI 48121 USA; Jian-Feng Nie, Monash University, Sch. of Phys. & Matls. Eng., PO Box 69M, Victoria 3800 Australia

2:00 PM
Precipitation and Strengthening in Selected Magnesium Alloys: *Jian-Feng Nie*[1]; [1]Monash University, Sch. of Phys. & Matls. Eng., PO Box 69M, Victoria 3800 Australia

Lightweight magnesium alloys have attracted increasing interest in recent years for potential applications in the aerospace, aircraft and automotive industries. One important group of magnesium alloys is those strengthened by precipitation hardening. The maximum yield strength achievable in these alloys, however, is substantially lower than that obtained in precipitation-hardened aluminium alloys. Further improvement in the strength of these alloys requires better understanding of precipitate microstructures and identification of microstructural factors that are important in controlling the strength of the alloys. This presentation will provide a review of recent results on characterisation of the structure, morphology and orientation of strengthening precipitate phases in Mg-Y-Nd and Mg-Al alloys using transmission electron microscopy. The formation mechanisms of the strengthening phases, and the effects of the shape, orientation and

distribution of precipitates on the yield strength of magnesium alloys, will be discussed in detail.

2:25 PM

Microstructure and High Temperature Creep Behavior of a Die Cast Magnesium-Rare Earth Alloy: Ian P. Moreno[1]; *Tapash K. Nandy*[1]; J. Wayne Jones[1]; John E. Allison[2]; Tresa M. Pollock[1]; [1]University of Michigan, Matls. Sci. & Eng., Ann Arbor, MI 48109 USA; [2]Ford Motor Company, Matls. Sci. Dept., Dearborn, MI 48126 USA

The microstructure and creep behavior of a new high-pressure die cast Mg-rare earth alloy, Elektron MEZ (Mg-2.5RE-0.35Zn-0.3Mn) has been investigated. The microstructure consists predominantly of equiaxed α-Mg dendrites with a partially divorced interdendritic eutectic. Diffraction and compositional analysis indicate the presence of $Mg_{12}Ce$ intermetallic and fine Mg particles in the eutectic aggregate. The evolution of the as-cast microstructure is related to the structures obtained in other die cast Mg alloys such as AZ91D and AE42. The steady state creep behavior can be represented by a standard power law creep equation with stress exponent and activation energy varying from 5-7 and 110-320 kJ/mole, respectively, depending upon stress and temperature. Based on the values of stress exponent and activation energy, possible creep mechanisms are discussed in relation to the deformation substructures. The alloy exhibits creep resistance superior to Mg-rare earth alloy, AE42, especially at higher temperatures, and a possible rationale is presented.

2:50 PM

Structural and High-Temperature Magnesium Alloys: *Alan P. Druschitz*[1]; Eric Showalter[2]; Joe McNeill[3]; Dave White[4]; [1]Intermet Corporation, Matls. R&D, 939 Airport Rd., Lynchburg, VA 24551 USA; [2]Intermet Corporation, Adv. Mfg. Eng., 5445 Corporate Dr., Ste. 200, Troy, MI 48098-2683 USA; [3]Intermet Corporation, Monroe City Plant, Monroe City, MO 63456 USA; [4]Intermet Corporation, Palmyra Plant, 7036 Country Rd. 328, Palmyra, MO 63461 USA

The automobile and light truck industries are increasingly using more magnesium castings in structural and high-temperature applications. Unfortunately, the casting and mechanical behavior of the commonly used alloys are largely undocumented and new high-temperature alloys are being promoted, but their casting and mechanical behavior is essentially unknown. Therefore, five high temperature magnesium alloys (N, AJ52, AS21X, MRI-153 and AE42) and two structural magnesium alloys (AM50 and AM60) were evaluated. This paper documents the results of tests that determined casting behavior and mechanical properties. The metallurgical factors affecting these properties are discussed and will assist the metallurgist in understanding the relationships between structure and properties. The property data will assist engineers and designers in using these lightweight materials efficiently and effectively.

3:15 PM

Thin-Wall Die Castability of Ca-Containing Magnesium Alloys: *Bob R. Powell*[1]; Alan A. Luo[1]; Basant L. Tiwari[1]; Vadim Rezhets[1]; [1]General Motors, R&D Ctr., Matls. & Proc. Lab., MC 480-106-212, Warren, MI 48090-9055 USA

The effect of calcium content on the high-pressure die castability of a family of newly developed creep-resistant magnesium alloys was evaluated for thin-wall applications. Calcium additions up to 6% were made to AM50. Castabilty was determined using a computer case die (2 mm wall thickness) in a 700-ton cold-chamber machine by determining the casting defect type and frequency. At 1% calcium die-sticking and soldering became significant. Increasing the calcium level to 2.6% all but eliminated both defects. Calcium, at all levels, increased the occurrence of hot-cracking slightly above that of the baseline alloy, AM50. Strontium additions up to 0.17% had no effect on castability.

3:40 PM Break

4:00 PM

Plane Stress Fracture Toughness Testing of Magnesium Alloy AM60B: *Toby V. Padfield*[2]; Cory J. Padfield[1]; [1]General Motors, Matls. & Fastening Eng., 7000 Chicago Rd., MC 480-202-226, Warren, MI 48090 USA; [2]Sachs Automotive of America, 2107 Crooks Rd., Troy, MI 48084 USA

J-R behavior was measured for magnesium alloy AM60B produced by high pressure die casting. Compact Tension (CT) specimens were obtained from plate samples with approximately 4 mm thickness. The compliance unloading technique was used to record crack extension for each specimen. The specimens exhibited stable crack extension beyond ASTM E 1820 limits for J_{max} (~33 kJ m^{-2}) and Δa_{max} (~2.1 mm). The data was in good agreement with a power law fit for J vs. Δa. Fracture was by microvoid coalescence, most likely initiated between the primary Mg grains and the brittle $Mg_{17}Al_{12}$ phase.

4:25 PM

Improvement of Strength-Ductility Balance in Commercial Magnesium Alloys under Dynamic Loading: *Toshiji Mukai*[1]; Hiroyuki Watanabe[1]; Koichi Ishikawa[1]; Kenji Higashi[2]; [1]Osaka Municipal Technical Research Institute, Mechl. Eng. Dept., 1-6-50 Morinomiya Joto-ku, Osaka 536-8553 Japan; [2]Osaka Prefecture University, Dept. of Metall. & Matls. Sci., 1-1 Gakuen-cho, Sakai, Osaka 599-8531 Japan

Magnesium alloys are generally brittle owing to their HCP structure under dynamic loading. In this study, improvement of the tensile mechanical properties has been demonstrated for commercial magnesium alloys by the modification of the grain structure. The yield stress of the alloy is effectively increased with refining the grain structure at the dynamic strain rate with a similar slope of Hall-Petch relation at a quasi-static strain rate. Enhancement of ductility can be also achieved by refining grain structures for magnesium alloys. The high ductility of the fine-grained alloy is due to the absence of macroscopic cracking at mechanical twin boundaries. It is found that the absorption energy per weight in the fine-grained magnesium alloys is twice higher than that of high strength aluminum alloys.

4:50 PM

High Strength Mg-Zn-Y Alloy Containing Quasicrystalline Particles: Donghyun Bae[1]; *Do Hyang Kim*[1]; [1]Yonsei University, Metall. Eng., 134 Shinchon-dong, Seodaemum-gu, Seoul 120-749 Korea

A new magnesium alloy strengthened by icosahedral quasicrystalline particles and precipitates was developed by the thermomechanical processes for an as-cast Mg-rich Mg95Zn4.3Y0.7 alloy. Quasicrystalline particles of 0.5-2 micrometer in size were distributed in the a-Mg matrix by the hot-rolling process, and nanoscale quasicrystals were also precipitated through out the a-Mg grains during this thermomechanical process. The alloy exhibits high strengths and large elongations at room and elevated temperatures. High strength observed in the alloy is mainly due to the strengthening effect of large number of quasicrystals, where the volume fraction of quasicrystals is around 9%. The quasicrystalline phase is found to be in equilibrium with the a-Mg phase and stable against coarsening under deformation up to near the melting temperature of the eutectic. The stable quasicrystalline particle/matrix interface with a low interfacial energy can provide the improved mechanical properties of the alloy.

5:15 PM

Effect of Thermal Cycling on QE22 + 22 Vol% Saffil MMC: *Subodh Kumar*[1]; Hajo Dieringa[2]; Karl Ulrich Kainer[2]; [1]Indian Institute of Science, Dept. of Metall., Bangalore 560 012 India; [2]GKSS Research Centre, Ctr. for Magnesium Tech., Max Plank Strasse, Geesthacht D-21501 Germany

Magnesium metal matrix composites (MMCs) are being developed for automotive applications as they give lower thermal expansion coefficient along with other property improvements. Lower thermal expansion coefficient of MMCs results in lower thermal fatigue due to repeated heating and cooling back to the ambient temperature of powertrain components in automobiles. However, differential thermal expansion of matrix and reinforcement gives rise to residual stresses on heating and cooling, which might result in net residual strain after each cycle. In the present investigation, the effect of thermal cycling is studied on a QE22 matrix reinforced with 20 vol % saffil short-fibres. The composite was prepared by infiltrating matrix alloy in to a preform made of reinforced by squeeze casting. The properties of short-fibre reinforced composites exhibit anisotropy. Therefore, the effect of thermal cycling is examined in longitudinal as well as transverse directions. The matrix material might undergo phase transformation or the interface might undergo chemical reactions during thermal cycling, Therefore, the thermal cycling results are supplemented by differential scanning calorimetery studies and microstructural examination by optical and scanning electron microscope.

5:40 PM

Shell Mould Investment Casting of AZ91E Magnesium Alloy: Zhan Zhang[1]; Guy Morin[2]; [1]Technologies Intermag, Inc., 357 rue Franquet, Sainte-Foy Quebec, G1P 4N7 Canada; [2]Centre Integre de fonderie et de Metallurgie, 3247 rue Foucher, Trois-Rivieres, Quebec, G8Z 1M6 Canada

Abstract Unavailable

6:05 PM

Quality in Mg Sand Castings-I. A Structured Approach Towards Microshrinkage Control: M. Galopin[1]; Y. Carbonneau[2]; S. Veilette[2]; [1]E. M. Optimisation, Inc., 4750 av. Henri-Julien, Bureau 308, Montreal, Quebec H2T 2C8 Canada; [2]Technologies Intermag, Inc., 357, Franquet St, Sainte-foy, Quebec, G1P 4N7 Canada

Abstract Unavailable

Materials Processing Fundamentals - II

Sponsored by: Extraction & Processing Division, Process Fundamentals Committee
Program Organizers: P. N. Anyalebechi, Grand Valley State University, Padnos School of Engineering, Grand Rapids, MI 49504-6495 USA; Adam Powell, Massachusetts Institute of Technology, Cambridge, MA 02139-4301 USA

Tuesday PM Room: 614
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: David G. Robertson, University of Missouri, Metlgcl. Eng., 215 Fulton Hall, Rolla, MO 65409-1460 USA; Mark Schlesinger, University of Missouri, Metlgcl. Eng., 1870 Miner Cir., Rolla, MO 65409-0001 USA

2:00 PM
Directional Annealing of Worked Alloys: *I. Baker*[1]; J. Li[1]; S. L. Johns[1]; B. Iliescu[1]; H. J. Frost[1]; [1]Dartmouth College, Thayer Sch. of Eng., 8000 Cummings Hall, Hanover, NH 03755-8000 USA

The effects of annealing temperature, hot zone velocity and temperature gradient ahead of the hot zone during directional annealing of a model system of cold-rolled copper crystals with and without fine silica particles have been investigated. Columnar grains were observed at hot zone velocities that increased with increasing annealing temperature and decreasing temperature gradient. Columnar grains did not occur either when the velocity of the hot zone was low or exceeded the maximum growth rate of grains, when equiaxed grains occurred. The twin boundary density decreased with increasing hot zone velocity, increasing temperature gradient and decreasing temperature. The results are explained in terms of the effects of temperature and temperature gradient on the grain boundary mobility and the nucleation of new grains and twins. Preliminary results on polycrystalline nickel and on the nickel-base superalloy MA 754 will also be presented. Research supported by AFOSR grant F49620-00-1-0076 and NSF grant DMI9976509.

2:30 PM
On the Evolution of Deformation Texture of AA 3105 DC and AA 3105 SC Aluminum Alloys during Cold Rolling: *Jiantao Liu*[1]; *Tonguang Zhai*[1]; *Xiyu Wen*[1]; Jian Chen[1]; *Xiangming Cheng*[1]; *Wenchang Liu*[1]; *James G. Morris*[1]; [1]University of Kentucky, Ctr. for Aluminum Tech., Col. of Eng., 1505 Bull Lea Rd., Lexington, KY 40511 USA

Industrially produced hot bands of AA 3105 DC and AA 3105 SC aluminum alloys were cold rolled with different cold rolling reductions. The evolution of deformation texture in AA 3105 DC and AA 3105 SC materials was investigated by means of a three-dimensional orientation distribution function (ODF) analysis. The effects of initial texture and cold rolling reduction on the development of deformation textures in both materials were studied. The deformation texture evolution was correlated with microstructure evolution. The comparison of deformation texture evolution between AA 3105 DC and AA 3105 SC materials was made and the differences were discussed.

3:00 PM
In-Situ TEM Study of Grain Coarsening under Electrical Current Application in Silver Thin Films: Xuyen Phung[1]; *Joanna R. Groza*[1]; Erich Stach[2]; [1]University of California, Matls. Sci. Dept., One Shields Ave., Davis, CA 95616 USA; [2]Lawrence Berkeley National Laboratory, Natl. Ctr. for Electron Microscopy, Berkeley, CA USA

Grain growth in nanocrystalline Ag films under simultaneous heating and electrical current application is investigated using an in-situ transmission electron microscope equipped with an electrical biasing heating holder. 70 nm thick Ag films with an initial grain size of 30-40 nm are sputter deposited onto back-etched amorphous silicon nitride supporting membranes. Specimens, one for each temperature, are heated from room temperature up to 125, 175, and 225°C with the applied current density on order of 108 A/m2. The kinetics of grain growth as a function of temperature and external electrical field are characterized by the equation Dn - Don = k(T,E) t, where D is the average grain diameter, Do is the initial grain diameter, k is the rate constant, and t is time. The normal grain growth exponent, n, is calculated by minimizing the error due to the deviation in the fitting function to the experimental data. The activation energy deduced from the Arrhenius-type rate constants in the above equation is used to determine the dominant mass transport mechanism for grain growth and to identify field effects from temperature effects alone in nanomaterial coarsening.

3:30 PM Break

4:00 PM
Particle Distribution and Recrystallization of a Continuous Cast AA5182 Aluminum Alloy: *Tongguang Zhai*[1]; Xifeng Yu[1]; Xiyu Wen[1]; Xiangming Cheng[1]; Jiantao Liu[1]; Wenchang Liu[1]; Jian Chen[1]; James Morris[1]; [1]University of Kentucky, Chem. & Matls. Eng., 177 Anderson Hall, Lexington, KY 40506 USA

It has been recognized that the distribution of second phase particles is non-uniform in continuous cast Al-Mg alloys, and that the heterogeneous particle distribution affects the microstructure and mechanical properties of the alloys. In this work, second phase particles were studied in continuous cast AA5182 hot band (gauge 3 mm). A homogenization treatment (1020°F for 16 hours) was conducted to improve the distribution of particles in the hot band. The homogenization treated and untreated hot bands were then cold rolled with a reduction of 70% and finally annealed at different temperatures. It was found that the homogenization treatment led to a lower temperature for complete recrystallization than for samples without the homogenization treatment. In addition, there was an influence on the recrystallization texture that was related to the specific particle structure produced.

4:30 PM
Columnar to Equiaxed Transition in 316 Stainless Steel: Alicia Esther Ares[3]; Carlos T. Rios[2]; Rubens Caram[2]; *Carlos Enrique Schvezov*[1]; [1]University of Misiones, Fac. of Sci., 1552 Azara St., Posadas, Misiones 3300 Argentina; [2]Universidade Estadual de Campinas, Dept. Engenharia de Matls., CP 6122, Campinas, Sao Paulo 13083-970 Brasil; [3]CONICET, CP 6122, Campinas, Sao Paulo 13083-970 Brasil

316 L Stainless Steel samples were solidified directionally under conditions which produced the columnar to equiaxed transition. The position of the transition was located in each sample and the distance from the bottom of the ingot was measured. During solidification the distribution of temperatures were measured by means of thermocuples located strategically. From the measured temperature the following parameters were derived; the local temperature gradient, the cooling rate and the velocities of the liquid and solidus fronts. Comparing the location of the transition and the values of these parameters it was found that the temperature gradient reaches negative values of as low as -3°C/cm, and velocities of the fronts of around 0.2 cm/s. Also, a supercooling of 3°C were measured which was associated with the supercooling for the nucleation and growth of the equiaxed grains ahead of the columnar front. In addition the primary and secondary dendritic spacing was measured in both regions. The spacing was compared with the predictions from available models. The results are presented and discussed in the frame of the results obtained for other alloys.

Materials & Processes for Submicron Technologies - II: Materials & Process Reliability of Advanced Metallization

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Thin Films & Interfaces Committee
Program Organizers: Seung H. Kang, Lucent Technologies, Orlando, FL 32819 USA; N. (Ravi) M. Ravindra, New Jersey Institute of Technology, Department of Physics, Newark, NJ 07102-1982 USA

Tuesday PM Room: 209
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Seung H. Kang, Agere Systems, VLSI Tech. Sys., 9333 S. John Young Parkway, Orlando, FL 32819 USA; Mahesh K. Sanganeria, Novellus Systems; Nuggehalli M. Ravindra, New Jersey Institute of Technology, Dept. of Phys., 161 Warren St., Newark, NJ 07102-1982 USA

2:00 PM Invited
Effect of Current Crowding on Electromigration in VLSI Interconnects: *King-Ning Tu*[1]; [1]University of California, Dept. of Matls. Sci. & Eng., Sch. of Eng. & Appl. Sci., Los Angeles, CA 90095-1595 USA

Effect of current crowding on electromigration in VLSI interconnects.

2:30 PM
Interface/Surface Instability during Electromigration Failure: *Nancy L. Michael*[1]; Choong-Un Kim[1]; Qing-Tang Jiang[2]; [1]University of Texas-Arlington, Arlington, TX 76019 USA; [2]International Sematech, Austin, TX 78741 USA

Though historically interface and surface instability have not been considered as technologically important factor in microelectronics,

recently many researchers are finding that interface structure and surface stability may play a key role in developing devices of the future. One of such examples was found in our investigation on electromigration failure in single level, damascene processed, 0.25μm wide, Cu interconnects. We found evidence that the interplay of instabilities caused by interface electromigration and surface energy driven diffusion work together in developing damage in the interconnects. TEM examination of failure sites reveals damage is initiated at top surface but, propelled by surface instability of Cu, encompasses entire periphery of the Cu, resulting in an isolated Cu strand. Furthermore, the diameter of Cu strand varies with the orientation of grain, suggesting that electromigration damage in Cu is a result of complex interaction between interface and Cu surface instability. The paper addresses the mechanism of the instability interplay observed along with metallurgical evidences

2:50 PM

Wafer-Level Investigation on the Electromigration Reliability of Cu Metal Lines: *Hun Sub Park*[1]; Joon Kiat Low[1]; Cher Ming Tan[1]; [1]Nanyang Technological University, Sch. of Matls. Eng., Nanyang Ave. 639798 Singapore

Electromigration reliability of the Cu metallization is a hot issue in development of the advanced semiconductor technology. This research has been focused on electromigration mechanisms of Cu metal lines in the 0.13μm semiconductor technology level. The multi-level interconnect samples have been prepared with a few process split conditions in order to study the impact of process factors on the Cu electromigration reliability. The failure mechanisms are explained based on the measured activation energy and current exponent factor. Wafer-level measurements of mean-time-to-failure have been implemented on the multi-level Cu interconnects at various conditions of current density and temperature. The measurements have been carried out on a multitude of test patterns at a time using a probe card, which can provide uniformity data of the electromigration reliability across the wafer as well as save measurement time. The samples that failed in the early stage of the measurement have been analyzed using SEM, TEM and FIB (focused ion beam). The mechanisms inferred from the mean-time-to-failure will be confirmed with the failure phenomena observed using the analytical instruments.

3:10 PM

Reliability Improvement of Cu Interconnects by Optimizing Materials and Processes: *Seung H. Kang*[1]; [1]Agere Systems, Tech. Dvlp., 9333 S. John Young Pkwy., Orlando, FL 32819 USA

The increasing demand for high-performance integrated circuits (ICs) has driven the integration of dual-damascene Cu interconnects in conjunction with low dielectric-constant (low-k) materials. One of the key concerns in introducing reliable Cu/low-k interconnects is to control the interface between Cu and its surrounding films (diffusion barrier and dielectric passivation/capping layer) since the integrity of the interface is known as a dominant factor that affects Cu electromigration. In order to understand the effect of interfacial properties on the electromigration reliability of Cu, we have studied various Cu/low-k interconnects (designed for the 0.13 m technology) that incorporate several types of passivation and barrier films. In particular, we have explored various surface treatments and thermal anneals to improve structural and chemical integrity of the interface. The results show that a significant reliability improvement can be achieved by modulating the interface between the Cu and the dielectric passivation layer. In addition, the electromigration lifetime and the activation energy of failure widely vary as a function of the variables studied in this work. The results were examined also using a thermoelectric finite element model to understand the resistance-increase kinetics and Joule heating associated with electromigration-induced voiding.

3:30 PM Break

3:50 PM Invited

Reliability and Early Failure in Cu/Oxide Dual-Damascene Interconnects: *Ennis T. Ogawa*[1]; K.-D. Lee[1]; H. Matsuhashi[1]; P. S. Ho[1]; V. A. Blaschke[2]; R. H. Havemann[2]; [1]The University of Texas at Austin, Lab. for Interconnect & Pkgg., Microelect. Rsrch. Ctr., PRC/MER, MC R8650, Austin, TX 78712-1100 USA; [2]International SEMATECH, 2706 Montopolis Dr., Austin, TX 78741-6499 USA

The issue of "early failure" has been a issue of concern since large scale integration of interconnects has gone beyond our abilities to test for them accurately. With the emergence of Cu interconnect technology after consistent use of Al-based interconnects for the previous 30 years, the general issue of interconnect reliability in this new system has become a major concern. Not only is the new choice of metal a matter of concern but also the use of the new damascene technologies to fabricate dense interconnections. While there are a number of reliability issues to consider in Cu, especially now with low-k materials being incorporated into present and subsequent interconnect generations, electromigration (EM) will be the primary focus of this discussion. The discussion will introduce the peculiarities of dual-damascene architecture that have impact on EM. Then, the notion of early failure will be addressed with its potential impact on interconnect reliability projection. Finally, the present status of electr omigration in Cu technology will be summarized with special focus on the early failure problem. This effort is greatly facilitated by the use of multiply-linked interconnect chains that permit greater precision in characterizing early failure distributions. Its use, its potential limitations, and some discussion of the statistically determined Blech-effect, time-permitting, will also be discussed.

4:20 PM

Effects of Mechanical Stress Evolution on Electromigration at No Current Stressed Area in Multilevel Interconnect Structure: *Young Bae Park*[1]; In Soo Jeon[1]; Young Ah Cho[1]; Hyuk Hyun Ryu[1]; Won Gyu Lee[1]; [1]Hynix Semiconductor, Inc., Adv. Logic Proc. Dvlp. Team, Sys. IC R&D Div., 1 Hyangjeong-dong Hungduk-gu, Cheongju-si 361-725 Korea

In addition to direct stress-voiding problem, electromigration-induced voiding is also strongly influenced by the mechanical stress evolution within metal lines, especially at no current stressed area (that is, reservoir region) in multilevel interconnect structure. Reservoir length (0.04 ~ 0.30μm) dependencies of electromigration lifetime in multilevel interconnect were compared for two passivation dielectrics, that is, FOX (Flowable Oxide) and HDP FSG (High Density Plasma Fluorinated Silicate Glass). Direct X-ray diffraction determination of the mechanical stress in Al-Cu interconnect lines was carried out with varying passivation dielectrics and metal line widths. Young's modulus of all constituent layers and residual stress of passivation dielectrics were measured by nanoindentation and wafer curvature method, respectively. Both finite element stress analysis and focused ion beam failure analysis for multilevel interconnect test structure were performed to investigate the effects of mechanical stress evolution on void nucleation and growth at no current stressed area during electromigration test. Possible mechanisms for passivation dielectric and reservoir length dependencies of electromigration lifetime in multilevel interconnect were discussed through measured stresses of metal lines and passivation dielectrics, mechanical properties of passivation dielectrics, microstructure failure analysis, and finite element stress analysis.

4:40 PM Invited

Critical Reliability Issues in Cu Interconnects: *Choong-Un Kim*[1]; [1]University of Texas at Arlington, Arlington, TX 76019 USA

Driven by desire for interconnects with improved reliability and performance, microelectronics companies are gradually transitioning from Al to Cu metallization in modern devices. With this transition, a new array of challenges are emerging because the physical properties of Cu, particularly those that influence microstructure and failure processes, are so different from those of Al. Over the past few years, we have investigated electromigration and thermal stress failure mechanisms in submicron Cu interconnects with a variety of structures, surrounding materials, and processing conditions. Our investigation reveals that the primary factors governing the reliability of Cu interconnects seem to be latent defects such as microvoids, poor adhesion, and interface microstructure instability, rather than the more conventional grain size and texture. This paper addresses metallurgical factors affecting the reliability of sub-micron Cu interconnects. This paper also introduces a new methodology for investigating Cu reliability, specifically designed to assess the influence of Cu-specific defects on reliability.

5:10 PM

Degradation of Electromigration Reliability due to Fencing: *Joon Kiat Low*[1]; Hun Sub Park[1]; Ismail Zainab[2]; Eng Chye Chua[2]; Lap Chan[2]; [1]Nanyang Technological University, Sch. of Matls. Eng.; [2]Chartered Semiconductor Manufacturing, 60 Woodlands Industrial Park D, Ste. 2, 738406 Singapore

Copper is the metal of the future, setting to replace aluminium and its alloys in the IC industry. With the use of copper, there begins the common usage of the dual damascene process due to the lack of volatile copper compounds. Fencing occurs when there is etch non-uniformity during the trench-patterning step of the dual damascene process. Such fences are undesirable as they negatively affect device reliability. The fences become important high stress points in the metal line structure. During the electromigration tests, such high stress points cause a different failure mechanism, leading to early failures of the tested samples possibly by current crowding. Experiments done to obtain the value of activation energy of copper electromigration shows

that with the presence of these fences, the value will drop to 0.25-0.33eV, much lower the expected value of 0.8eV for copper diffusion along the interface. The experiment will involve using FIB cuts to observe for the failure mechanism associated with the fence. This suggests that the presence of the fences provides an easier pathway for the samples to fail. The following SEM picture shows the presence of the mentioned fence.

Modeling of Multi-Scale Phenomena in Materials Processing: Deformation Analysis

Sponsored by: ASM International: Materials Science Critical Technology Sector, Materials Processing & Manufacturing Division, Jt. Computational Materials Science & Engineering, Solidification Committee

Program Organizers: Adrian Sabau, Oak Ridge National Laboratory, MS-602, Oak Ridge, TN 37831-6083 USA; Boyd A. Mueller, Howmet Corporation, Whitehall, MI 49461-1832 USA; Anthony D. Rollett, Carnegie Mellon University, Department of Materials Science & Engineering, Pittsburgh, PA 15213-2890 USA

Tuesday PM — Room: 304
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chair: Joachim H. Schneibel, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6115 USA

2:00 PM

A Continuum Approach to Stage IV Hardening: Schalk Kok[1]; *Armand J. Beaudoin*[1]; Daniel A. Tortorelli[1]; [1]University of Illinois at Urbana-Champaign, Dept. of Mech. & Indl. Eng., 1206 W. Green St. MC-244, Urbana, IL 61801 USA

A hardening law is developed that is capable of simulating stage IV hardening. The hardening law is based on a physically motivated dislocation density evolution equation, intended for use in a detailed FEM analysis. A remarkably simple result obtained from mesoscale FEM analyses, that lattice incompatibility evolves linearly with plastic strain, allows the development of a two state variable hardening law. Detailed examples are presented where the developed hardening law is embedded in a rate and temperature dependent viscoplastic polycrystal model. Large strain compression and torsion of both fcc (copper) and bcc (HY100 martensitic steel) metals are simulated. Grain-size effects can also be modeled, illustrated through the compression of coarse and fine grained silver. All material parameter estimates, and state variable initial conditions, are obtained simultaneously by solving an identification problem.

2:25 PM

Deformation Analysis of Superplastic Aluminum Alloy Al-8090 using Finite Elements: *Noha M. Hassan*[1]; Maher Y.A. Younan[2]; Hanadi A. Salem[2]; [1]The American University in Cairo, Mechl. Eng. Dept., 52 Mohy ElDin Abou El Ezz St., Mohandessein, Cairo Egypt; [2]The American University in Cairo, Mechl. Eng. Dept., 113 Kasr El Aini St., PO Box 2511, Cairo 11511 Egypt

The deformation behaviors of an Al-Li-Cu alloy (Aluminum 8090) during uniaxial and biaxial stress states were modeled using the nonlinear finite element analysis package ABAQUS 5.8. Two different material models were used for the purpose of comparison and to propose a valid and accurate super-plastic material deformation behavior. The two models are a material model with constant properties (strain rate sensitivity m, and strain hardening exponent n) and a material model with variable properties (m & n). The results from the numerical models were compared to the experimental results published by Chen and Huang (1995). The strain rate sensitivity, m, and strain hardening exponent, n, are dependable variables on the strain and strain rates. Even under extreme conditions of running the test with constant strain rates, these variables are still varying since it is impossible to keep the same strain and strain rate throughout section with time. Mainly due to this fact, the assumption of having constant properties when dealing with superplastic materials does not represent a reliable one. Upon comparing the two material models in both stress states whether biaxial or uniaxial, material model with variable properties gives better representation of the true behavior of the material when compared to the actual experimental results.

2:50 PM

Limit Strain Analysis of Superplastic Materials using Finite Elements: *Noha M. Hassan*[1]; Maher Y.A. Younan[2]; Hanadi A. Salem[2]; [1]The American University in Cairo, Mechl. Eng. Dept., 52 Mohy El-Din Abou El-Ezz St., Mohandessein, Cairo Egypt; [2]The American University in Cairo, Mechl. Eng. Dept., 113 Kasr El Aini St., PO Box 2511, Cairo 11511 Egypt

The failure behavior of superplastic materials was modeled using the nonlinear finite element analysis package ABAQUS 5.8 during both uniaxial and biaxial stress states. Fracture mechanism diagrams were used to predict the failure limit during deformation. The fracture mechanism diagrams utilized were chosen after a comprehensive study on plastic stability and strain to fracture during superplastic deformation. It considers the two modes of failure for superplastic materials whether plastic instability or cavitations. In order to expand the validity of the proposed failure model, it was further applied to different products using different materials. Free bulging and cone modeling were the geometrical variation considered for the assessment of this case. That is in addition to the uniaxial tension test. The two material models used were Al-8090 and Weldalite-049. Fortran subroutines were developed and added to the main program that calculated the failure strain based on the current stress-strain state of the material and terminated the analysis when exceeding the failure strain. Upon comparing the different models with the experimental results, the necessity for using the proposed failure model to achieve accurate results is seen. The necessity of accurate modeling is explained in relation to the industrial need for the pressure time profile obtained by accurate simulation of real life application.

3:15 PM

Phase Field Microelasticity Approach to Multi-Crack Evolution: *Yongmei M. Jin*[1]; Yu U. Wang[1]; Armen G. Khachaturyan[1]; [1]Rutgers, The State University of New Jersey, Dept. of Cer. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854-8065 USA

The Phase Field Microelasticity theory of an arbitrary 3D multiply connected elastically anisotropic system of voids/cracks under applied stress is proposed. The theory is based on the variational theorem which gives the exact solution of the elasticity equation for a system with voids/cracks. The theory reduces the problem of evolution of a crack system in elastically anisotropic crystal to a solution of the nonlinear integro-differential Ginzburg-Landau equation. The long-range strain-induced interaction of individual cracks is directly taken into account. Other defects, such as dislocations and precipitates, are trivially integrated into this theory. The proposed model does not require ad hoc assumption about possible crack configurations or their evolution paths. Examples of computations of elastic equilibrium of systems with voids/cracks and the evolution of cracks in single and polycrystal under applied stress are considered.

3:40 PM Break

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Finite Element Modeling of a Ball Plug and a Cylindrical Plug Press-Fit Systems: W. Kasprzak[1]; J. H. Sokolowski[1]; *D. Szablewski*[1]; N. Zamani[2]; [1]NSERC/Ford-Nemak/University of Windsor, Mech., Autom. & Matls. Eng., Rm. 203, Essex Hall, 401 Sunset Ave., Windsor, ON N9B 3P4 Canada; [2]University of Windsor, Mech., Autom. & Matls. Eng., Windsor, ON Canada

Press-fit systems are governed mainly by the plug/bore diameter difference, and plug shape. This paper addresses the plug shape performance. In the case studied the insertion of a ball plug and a cylindrical plug made from an Al alloy into a 319 Al alloy cast component was modeled using ANSYS 5.6 Finite Element Analysis software. Von Mises yield stress, and longitudinal axis stress distributions were analyzed for the ball and cylindrical plugs at the plug's final rest position. It was shown that cylindrical plugs offer a better surface contact with the hole wall over the ball plugs as well as distribute the stress evenly through the contact zone.

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Phase Field Microelasticity Approach to Dislocation Dynamics: *Yu U. Wang*[1]; Yongmei M. Jin[1]; Armen G. Khachaturyan[1]; [1]Rutgers, The State University of New Jersey, Dept. of Cer. & Matls. Eng., 607 Taylor Rd., Piscataway, NJ 08854-8065 USA

A 3D multi-dislocation system under applied stress is treated as a particular case of Phase Field Microelasticity theory of multi-variant stress-induced martensitic transformation previously developed in our group. This approach reduces the problem of evolution of a dislocation system in elastically anisotropic crystal to a solution of the nonlinear integro-differential Ginzburg-Landau equation describing the interaction between all dislocations as well as between dislocations and applied stress. In this formalism, the elastic interaction between dislocations and elastic coupling between grains are taken into consideration through the exact analytical solution of the elasticity problem. This approach also automatically describes the dislocation reactions such as multiplication/annihilation and does not require any ad hoc

assumption about them: the dislocations are "free" to choose the optimal evolution path. Examples of 3D computer simulations of the dislocation dynamics in single and polycrystal are considered.

4:45 PM
Submicron Resolution 3D Investigation of Nanoindent Deformation Structure in Cu using X-Ray Microbeams: *Wenge Yang*[1]; B. C. Larson[1]; G. E. Ice[1]; J. D. Budai[1]; G. M. Pharr[1]; J. Z. Tischler[1]; K.-S. Chung[1]; [1]Oak Ridge National Laboratory, PO Box 2008, Oak Ridge, TN 37831-6030 USA

We have developed a differential-aperture x-ray microscopy (DAXM) technique that provides submicron resolution, nondestructive, 3D spatial resolution measurements of the structure, orientation, and residual stress in single-crystal, polycrystal, and deformed materials using x-ray microbeams. Key elements of the technique will be described and methods of obtaining micron depth resolution will be discussed. Application of this technique to the investigation of the deformation structure produced by Berkovitch nanoindentations in single crystal copper will be presented. Micron resolution mapping of local misorientations to depths ~20 microns below the indent reveal intricate plastic deformation structures with dislocation boundary patterning. These results will be compared with available simulation and modeling. The work was performed on the MHATT-CAT beam line at the Advanced Photon Source (APS), which is supported by the US DOE Office of Science. The research was sponsored by the US DOE under contract DE-AC05-00OR22725 with ORNL, managed by UT-Battelle, LLC.

Phase Stability, Phase Transformations & Reactive Phase Formation in Electronic Materials - I

Sponsored by: Electronic, Magnetic & Photonic Materials Division, Structural Materials Division, Alloy Phases Committee
Program Organizers: Sinn-Wen Chen, National Tsing-Hua University, Department of Chemical Engineering, Hsinchu City 300 Taiwan; C. Robert Kao, National Central University, Department of Chemical Engineering, Chungli City, Taiwan; Hyuck Mo Lee, Korea Advanced Institute of Science & Technology, Department of Materials Science & Engineering, Taejon 305-701 Korea; Michael R. Notis, Lehigh University, Department of Materials Science, Bethlehem, PA 18015 USA; Douglas J. Swenson, Michigan Technological University, Department of Metallurgical & Materials Engineering, Houghton, MI 49931 USA

Tuesday PM — Room: 211
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Douglas J. Swenson, Michigan Technological University, Dept. of Metlgcl. & Matls. Eng., 1400 Townsend Dr., Houghton, MI 49931-1295 USA; H. M. Lee, Korea Advanced Institute of Science and Technology, Dept. of Matls. Sci. & Eng., Kusung-Dong 373-1, Yusung-Gu, Taejon 305-701 Korea

2:00 PM Invited
Contact Formation to IIIV Semiconductors and their Alloys: *Y. Austin Chang*[1]; [1]University of Wisconsin, Dept. of Matls. Sci. & Eng., 1509 University Ave., Madison, WI 53706-1595 USA

Application of metallurgical principles and characterization techniques to the study of metal/IIIV semiconductor interfaces has led to significant advances in IIIV contact technology. In this presentation, following a brief review of the physics of the metal-semiconductor interfaces, I will introduce a combined thermodynamic/kinetic model for an exchange reaction which occurs in a specific quaternary system involving the IIIV semiconductors. On the basis of this model, we can select appropriate contact materials which may react with a specific IIIV semiconductor (or an alloy of the semiconductors at a specific composition) at the interfaces in a manner as predicted by this model. Several examples involving gallium arsenides, i.e. n-GaAs, n-(Ga,Al)As, and n-(In,Ga)As, and gallium nitrides, i.e. n-GaN and n-(Ga,Al)N, will be presented to illustrate the utility of this approach.

2:25 PM Invited
Structural Evolution in Amorphous Semiconductors: *L. J. Chen*[1]; [1]National Tsing Hua University, Matls. Sci. & Eng., Hsinchu 300 Taiwan

The structure of amorphous semiconductor thin films has intrigued scientists for many decades. The structure evolution is also a long-standing problem. Auto-correlation function analysis is a statistical analysis of the scanned high-resolution transmission electron microscope images in real space. The technique is capable of detecting nanocrystallites as small as 1 nm in size embedded in the amorphous materials. For both electron beam evaporated and ion implanted amorphous silicon thin films, a high density of Si nanocrystallites was detected in as-deposited films. The density was found to diminish in amorphous films with annealing temperature first then increase. The results established the presence of Si nanocrystallites in as-prepared films and a decrease in density after annealing up to a certain temperature. The conclusions are discussed in the context of free energy change with annealing temperature. Results for amorphous Ge, Ge-Si and metal-Si films will also be presented.

2:50 PM Invited
Phase Transformations in Au/a-Si and Cu/a-Si Thin Film Multilayers: *Richard R. Chromik*[1]; Eric J. Cotts[1]; [1]SUNY Binghamton, Phys. Dept., Vestal Pkwy. E., Binghamton, NY 13902 USA

As dimensions of thin film devices decrease, the number of material interfaces and, often, the grain boundary density increases. The large surface energy and possible fast grain boundary diffusion in thin films of nanometer length scales can affect the thermodynamics and kinetics of reactions between two thin film components. Non-equilibrium phases and unique kinetics of reaction may be observed. We have studied solid state reactions in Au/a-Si and Cu/a-Si thin film multilayers using differential scanning calorimetry, x-ray diffraction, and transmission electron microscopy. In the Au/a-Si samples, two non-equilibrium crystalline silicides formed below 500 K. Each phase formed by a different mechanism, and the competing growth of the two phases over the temperature range of 375 K to 500 K was found to depend greatly on the thickness and grain size in the Au layers. At higher temperatures (500 K-600 K), these metastable phases decomposed and the a-Si crystallized by metal-induced crystallization (MIC) to yield an equilibrium phase mixture of Au and x-Si. The heat of crystallization of a-Si was measured to be -12.1±1.2 kJ/mol. For Cu/a-Si thin films, the parent phases readily interdiffused to form three equilibrium compounds: Cu_3Si, $Cu_{15}Si_4$ and Cu_5Si. The thermodynamics and kinetics of the formation of the Cu_3Si phase were studied in detail. The heat of reaction for the Cu_3Si phase forming from Cu and a-Si was measured to be -13.6±0.3 kJ/mol. Reaction constants measured for the growth of Cu_3Si in thin films were found to be 2-3 orders of magnitude smaller than for bulk samples studied by previous researchers. These results, combined with the observation of nanocrystalline Cu_3Si at the interfaces of as-prepared samples, were examined using kinetic theories that take into account the grain size.

3:15 PM Invited
Influence of the Environment on Contacts to GaN and AlGaN: *Suzanne E. Mohney*[1]; Eric D. Readinger[1]; Sammy Wang[1]; [1]Pennsylvania State University, Matls. Sci. & Eng., 109 Steidle Bldg., University Park, PA 16802 USA

Chemical interactions with the environment can greatly influence the electrical performance of contacts to the wide band gap semiconductors GaN and AlGaN. These interactions can occur either during annealing or when contacts are stored at room temperature in the laboratory. We first consider the exchange of nitrogen with the environment and its influence on phase formation during the annealing of contacts to GaN. This interaction can be predicted using thermodynamics, and it is important because the annealing environment can sometimes determine whether or not a contact becomes ohmic upon heat treatment. We have more recently investigated the aging of contacts to GaN and AlGaN stored at room temperature for just days in a laboratory drawer. Based on the results of environmental studies, we propose mechanisms for the dramatic improvement in Schottky barriers to n-AlGaN and the degradation of low resistance ohmic contacts to p-GaN with aging.

3:40 PM Break

4:00 PM Invited
Sputtered Copper Films with Dilute Insoluble Tungsten for Cu Metallization: A Thermal Annealing Study: *Jinn P. Chu*[1]; C. H. Lin[1]; [1]National Taiwan Ocean University, Inst. of Matls. Eng., No. 2, Pei-Ning Rd., Keelung 20224 Taiwan

Copper is an attractive material for metallization in microelectronics, because of its low resistivity and high reliability against electromigration compared with Al and its alloys. However, Cu diffuses readily into Si and SiO_2, resulting in the formation of copper silicide compounds at low temperatures. This work was directed toward a study of thermal stability, microstructure and electrical properties of Cu films containing dilute insoluble W in vacuum and rapid thermal annealing (RTA) conditions. Cu-2.3at.%W films deposited on Si(100) substrate by R.F. magnetron sputtering were annealed at temperatures ranging from 200 to 800°C. As-deposited Cu-W films consisted of non-equilibrium solid solutions of W in Cu with nanocrystalline microstructures. Annealed Cu-W films were examined by XRD, four-point

resistivity probe, SIMS depth profile measurement, SEM and TEM. Thermal stability of Cu-W films will be presented and discussed in light of results obtained.

4:25 PM
Nucleation Mechanisms of Copper during Electrodeposition of Thin Films from Ammoniacal Solutions: *Batric Pesic*[1]; *Darko Grujicic*[1]; [1]University of Idaho, Matls. Sci. & Eng., COMER-McClure Hall, Moscow, ID 83844-3024 USA

The electrodeposition nucleation mechanisms of copper thin films on glassy carbon from ammoniacal solutions were studied by utilizing cyclovoltammetric (CV) and chronoamperometric (CA) electrochemical techniques. Morphological characterization was performed by atomic force microscopy. The copper nucleation mechanisms were examined as a function of solution pH, copper concentration, ammonia concentration, solution buffering and oxygen concetration. Electrochemical surface conditioning effects were also studied. It was found that all parameters had profound effects on the morphology of copper nuclei in the thin film. Chronoamperometric studies were utilized as a diagnostic tool for mechanisms of copper nucleation. Also, thin film of copper produced from copper-ammonia system was radically different from the films produced from pure copper sulfate systems. In addition to the discussion of CV and CA results the proof for instantaneous nucleation mechanisms will be presented.

4:45 PM
Phase Transformations in NiMn and PtMn Thin Films: *Peter F. Ladwig*[1]; Y. Austin Chang[1]; [1]University of Wisconsin, Matls. Sci. Prog., 1509 University Ave., Madison, WI 53706 USA

NiMn and PtMn thin films are of interest to the magnetic recording industry for use in giant-magnetoresistive (GMR) devices. However, sputter deposited, equiatomic, Ni-Mn and Pt-Mn thin films are observed to possess metastable, nanocrystalline, chemically disordered, face-centered-cubic (fcc) structures which are not antiferromagnetic. This study investigates the microstructural evolution of these films to the antiferromagnetic L10 structure. Differential scanning calorimetry (DSC) experiments on these films reveal exothermic peaks. The peak location, area, and shift with heating rate are used to calculate reaction onset temperatures, enthalpies of reaction, and activation energies, respectively. Transmission electron microscopy (TEM), and electron/X-ray diffraction of annealed films characterize these identified reactions as grain growth and polymorphic phase transformations to the L10 structure. From the grain growth experiments, the fcc grain boundary energy is approximated and growth modes are determined. Changes in thermodynamic and kinetic properties with film thickness are also investigated.

Processing and Properties of Lightweight Cellular Metals and Structures
The MPMD Third Global Symposium
Mechanical Behavior of Cellular Metals - Session IV

Sponsored by: Materials Processing & Manufacturing Division, Jt. Computational Materials Science & Engineering, Powder Materials Committee, Jt. Processing Modeling Analysis & Control Committee, Surface Engineering Committee, Shaping and Forming Committee, Solidification Committee
Program Organizers: Amit K. Ghosh, University of Michigan, Department of Materials Science and Engineering, Ann Arbor, MI 48109-2136 USA; T. Dennis Claar, Fraunhofer USA, Newark, DE 19716 USA; T. H. Sanders, Georgia Institute of Technology, Department of Materials Science and Engineering, Atlanta, GA 30332 USA

Tuesday PM — Room: 205
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Joe Cochran, Georgia Institute of Technology, Matls. Sci. & Eng., 771 Ferst Dr., Atlanta, GA 30332-0245 USA; Winston O. Soboyejo, Princeton University, Dept. of Mechl. Aeros. Eng., Olden St., Princeton, NJ 08544 USA

2:00 PM
Mechanical Behavior of Periodic Hollow Sphere Foams: *Wynn S. Sanders*[1]; Lorna J. Gibson[1]; [1]Massachusetts Institute of Technology, Dept. of Matls. Sci. & Eng., 77 Massachusetts Ave., Rm. 8-135, Cambridge, MA 02139 USA

Many metallic closed-cell foams contain manufacturing defects such as cell wall curvature and irregular cell sizes which greatly reduce their overall mechanical properties. Eliminating these defects could improve performance by as much as a factor of ten at low densities. Hollow sphere foams provide a possible solution to the drawbacks of closed-cell metal foams because they can be manufactured into relatively defect-free structures. In this work, finite element modeling was used to evaluate the mechanical behavior of periodic hollow sphere structures. The relative thickness of the spheres and the bond size between the spheres was varied to produce relative densities of 2-20 percent. Both the elastic and yield behavior of the hollow sphere structures were fully evaluated. It was determined from the simulations that the mechanical performance of hollow sphere foams is between the theoretical performance of open- and closed-cell foams. Preliminary experiments have validated the finite element simulations.

2:25 PM
Mechanical Testing of IN718 Lattice Block Structures: *David L. Krause*[1]; John D. Whittenberger[2]; Pete T. Kantzos[3]; [1]National Aeronautics and Space Administration, Glenn Rsrch. Ctr., 21000 Brookpark Rd., MS 51-1, Cleveland, OH 44135-3191 USA; [2]National Aeronautics and Space Administration, Glenn Rsrch. Ctr., 21000 Brookpark Rd., MS 24-1, Cleveland, OH 44135-3191 USA; [3]Ohio Aerospace Institute, NASA Glenn Rsrch. Ctr., 21000 Brookpark Rd., MS 49-7, Cleveland, OH 44135-3191 USA

Lattice block construction produces a basic flat panel composed of thin ligaments of material arranged in a three-dimensional triangulated truss-like structure. Low-cost methods of producing cast metallic lattice block panels are now available that greatly expand opportunities for using this unique material system in today's high-performance structures. Additional advances are being made in NASA's Ultra Efficient Engine Technology (UEET) program to extend the lattice block concept to include superalloy materials. Advantages offered by this combination include high strength, light weight, high stiffness and elevated temperature capabilities. Recently under UEET, the nickel-based superalloy Inconel 718 (IN718) was cast into lattice block panels with great success. To evaluate the casting quality and the configuration merit, individual ligaments were extracted for tensile testing, and also structural compression and bend test specimens were machined from the panels. This paper first presents metallurgical and optical microscopy analysis of the castings. Then surprising mechanical test results are described that demonstrate the advantages in strength testing of redundant load paths inherent in lattice block, and it is shown that fatigue life is not sacrificed. Fractographic analysis is then presented for several ligament failure sites. Finally, future test plans are listed for this promising innovation.

2:45 PM
Structure and Deformation of Aluminum Lattice Block Structures: *Jikou Zhou*[1]; *Pranav Shrotriya*[1]; *Christopher Mercer*[1]; *W. O. Soboyejo*[1]; [1]Princeton University, Dept. of Aeros. & Mechl. Eng., D404 E-Quad, Olden St., Princeton, NJ 08544 USA

This paper examines the structure and deformation of aluminum lattice block structures. Following a multi-scale characterization of microstructure via scanning and transmission electron microscopy, the paper explores the effects of strut microstructure on the tensile and compressive deformation behavior. The measured variabilities in the strut properties are then characterized with appropriate statistical distributions before developing a mechanics framework for the estimation of lattice block deformation characteristics. The implication of the models are discussed for the functional design of lattice block structures.

3:05 PM
Mechanical Behavior of a Closed-Cell Aluminum Foam: *Carl M. Cady*[1]; George T. Gray[1]; Carl P. Trujillo[1]; Toshi Mukai[2]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA; [2]Osaka Municipal Technical Research Institute, Osaka 536-8553 Japan

The compressive deformation behavior of a closed-cell Aluminum foam manufactured by Alporas was evaluated under static and dynamic loading conditions as a function of temperature. High strain rate tests (2000/s) were conducted using the split Hopkinson pressure bar. Quasi-static and intermediate strain rate tests were conducted on a hydraulic load frame. There appears to be little change in the flow stress behavior as a function of strain rate, but the behavior is strongly influenced by temperature. Annealed specimens will be tested to evaluate the effect of pre-existing substructure and the strain rate sensitivity of the material. Localized deformation and Stress State instability issues will be discussed in detail since the behavior over the entire range of strain rates indicates non-uniform deformation.

3:25 PM Break

3:45 PM
Study on the Collapse of Egg-Box Core Sandwich Beams: *Marc Zupan*[1]; N. A. Fleck[1]; M. F. Ashby[1]; [1]University of Cambridge, Eng. Dept., Trumpington St., Cambridge CB2 1PZ UK

The desire of the shipbuilding and aerospace industries to replace conventional plate-beam metallic structures has driven the development of innovative lightweight structural concepts. The collapse modes of sandwich panels with aluminum egg-box cores of a square geometry and aluminum face sheets are investigated in uniaxial compression, shear and 3-point bending. The effects of core relative density and geometry of sandwich beam upon stiffness and collapse mode are explored, and compared with simple analytical models.

4:05 PM
3D Micro Tomography (µCT) of Cellular Metals using an up to 320kV X-Ray Tube: *B. Illerhaus*[1]; E. Jasiuniene[1]; A. Kottar[2]; [1]Federal Institute for Material Testing and Research (BAM), Berlin Germany; [2]Institute of Material Science and Testing, Vienna Univ. of Tech., Vienna Austria

Cellular metals were investigated by 3D micro tomography (3DµCT) using up to 320kV in a micro focus X-ray tube. The tomograms were investigated with 3d image processing methods. Aluminium foams as well as probes of hollow iron spheres were investigated. The aim was to enhance the quality of the foam by non destructive testing and to generate software tools which will be able to link the measured properties to mechanical parameters of foams. A way to characterize the density and the homogeneity at one glance is proposed. Internal deformation of probes after subsequent pressing is shown. A software tool calculates the partial shift in 3D and allows to combine it with other foam properties. The pores are detected and the pore size distribution is calculated. Also the centre of gravity and the volume of each pore is stored and thus allows to generate a FEM model of a real foam.

4:25 PM
An In-Situ Study on Compressive Deformation Mechanism of Metallic Foams: *Jikou Zhou*[1]; Rob Cirincione[1]; *Robert Anderson*[1]; *Pranav Shrotriya*[1]; *Christopher Mercer*[1]; *W. O. Soboyejo*[1]; [1]Princeton University, Dept. of Mech. & Aeros. Eng., D404 E-Quad, Olden St., Princeton, NJ 08544 USA

This paper presents the results of a combined experimental and analytical/computational in-situ study on the compressive deformation mechanism in porous aluminum alloys and Titanum alloys. Following a brief description of pore and cell wall/strut structure, the micromechanisms of compressive deformation are discussed by presenting the results of incremental and continuous experiments on foams. The observations reveal a complex sequence of cell wall plastic buckling, slip band formation and cell wall collapse in metallic foams. The observed phenomena are modeled within a mechanics framework that explores the effects of strut deformation on the deformation and strength of foams.

4:45 PM Cancelled
Evolution of Plastic Deformation in Aluminum Foam: *Zhenlun Song*

4:45 PM
Tailored Component Fabrication using Stabilized Aluminum Foam: *Greg Mills*[1]; Scott Nichol[1]; [1]Cymat Corporation, 1245 Aerowood Dr., Missasauga, ON L4W 1B9 Canada

The use of stabilized aluminum foam (SAF) in component design can have numerous advantages. By taking advantage of SAF's ability to provide energy absorption, acoustic damping, lightweight, electromagnetic shielding, recyclable or non-flammable nature, components can be tailored to meet a broad range of criteria with a single material. With these material advantages in mind, Cymat is targeting the transportation industry as having the greatest need for a product such as ours. The use of SAF in automotive interiors and crush zones can provide increased passenger safety as well as producing a more fully recyclable automobile. Along with automotive opportunities, applications in architectural and military applications are also being developed. Cymat Corp is in a position to provide these benefits in a variety of forms; with our foam technology we are capable of producing a wide range of densities and thickness in foam panels and 3-D castings.

Recycling – General Sessions: Dross Processing and Aluminum Recycling: Joint Cast Shop and Recycling Session

Sponsored by: Extraction & Processing Division, Light Metals Division, Recycling Committee
Program Organizers: John N. Hryn, Argonne National Laboratory, Argonne, IL 60439-4815 USA; Wolfgang Schneider, VAW Aluminium AG, Reseach & Development Manager Cast Technology, Bonn 53177 Germany

Tuesday PM — Room: 604
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Han Spoel, DrossTec, Inc., Toronto, Ontario M5R 1W8 Canada; Donald L. Stewart, Jr., Alcoa Techl. Ctr., Alcoa Center, PA 15069-0001 USA

2:00 PM Invited
Review of Aluminum Dross Processing: *Ray D. Peterson*[1]; C. Lee Newton[2]; [1]IMCO Recycling, Inc., PO Box 268, 397 Black Hollow Rd., Rockwood, TN 37854 USA; [2]IMCO Reciclaje de Mexico, S. de R. L. de C. V., Monterrey, Nuevo Leon Mexico

Dross is a by-product of melting aluminum metal. It is a mixture of aluminum metal and aluminum oxide with minor amounts of other constituents. The type and "quality" of a dross is determined by the method of melting, the initial feed materials, temperature, and agitation to name just a few of the process variables. This paper will review current common dross processing practices and as well as piloted and proposed processing methods. The paper will discuss how drosses and their by-product residues may be treated in the near future. The impact of the dross-producing plant practices on metal recovery will be examined. A discussion on maximizing "Total Metal Recovery" will be made. Finally, dross is not a waste - it is a by-product with significant value. A short discussion will be made on responsible handling and processing in an efficient manner to maximize the value of the dross.

2:25 PM
Plant Operating Results on Hot Dross Processing at Hydro Aluminum Holmestrand Rolling Mill AS: *Niels Erik Hald*[1]; David J. Roth[2]; [1]Hydro Aluminum Hycast AS, Holmstrand Rolling Mill AS, PO Box A, Holmstrand 3081 Norway; [2]Altek, LLC

The Hydro Aluminum Holmestrand Rolling Mill has been operating two ALTEK Model 110 Tilting Rotary Furnaces since August of 2000. This facility, in operation since 1917, is a hot and cold rolling facility with casting capabilities of 100,000 tons per year of sheet ingot. These rotary furnaces were purchased to process dross hot from their melting and holding furnaces. Local management's goal was to maximize aluminum unit recovery in order to optimize the facility's output of quality cast pounds. The average metal recovery of the dross has increased by 6% from the base line of processing with a rotary cooler and dross press. This paper will discuss the procedure of hot dross processing at a rolling mill facility. It will focus on the important aspects of making such a system successful and detail other advantages of hot dross processing besides aluminum recovery. We will also discuss future activities to further improve the recovery from the dross.

2:50 PM
Impact of Parameter Changes on the Aluminum Recovery in a Rotary Kiln: *Bernd Friedrich*[1]; Abyl Sydykov[1]; Alexander Arnold[1]; [1]RWTH Aachen University of Technology, IME - Inst. of Proc. Metall. & Metal Recycling, Intzestr. 3, Aachen 52056 Germany

The metal recovery of the aluminum scrap remelting in a short rotary kiln is influenced by metal losses in salt slags due the luck of coalescence of dispersed droplets. The polythermic section of the ternary system NaCl-KCl-CaF_2 related to salt fluxes was studied. The effects of fluoride type and concentration, kind of aluminum alloy and temperature on the coalescence of aluminum droplets in salt flux was investigated. The remelting process of a coarse metal fraction from salt slag recycling in a rotary kiln was investigated to research the effects of used salt flux quantity, fluoride addition, aluminum oxide content, temperature and rotation speed of kiln on metal yield, salt slag properties and its constitution. The efficiency of aluminum recovery in a rotary kiln depends also on the salt flux constitution and charge, aluminum oxide content, temperature, melt stirring, constitution of salt slag.

3:15 PM
Recycling Aluminum Salt Cake: J. N. Hryn[1]; *G. K. Krumdick*[1]; [1]Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439 USA

Electrodialysis technology was used to recover salt from brines generated in the commercial process for recycling aluminum salt cake. Salt cake, a waste stream from the aluminum industry, contains aluminum metal, salt (NaCl and KCl), and nonmetallic impurities. After grinding and screening to separate out aluminum metal, salt cake solids are leached in water and filtered to recover a nonmetallic product (NMP). The filtered brine is then processed in an evaporator to separate salt and water. Researchers at Argonne National Laboratory identified electrodialysis as a promising process for salt recovery that may be less cost prohibitive than evaporation. A large-scale electrodialysis pilot plant has been designed and constructed to verify laboratory results and the commercial viability of the process. Results of pilot plant tests performed with industrially generated brine will be presented. Conditions that result in cost-effective operation of the technology will be discussed. The cost-effective scenario for salt recovery from salt cake brine consists of using electrodialysis in tandem with a small evaporator or crystallizer.

3:40 PM Break

3:50 PM
Decoating of Aluminium Scrap in Different Atmospheres: *Anne Kvithyld*[1]; Thorvald Abel Engh[1]; Rita Illés[2]; [1]Norwegian University of Science and Technology, Dept. of Matls. Sci. & Electrochem., Alfred Getz vei 2b, Trondheim 7491 Norway; [2]Budapest University of Technology and Economics, Inst. of Gen. & Analytl. Chem., Szt. Gellért tér 4, Budapest 1111 Hungary

Aluminium scarp contains (more often than not) lacquer and paint, which can be destroyed by heating prior to melting the scrap. The degradation of polyester coating on aluminium sheets has been studied (in air, O2, N2, Ar, and Ar with 1% O2) using thermal analysis and mass spectrometry. The samples were heated up to 620°C (heating rates 0.5-50°C/min). The data have been used to develop a phenomenological model of pyrolysis and combustion of the coating. A second model estimating the activation energy and constant-temperature degradation time is also proposed. The models can be used to choose a suitable atmosphere, temperature and residence time in an industrial delacquering unit.

4:15 PM
Refining Aluminium Scrap with Fractional Crystallisation: Assessing its Feasibility with Thermodynamics: *Wim Boender*[1]; Cor J. Waringa[1]; Gerben P. Krielaart[2]; Anton Folkertsma[2]; Dirk Verdoes[3]; [1]Corus Research, Development & Technology, PO Box 10,000, IJmuiden 1970 CA The Netherlands; [2]TNO Industrial Technology, Apeldoorn The Netherlands; [3]TNO Environment, Energy and Process Innovation, Apeldoorn The Netherlands

TNO and Corus RD&T are working on the modification of the TNO-Thijssen process for fractional crystallisation so that it can be applied to the purification of molten aluminium scrap. This process comprises a suspension crystalliser, which produces relatively pure aluminium crystals suspended in molten metal, and a hydraulic wash column, which subsequently separates these crystals from the relatively impure liquid. The thermodynamic background of the crystalliser has been studied to establish the applicability of the process. To that end, the thermochemical programs ThermoCalc, and ChemSage have been used to calculate the solidification paths for the alloy systems studied. DSC experiments have been performed to check their results. The upshot of the calculations and the measurements is that fractional crystallisation can purify these alloy systems. Whether this process will meet the requirements for the purification of a specific scrap type depends on the behaviour of alloying elements or impurities present. Other important factors are the requirements for the product and the residue, and the desired yield of the process.

4:40 PM Cancelled
The Ultimate Aluminum Extrusion Scrap Recycling System: *Roger A.P. Fielding*

5:05 PM
A New Aluminum Recycling Process for High Performance Foam Materials: *Won Ha*[1]; Shae K. Kim[1]; Young-Jig Kim[1]; [1]Sungkyunkwan University, Sch. of Metlgcl. & Matls. Eng., Solidification Control Lab., 300, Chunchun-dong, Jangan-gu, Suwon, Gyeonggido 440-746 Korea

Aluminum has good recyclability because it is resistant to corrosion under most environmental conditions and a low ratio of energy is required to remelt aluminum scrap compared with that required for its primary production. However, the products, which were manufactured by the recycled aluminum, are within the limits of the same or lower performance level compared with the primary one. Therefore, to overcome this limit of recycling process and develop high-profit recycling process, we have tried to produce sound-absorbing, ultra-light and heat-resistant aluminum foam materials directly from aluminum scraps. In this paper, we discuss the relationships between the melt viscosity and the surface area of scarp, the effect of calcium on the melt viscosity and the optimum amount of the foaming agent with regards to the melt viscosity and pore sizes and distributions.

Second International Symposium on Ultrafine Grained Materials: Theory and Modeling

Sponsored by: Materials Processing & Manufacturing Division, Shaping and Forming Committee
Program Organizers: Yuntian Ted Zhu, Los Alamos National Laboratory, Materials Science and Technology Division, Los Alamos, NM 87545 USA; Terence G. Langdon, University of Southern California, Department of Mechanical Engineering, Los Angeles, CA 90089-1453 USA; Terry C. Lowe, Technanogy, Newport Beach, CA 92627 USA; Rajiv S. Mishra, University of Missouri, Metallurgical Engineering, Rolla, MO 65409-0340 USA; Michael Jeremi Saran, Case Western Reserve University, Cleveland, OH 44106 USA; S. Lee Semiatin, Air Force Research Laboratory, Materials & Manufacturing Directorate, WPAFB, OH 45433 USA

Tuesday PM — Room: 210
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: S. Lee Semiatin, Air Force Research Laboratory, Matls. & Mfg. Direct., WPAFB, OH 45433 USA; Michael J. Saran, OES, Inc., 3715 Traynham Rd., Cleveland, OH 44122 USA

2:00 PM Keynote
Deformation of Ti-6Al-4V via Equal Channel Angular Extrusion: *David P. DeLo*[1]; S. Lee Semiatin[2]; [1]Extrude Hone Corporation, 1 Industry Blvd., Irwin, PA 15642 USA; [2]Air Force Research Laboratory, Matls. & Mfg. Direct., AFRL/MLLM, Wright-Patterson Air Force Base, OH 45433-7817 USA

Macroscopic and microscopic deformation effects during the equal channel angular extrusion of Ti-6Al-4V billets were established using physical experiments and finite element modeling (FEM) techniques. Experimental observations of ECAE processing ranged from localized flow and fracture to uniform shearing deformation. Coupled simulations and experiments were used to gain insights into the effects of material properties and processing conditions on deformation behavior. Billets subjected to an initial increment of prestrain followed by ECAE deformation exhibited partially globularized, directional microstructures with macrotextural strengths that depend on the processing route. Issues associated with process modeling, process design, and scaleup are discussed.

2:25 PM Invited
Polycrystal Constitutive Modeling of ECAP: Texture and Microstructural Evolution: Irene J. Beyerlein[1]; Ricardo A. Lebensohn[1]; *Carlos N. Tome*[1]; [1]Los Alamos National Laboratory, MST-8, MS G755, Los Alamos, NM 87545 USA

To date many experimental studies demonstrate that Equal Channel Angular Processing (ECAP) can potentially refine the grain size of coarse-grained high strength alloys to at least a few hundred nanometers. Our work develops a model to predict both texture development and grain refinement during the ECAP process. The model is built on the Visco Plastic Self Consistent (VPSC) 3D model for describing the plastic constitutive response of polycrystalline aggregates. Currently an empirical grain size evolution model is employed for insightful comparison with microstructural characterization. The influences of processing route, number of passes, grain size dependent constitutive law, and grain refinement criteria on the texture and grain size distribution are discussed. With these results and modeling approach, we will progress towards achieving predictive capability of the microstructures expected from various ECAP processing routes, crucial for advancing understanding and optimal design of this ECAP technology.

2:45 PM Invited
Structural Studies of Nanocrystalline Intermetallic Aluminides and Ordering Phenomena: *Hans-Eckhardt Schaefer*[1]; Klaus Reimann[1]; [1]Stuttgart University, Inst. of Theoretl. & Appl. Phys., Pfaffenwaldring 57, Stuttgart 70550 Germany

In nanocrystalline FeAl, which is athermally discovered by plastic deformation, ordering as studied by x-ray diffraction occurs at 420 K upon isochronal annealing. The disordering originates from the generation of antisite atoms as concluded from the lattice parameter increased in comparison to the ordered phase. The ordering, which occurs by the formation of ordered nanodomains within the 20 nm crystallites, can be quantitatively modeled by migrating vacancies employing a migration enthalpy which decreases from a value of 1.7 eV in the ordered state of 1.1. eV in the fully disordered state. The disappearance of the antiphase boundaries and the full annealing to the lattice parameter at temperatures exceeding the temperature range of the ordering process is ascribed to the formation and migration of thermal vacancies. The annealing of the relatively high elastic strains appears to be affected by the ordering and the growth of the ordered domains.

3:05 PM
Evaluation of Strain Rate during Equal Channel Angular Pressing (ECAP): *Hyoung Seop Kim*[1]; [1]Chungnam National University, Dept. of Metall. Eng., Yusong, Taejon 305-764 Korea

In the present study, the strain rate of workpiece materials during equal channel angular pressing (ECAP) was evaluated using a geometric approach. The results were compared to those of the finite element analyses of a model ideally plastic material with various mesh sizes. The derived equation for strain rate is in reasonably good agreement with the results of the finite element method. The effects of the die geometry (a channel angle and a corner angle) were investigated. The strain rate during ECAP increases with punch speed and decreases with die channel angle, die corner angle and the width of the workpiece. The relation obtained can be used for analytical calculations of the deformation, thermal and microstructural evolution behavior of materials during ECAP. In particular, the size of the deforming zone and the effect of the finite element size have been discussed.

3:20 PM
Analysis of Principal and Equivalent Strains in Equal Channel Angular Deformation: *Kenong Xia*[1]; Jingtao Wang[2]; [1]University of Melbourne, Dept. of Mech. & Mfg. Eng., Parkville, Victoria 3010 Australia; [2]Xi'an University of Architecture and Technology, Sch. of Metlgcl. Eng., 13 Yanta Rd., Xi'an 710055 China

Only shear and "equivalent" strains are commonly used to describe the intensity of equal channel angular deformation (ECAD). However, the equations used in their calculation are often only valid when the strain concerned is very small, and it is important to know about the principal strains. In this analysis, the magnitude and direction of the principal strains in ECAD were calculated using both a geometric method and a matrix algebra method which are suitable for describing large plastic deformation. It showed that the principal strain axes rotated during ECAD although the principal stress axes remained fixed. It was further demonstrated that the so-called "equivalent strain" widely used in the literature was not an invariant as its name appeared to suggest. It instead varied depending on what strains were used in its calculation and should be used with caution. The matrix algebra method represents a potential analytical tool for predicting the geometric shapes after ECAD.

3:35 PM
Kinematics of Damage Governed by Severe Plastic Deformation: *Rimma Ye. Lapovok*[1]; R. E. Cottam[1]; [1]Monash University, Sch. of Phys. & Matls. Eng., Melbourne, Victoria 3800 Australia

SPD techniques are known as a tool to produce fine-grained materials, which after that can exhibit superplastic behaviour in a certain range of thermo-mechanical parameters. However, damage introduced during SPD processing can be a reason for further cavitation in superplastic forming. The development and recovery of damage in continuously cast aluminium alloy 6061 is investigated using the processes of Equal Channel Angular Extrusion and Drawing. The development of damage was observed to increase proportionally with the extent of accumulated plastic shear strain. The influence of stress history, characterised by a stress index, was found to be twofold. First, the stress index defines the intensity of the porosity development, which increases with the stress index as it changes from negative to positive values. Second, the stress index, when in the negative value region, governs the recovery process. The kinematic equation for damage evolution is proposed and its coefficients are defined.

3:50 PM Break

4:00 PM Keynote
Mechanisms of Deformation of Nanostructured Metals: *Robert J. Asaro*[1]; [1]University of California, Dept. of Struct. Eng., La Jolla, CA 92093 USA

This talk will first review the currently understood phenomenology of inelastic deformation of nanostructured metals and alloys. Specific attention will be placed on the behavior of nanostructured metals and alloys processed via severe plastic deformation (SPD) methods. The perspective obtained will then be used to develop a framework for describing the mechanisms of deformation and contrasting them with the deformation mechanisms that occur in polycrystalline METALS with grain sizes more common in structural alloys. Quantitative models to describe deformation will be presented, in particular models that account for mechanisms such as grain boundary sliding and grain rotation. Local accommodation processes, that are required to support grain boundary sliding and grain rotation, including grain matrix slip and (very) short range diffusion at grain boundary junctions will be detailed as part of the modeling. Both analytical and computational models that include combined grain matrix crystal plasticity and grain boundary sliding will be presented.

4:25 PM Invited
Strength and Ductility of Ultra Fine Grained Metallic Materials: *Yuri Estrin*[1]; *Hyoung Seop Kim*[2]; [1]IWW, Clausthal University of Technology, Agricolastr. 6, Clausthal-Zellerfeld D-38678 Germany; [2]Chungnam National University, Dept. of Metlgcl. Eng., Taejon 305-764 Korea

A constitutive model designed to describe the mechanical properties of ultra fine grained metallic materials will be discussed. A single-phase material is considered to be made up of two phases: the grain interiors and the grain boundaries. The deformation of the interior of a grain is taken to occur by a combination of the dislocation glide mechanism and the diffusion flow by lattice and grain boundary diffusion. The deformation of the grain boundary phase is controlled by diffusion flow alone. A particular example to be presented is that of Cu for which the strain hardening behaviour and tensile ductility were determined as a function of the average grain size. The effect of grain size distribution was also included. In agreement with experiment, a pronounced decrease of ductility with grain size reduction was found at room temperature.

4:45 PM Invited
Features of Severe Plastic Deformation as Compared to Conventional Deformation Modes: *Michael Josef Zehetbauer*[1]; [1]Universitaet Wien, Inst. fuer Materialphysik, Strudlhofgasse 4, Wien A-1090 Austria

Different stress-strain characteristics of a material deformed by different conventional deformation modes get identical if one considers the texture evolution being specific of the deformation mode. Densities of deformation induced lattice defects, and also the average sizes of cells/subgrains are identical, although their shape is highly anisotropic with ultra fine grain (UFG) size in one dimension only. After Severe Plastic Deformation (SPD) the grain shape is much more isotropic, with UFG sizes in all dimensions. This effect is due to the enhanced hydrostatic pressure at SPD techniques which not only allows for much larger strains but also for much higher lattice defect densities, yielding a natural explanation for the higher density of grain boundaries. The higher defect densities also suggest that the enhanced hydrostatic pressure restricts the dynamic defect annihilation, via restriction of lattice diffusion. A former composite-type model has been adapted to these ideas and gives good fits to the experimental data.

5:05 PM
Numerical Analysis of Plastic Deformation in Constrained Groove Pressing: *Jong-Jin Park*[1]; Dong-Hyuk Shin[2]; [1]Hong-Ik University, Mech. & Sys. Design Eng., 72-1, Sangsu-Dong, Mapo-Ku, Seoul 121-791 S. Korea; [2]Hanyang University, Metal. & Matls. Sci., Ansan, Kyunggi-Do 425-791 S. Korea

A new intense plastic straining technique, CGP (constrained groove pressing), has been developed for fabrication of ultrafine-grained metallic materials. In CGP, the material is subjected to repetitive shear deformation under the plane strain condition and the strain can be accumulated as comparable as that can be obtained by ECAP. However, it was found that there is a discrepancy of grain size in the thickness direction, which might be due to non-uniform plastic deformation in that direction. In the present study, the plastic deformation during CGP was analyzed by the rigid-plastic finite element method and normal and shear strains were obtained at different locations in the thickness. It was found that, along with large rotation, the surface undergoes stretching while the core undergoes shear strain, and that the effective strain at the core is greater than that at the surface. CGP was then fully developed into a CGR (constrained groove rolling), where the friction at the roll and the material could add some degree of shear strain at the surface which could help to improve the plastic deformation to be near uniform in the thickness direction. The present paper will discuss

more aspects found in the numerical as well as experimental investigations of CGP and CGR.

5:20 PM
The Work Hardening Behavior of Aluminium over a Wide Range of Strain: *Chinh Q. Nguyen*[1]; György Vörös[1]; Zenji Horita[2]; Terence G. Langdon[3]; [1]Eötvös University, Dept. of Gen. Phys., 1117 Pázmány P. sétány 1/A., Budapest Hungary; [2]Kyushu University, Dept. of Matls. Sci. & Eng., Fukuoka 812-8581 Japan; [3]University of Southern California, Dept. of Aeros. & Mech. Eng. & Matls. Sci., Los Angeles, CA 90089-1453 USA

The work hardening of high purity (4N) aluminium was investigated over a wide range of strain. The true stress-true strain curves of both annealed and equal-channel angular pressed (ECAP) Al samples were analyzed mathematically after deformation in tensile and compression tests. In this analysis, the absolute strain calculated from the annealed state was used where the annealed state relates to zero strain and the ECAP samples have an imposed strain before subsequent deformation in tensile or compression testing. The results show that in the range of low strains, as in the deformation of annealed samples, the rate of hardening is always positive and the magnitude of the hardening rate decreases monotonously with the total strain. In the range of high strains, as in the samples subjected to ECAP, the yield stress saturates because a homogeneous microstructure is ultimately established through ECAP processing.

Surface Engineering: Science & Technology - II: Nanotechnology

Sponsored by: Materials Processing & Manufacturing Division, Surface Engineering Committee
Program Organizers: Ashok Kumar, University of South Florida, Department of Mechanical Engineering, Tampa, FL 33620 USA; Yip-Wah Chung, Northwestern University, Department of Materials Science & Engineering, Evanston, IL 60208 USA; Gary L. Doll, The Timken Company, Canton, OH 44706 USA; D. S. Misra, Indian Institute of Technology-Bombay, Department of Physics, Powai, Mumbai 400076 India; John J. Moore, Colorado School of Mines, Department of Metallurgy and Materials Engineering, Golden, CO 80401 USA; Kryoshi Yatsui, Nagoka University of Technology, Nagaoka, Niigata 840-2188 Japan

Tuesday PM — Room: 203
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: John J. Moore, Colorado School of Mines, Golden, CO 80401 USA; J. Narayan, North Carolina State University, Dept. of Matls. Sci. & Eng., Raleigh, NC 27695 USA

2:00 PM Invited
Advantages and Challenges of Nanomaterials and Nanotechnology: *J. Narayan*[1]; [1]North Carolina State University, Dept. of Matls. Sci. & Eng., Raleigh, NC 27695-7916 USA

Nanomaterials and devices thereof offer many advantages over conventional coarse-grained counterparts. By varying nanocrystallite size and interfacial properties, it is possible to control mechanical properties and create functionally gradient thin films and composites. Superior electronic properties can be obtained by confining carriers within nanocrystallites where they do not see defects typically present in large-grained materials. Similarly, single-domain nanomagnets offer many advantages over multi-domain coarse-grained counterparts. However, these advantages can be realized only if processing challenges can be overcome to create suitable nanostructured materials. Nanocrystalline materials have been synthesized extensively in powder form or in solution. However, assembling or processing of these materials into useful form invariably leads to creation of porosity “artifacts” and the presence of undesirable interface states. Using a controlled pulsed laser processing, we have produced "artifact" free thin films and composites and measured “true” properties of nanocrystalline materials. Similarly, uniform-size nanodots (produced by self-assembly processing) were embedded into well-characterized matrices to study quantum confinement effects and correlate with optical and electronic properties. Single-domain to multi-domain magnetic properties have been obtained by tuning the size via our self-assembly processing.

2:25 PM
Dense Deposition of Nanocomposites by a Compact YAG Laser: *Mamoru Senna*[1]; Kenji Hamada[2]; [1]Keio University, Fac. of Sci. & Tech., 3-14-1 Hiyoshi, Kohoku-ku, Yokohama 223-8522 Japan; [2]Nara Machinery, 2-5-7 Jonanjima, Oota-ku, Tokyo 143-0002 Japan

Dense nanocoating of ceramers and complex glasses was achieved on a flat or particulate substrates by a laser ablation technique. Microstructure and chemical states of the coated nano composite layer were analyzed by electron microscopy and various spectroscopic techniques. A compact YAG laser apparatus equipped with substrate holder and particle fluidizer was devised. Conditions to make nanolayers as dense and as homogeneous as possible were discussed.

2:40 PM
Synthesis and Characterization of Thermally Stable TiB_2TiC Nanolayered Superlattice Coatings: *Kitty W. Lee*[1]; Yip-Wah Chung[1]; Leon M. Keer[2]; Kornel Ehmann[2]; [1]Northwestern University, Matls. Sci. & Eng., 2225 N. Campus Dr., Evanston, IL 60208-0876 USA; [2]Northwestern University, MEAS Mechl. Eng., 2145 Sheridan Rd. #A319, Evanston, IL 60208-3109 USA

Thermally stable TiB_2TiC nanolayered superlattice coatings were synthesized by dc dual-cathode magnetron sputtering at various substrate biases and rotation speeds. We demonstrated that these nanolayered superlattice coatings, with TiB_2(001) and TiC(111) preferred orientations, are thermally stable against interdiffusion after annealing in vacuum for 1 hour at 1000C. Transmission electron microcopy and low-angle x-ray diffraction studies confirmed that layer structures were preserved after annealing. Room-temperature hardness of these coatings approaches 50 GPa both before and after annealing, far exceeding the rule-of-mixture value. Wear and durability tests on coated M2 steel and C6 WC cutting inserts demonstrated the improved tribological performance of these coatings under unlubricated conditions compared with other standard coatings such as TiN. These thermally stable coatings are therefore desirable for protection of cutting tools in dry machining applications. In order to have more quantitative information on the feasibility for dry machining, coatings were separately exposed in argon gas and an oxidative environment at elevated temperatures. Substrates included Si(001) and C6 WC cutting tool inserts. These coatings were characterized before and after the heat treatment in terms of surface roughness, residual stress and nanoindentation hardness. These results will be presented.

2:55 PM
Multiscale Modeling of the Effects of Nanostructure at Organic-Inorgnic Interfaces on Bulk Mechanical Response in Bilological Nanocomposites: *Kalpana S. Katti*[1]; Dinesh R. Katti[1]; Mohamed Matar[1]; [1]North Dakota State University, Civil Eng., CIE 201, Fargo, ND 58105 USA

Nacre, the inner layer of seashells, is a laminated, segmented, hybrid nanocomposite of 10-20 nm-thick organic matrix and ~250 nm-thick CaCO3 pseudo-hexagonal aragonitic platelets. We have developed a multiscale modeling approach that predicts mechanical responses of nacre, incorporating experimentally-determined nanoscale properties into mesoscale 3D- numerical models. Structural details of nano and micro-structural characteristics including aragonite crystallography and morphology are incorporated in the models. The local properties of the individual components, biogenic aragonite and the organic matrix, measured by nanoindentation are used. Our numerical simulations indicate that the organic layer, has high yield strength and elastic modulus, orders of magnitude higher than those of synthetic polymers. Organic-inorganic interfacial characteristics are also incorporated in our simulations. Specifically, the role of nanoscale asperities and chemical compatibility at these interfaces is numerically evaluated. This represents an approach towards a simulation-based design of advanced nanocomposite systems mimicking those found in nature.

3:10 PM
Purification of Aluminum Nitride Nanosize Powder Synthesized by Pulsed Wire Discharge: C. Cho[1]; Y. Kinemuchi[1]; T. Suzuki[1]; H. Suematsu[1]; W. Jiang[1]; *K. Yatsui*[1]; [1]Nagaoka University of Technology, Extreme Energy-Density Rsrch. Inst., 1603-1 Kamitomiokamachi, Nagaoka, Niigata 940-2188 Japan

Aluminum nitride (AlN) nanosize powders have been synthesized by a novel pulsed wire discharge (PWD) method. Although their grain sizes were as small as 30 nm, their purities were at most 80%. In the present study, the effect of circuit inductance on the purity of the synthesized powder was investigated. An aluminum wire was placed in a chamber filled with mixed gas of nitrogen and ammonia and was discharged by charged capacitors. Between the wire and the capacitors, an additional coil was inserted to change the circuit inductance. From the quantitative analysis by X-ray diffraction, the volume fraction of

the AlN powder increased with the decrease in the circuit inductance. The waveform of each discharge indicated that the absorbed energy to the wire increased with the decrease in the circuit inductance.

3:25 PM Invited

Formation of Photonic Nonocomposites by Surface Engineering over Inorganic Nanoparticles: *Nobuyuki Kambe*[1]; [1]NanoGram Corporation, 46774 Lakeview Blvd., Fremont, CA 94538 USA

Surface engineering of inorganic nanoparticles can add or substantially enhance physical functions. NanoGram has developed a new class of nanoparticle-polymer composites by chemically bonding premade nanoparticles with organic polymers through surface-modifying linkage molecules. This technology has immediate applications in photonics. Photonic device fabrication requires particularly precise control of the refractive index. NanoGram has been able to finely tune the refractive index through a linear combination of the index for nanoparticles and for the polymer hosts. Successful index tuning requires, among other properties, (a) high levels of uniformity in nanoparticle size and shape and (b) high levels of particle dispersion in solvent both before and after surface modification. This paper outlines the important nature of nanoparticles and nanocomposites synthesized by NanoGram's technology. Self-assembled structures have been observed in coatings of nanocomposites and are discussed in relation to surface modifications.

3:50 PM Break

4:05 PM Invited

Conformal Coatings for LIGA MEMS: Issues and Challenges: *S. V. Prasad*[1]; T. R. Christenson[1]; [1]Sandia National Laboratories, Albuquerque, NM 87111 USA

Many microelectromechanical systems (MEMS) fabricated by LIGA [German acronym for Lithography, Galvanoformung (electroforming) and Abformung (molding)] utilize electrodeposited metals such as nickel and Ni alloys. While Ni alloys may meet the structural requirements for MEMS, their tribological (friction and wear) behavior remains somewhat undefined. For instance, the friction coefficient of pure Ni (μ = 0.6 to 1.2) does not meet the design criteria. Additionally, generation of wear debris and the stick-slip behavior could interfere with the performance and reliability. In a number of microsystems applications such as gear trains, comb drives and transmission linkages, tribological considerations, particularly sliding contacts amongst sidewalls, is of paramount importance. The miniature nature of LIGA MEMS elements–several hundred microns to millimeters in size–poses a tough challenge to the coating technology. This paper describes a novel technique that has been successfully adapted to coat the sidewalls of intricate LIGA MEMS parts with commercial coatings, e.g. diamond like nanocomposite, DLN. Coating uniformity on the sidewalls has been conformed by cross sectional TEM. Tribological measurements of DLN films on electrodeposited Ni test coupons showed much improved tribologcal behavior with friction coefficient of 0.04 and practically no signs of debris generation or stick-slip behavior.

4:30 PM

Surface Engineering with Dendrimer Nanocomposites: *Lajos Balogh*[1]; [1]University of Michigan, Ctr. for Biologic Nanotech. & Macromolecular Sci. & Eng., 4010 Kresge Res. Bldg. II, 200 Zina Pitcher Place, Ann Arbor, MI 48109-0533 USA

Dendrimer based nanocomposites (DNC-s) are recently developed materials that are composed of nanoscopic guest domains dispersed in polymer hosts with no covalent bonds between host and guest domains. These inorganic-organic hybrid materials are uniform, and can be made in controlled sizes. They often display unique physical and chemical properties as a consequence of the atomic/molecular level dispersion of their components. Solubility and compatibility of these materials are determined by the host polymer molecule; however, these nanocomposites also possess many of the desirable chemical and physical properties of the guest molecule(s) or atom(s). Utility and properties of these novel materials will be illustrated on three examples, such as self-assembled monolayers of DNCs, ultrathin multilayers of dendrimer nanocomposites on different substrates, and surface derivatization of CdSe nanocrystals. Advantages of using dendrimer structures and dendrimer nanocomposites as well as characterization and optical properties of the resulting complex multilayers will be compared and discussed.

4:45 PM

Aqueous Chemical Growth of Advanced Nanostructured Metal Oxides Thin Films: *Lionel Vayssieres*[1]; [1]Uppsala University, Phys. & Physl. Chem., Box 532, Uppsala SE-75121 Sweden

A novel concept and thin film growth technique have been developed in order to create a new generation of smart and functional thin film materials, modeled, designed and engineered to match the physical and structural requirements of their applications. This concept, well-sustained by a thermodynamic model, monitoring the nucleation, growth and ageing processes through the chemical and electrostatic control of the interfacial free energy allows to monitor the particle size, the surface morphology and the orientation as well as the ability to thermodynamically stabilized metastable crystal phases. The template- and surfactant-free aqueous chemical growth synthesis allows to generate, at large scale and low-cost, novel designed and well-ordered metal oxide nano- to microparticulate multilayered thin film materials with a complex architecture such as 3D highly oriented crystalline arrays of metal oxides such as nanorods, microrods and microtubes as well as nanowires and nanocomposites materials grown on various substrates at low temperature.

5:00 PM

Magnetron Sputtering of Nanocomposite (Ti, Cr)CNX Coating for Precision Engineering Applications: *Sam Zhang*[1]; Yongqing Fu[1]; Hejun Du[1]; Yuchan Liu[2]; [1]Nanyang Technological University, Sch. of MPE, 50 Nanyang Ave. 639798 Singapore; [2]Gintic Institute of Manufacturing Technology, 71 Nanyang Dr. 638075 Singapore

Nanocomposite coatings were prepared by co-sputtering of titanium, graphite and chromium targets with a gas mixture of argon and nitrogen. Surface morphology of the coating was examined using AFM, and the average surface roughness was only 4-6 nm. Substrate bias voltage and titanium target power rendered significant influence on coating surface morphology. Superb film hardness with adequate toughness were measured by a nano-indenter. Coating hardness increased significantly with the increase in Ti content, and decrease in nitrogen gas ratio. Increase of substrate bias power and Ti target power brought about significant increase in adhesion strength. A ball-on-disk tribometer was employed to study the wear behavior. Results indicated that the film had low-friction coefficient and was wear-resistant. Residual stress and coating stability under a high temperature were also studied. The nanocomposite coating was shown to be able to withstand a high temperature up to 750°C.

5:15 PM

Tunable Self-Assembly of Carbon Nanotubes on Silica Surface: *Zhengjun Zhang*[1]; [1]Tsinghua University, Matls. Sci. & Eng., Qinghua Yuan, Beijing 100084 China

We report here tunable self-assembly behavior of carbon nanotubes on planar silica substrates by a catalytic CVD process. Different from other CVD approaches, the catalyst and hydrocarbon are introduced simultaneously in this process into the deposition chamber, via the vapor phase. Thus the growth of nanotubes can be tuned by adjusting the ratio of catalyst/hydrocarbon, and the deposition time. Depending on the ratio catalyst/hydrocarbon, nanotubes can organize themselves into crystal-like, spherulite-like and honeycomb-like growth units at early growth stages, based on which they develop into films of nanotubes of different morphologies at prolonged deposition. Our study provides the first glimpse of the early stages of nanotubes growth and possible pathways by which nanotunes assemble and grow. From these results an easy way to fabricate architectures of aligned carbon nanotubes, namely substrate-site selective growth, was developed.

5:30 PM

Reinforced Epoxies using Carbon Nanotubes: *Richard A. Bley*[1]; [1]Eltron Research, Inc., 4600 Nautilus Ct. S., Boulder, CO 80301-3241 USA

We are developing a method for incorporating Single Walled Carbon Nanotubes (SWNT) into epoxy thermosets. The tendency of the polymer backbone in poly (m-phenylenevinylene-co-2, 5-dioctoxy-p-phenylenevinylene), (PmPV) to adopt a helical configuration acts to promote the winding of this polymer around both individual SWNT's and multiple SWNT ropes. The interaction between the PmPV and SWNTs is purely mechanical so there is no incursion into the bond structure of the SWNTs. Attaching various functional groups to the side chains of the polymer will make covalent bonding possible between the composite reinforcement (the PmPV/SWNT) and the composite's epoxy matrix. This will provide optimal load transfer properties in the final composite. To what extent this final composite displays the extraordinary strength and durability of the carbon nanotubes will depend on the PmPV derivative's ability to transfer the applied load from the composite's epoxy matrix to the SWNTs.

Teaching and Learning Hydrometallurgical Science and Engineering: Process Modeling

Sponsored by: Extraction & Processing Division, Aqueous Processing Committee, Copper, Nickel, Cobalt Committee, Precious Metals Committee, Waste Treatment & Minimization Committee
Program Organizers: Kwadwo Osseo-Asare, Pennsylvania State University, Metals Science and Engineering, University Park, PA 16802-5006 USA; Saskia Duyvesteyn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112-0114 USA

Tuesday PM Room: 601
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: Renato G. Bautista, University of Nevada-Reno, Metlgcl. & Matls. Eng., MS 388, Reno, NV 89557-0140 USA; David B. Dreisinger, University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

2:00 PM Introductory Remarks

2:05 PM Keynote
Hydrometallurgical Process Modeling for Design and Analysis, Part I: Mass and Heat Balances: *David B. Dreisinger*[1]; David G. Dixon[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Mass and heat balance modeling is a powerful tool for the design and analysis of hydrometallurgical process flowsheets. In this first of two papers, we examine the basic fundamentals of mass and heat balance modeling, and apply these concepts to several flowsheets using a simple spreadsheet model. In this way, we aim to demonstrate how such models are not only easy to construct, but can also be indispensible for comparing various flowsheet alternatives, for making rational economic decisions, and for developing a basic understanding of existing process behaviour.

2:40 PM Invited
Hydrometallurgical Process Modeling for Design and Analysis, Part II: Leaching Kinetics and Associated Phenomena: *David G. Dixon*[1]; David B. Dreisinger[1]; [1]University of British Columbia, Dept. of Metals & Matls. Eng., 309-6350 Stores Rd., Vancouver, BC V6T 1Z4 Canada

Although the kinetics of leaching processes are of paramount importance in the design and analysis of nearly all hydrometallurgical flowsheets, these concepts remain poorly understood and, as a result, are only rarely applied to mass and heat balance models. In this second of two papers, we demonstrate how simple yet powerful models of leaching kinetics and other rate processes may be added to mass and heat balance models, and apply these concepts to several flowsheets using a simple spreadsheet model. In particular, we focus on the interpretation and scale-up of batch leaching data, and on simple means for modeling other phenomena commonly associated with leaching, such as reagent depletion, gas-liquid mixing, and the precipitation of oxides and basic salts.

3:05 PM Invited
Shrinking Core Models in Hydrometallurgy: *KNona Liddell*[1]; [1]Washington State University, Chem. Eng. Dept., PO Box 642710, Pullman, WA 99164-2710 USA

Shrinking core models can be derived in at least two ways. The pseudo-steady state derivations presented by Levenspiel are considerably more familiar to hydrometallurgists, but the same model equations were obtained over 30 years ago from very general mass transport considerations. The latter approach provides additional insight into the validity of shrinking core models. Aspects of the two derivations are contrasted.

3:30 PM Invited
Using Avecia's Minchem Copper Solvent Extraction Process Modeling Program for Education: *Keith Cramer*[1]; James Morrison[1]; Tony Moore[2]; Sandra Kentish[3]; [1]Avecia, Metal Extraction Products, 3259 E. Harbour Dr., Ste. 100, Phoenix, AZ 85034 USA; [2]Avecia, Metal Extraction Products, 15 Sammut St., PO Box 6187, Wetherill Park, NSW 2164 Australia; [3]University of Melbourne, Dept. of Cheml. Eng., Melbourne, Victoria 3010 Australia

As a leader in the development of metal extraction technology, Avecia continues to pioneer advancements for the processing of copper through leach, solvent extraction, and electrowinning of copper. From the initial patent of the second generation of oxime 5-nonylsalicylaldoxime in the 1970's; to the effective use of modifier technology; and development of computer assisted modeling programs, Avecia is driving SX technology to maximize extraction productivity. Keys to consider when advancing productivity are: 1) high quality, high performance products; 2) a responsive, partnering approach to technical service; and 3) tools to aid in the education of industry wide technical personnel with a view on continuous improvement. The focus of this paper is on the advancement of tools to aid in the education for optimization of processing copper through solvent extraction. One such tool is Avecia's own customized solvent extraction modeling program called MEUM. MEUM is currently used world wide to analyze plant operations, test ideas for improving operations, assist engineering companies in design, and to teach basic solvent extraction concepts. Recently MEUM has also been incorporated into a University setting. In the spring of 2000 Avecia worked with the University of Melbourne to integrate MEUM into their Mass Transport tutorial class. Students completed exercises to evaluate how operational changes affected a solvent extraction plant's performance. This paper introduces the basic functions of the program and discusses the successful implementation of MEUM into the University of Melbourne curriculum.

3:55 PM Break

4:10 PM Invited
Elaboration of Industrial Material and Energy Balances as a Didactic Aid: *Gretchen T. Lapidus*[1]; [1]Universidad Autónoma Metropolitana, Dept. Ing. de Procesos e Hidraúlica, Av. San Rafael Atlixco 186, Col. Vicentina, DF 09340 México

With today's powerful process simulators, there is no apparent need for students to elaborate detailed material and heat balances for specific plant flow sheets. However, many academic and practical benefits result from this exercise. Firstly, it allows the students to visualize the workings of the entire plant and to grasp the importance of the variables of each unit operation on the overall performance of the proposed scheme. When the balances are programmed in a PC worksheet (eg. EXCEL), the detailed contents of each flow stream are calculated, allowing the students to better understand the interactions between equipment. Industry could employ the "products" of this activity as training tools and in the evaluation of water balances and for the detection of possible impurity build-ups. Two examples of plant balances are presented: galena leaching with ferric chloride brines and precious metals leaching with ammonium thiosulfate.

4:35 PM Invited
Incorporating Multistage Unit Operations into a Hydrometallurgy Course: *Renato G. Bautista*[1]; [1]University of Nevada-Reno, Metlgcl. & Matls. Eng., MS 388, Reno, NV 89557-0140 USA

The traditional hydrometallurgy course includes an extensive discussion of the chemical aspects of the separation process. The discussion on the engineering of the process is usually kept to a single stage operation. The discussion on the required unit operations can be approached in terms of an equilibrium stage (material balance) or in terms of rate processes (mass transfer). The equilibrium stage contacting involves two separate incoming streams that interact to attain equilibrium as they leave each stage, producing two product streams in equilibrium. The rate process includes operations in which a component of a phase diffuses or is transferred under the influence of a driving force. In many industrial hydrometallurgical processes, multistage operations are usually necessary. The development of the appropriate equations for selected multistage operations is illustrated in this paper.

5:00 PM Invited
Process Modelling and Simulation for Teaching Industrial Hydrometallurgy: *Michael W. Wadsley*[1]; [1]Austherm Pty, Ltd., PO Box 2049, N. Brighton, Victoria 3186 Australia

The hydrometallurgical industry needs people who have an understanding of aqueous solutions in leaching, purification and product recovery. However, aqueous solutions are different from the solutions encountered in petroleum and petrochemical engineering and in pyro- and physical metallurgy with respect both to their molecular structure and to how they are modelled. Hence hydrometallurgists require specialist training. There is anecdotal evidence that pre-university education does not always provide the foundation required for teaching hydrometallurgy. The university system has to provide management, communication and safety as well as technical skills. Part of the student body sees technical education leading to management rather than to a lifetime practice of specialist technical skills. Those planning courses cannot assume that all students will have the same basic skills on entry or will have similar expectations on exit. Instruction in the

practice and management of computer modelling and simulation may be used to teach hydrometallurgical principles. Process modelling exposes students to descriptions of aqueous solutions over various concentration ranges. In learning to manage process simulation, students are exposed to verification and validation techniques. Courses may be presented at either senior undergraduate or post-graduate level. Post-graduate degree and diploma courses may be a practical method to provide appropriate technical skills in industry.

Third International Sulfide Smelting Symposium - "Sulfide Smelting '02": Operational Improvements and Sulfide Smelting Fundamentals I

Sponsored by: Extraction & Processing Division, Pyrometallurgy Committee, Copper, Nickel, Cobalt Committee, Lead and Zinc Committee, Non-Ferrous Metals Committee
Program Organizers: Robert L. Stephens, TeckCominco Metals, Ltd., Trail, British Columbia V1R 4L8 Canada; Hong Yong Sohn, University of Utah, Department of Metallurgical Engineering, Salt Lake City, UT 84112 USA

Tuesday PM — Room: 607
February 19, 2002 — Location: Washington State Conv. & Trade Center

Session Chairs: Frank R.A. Jorgensen, CSIRO Minerals, PO Box 312, Bayview Ave., Clayton South, Victoria 3169 Australia; James C. Daley, Daley and Associates, 1020 W. Cactus Wren Dr., Pheonix, AZ 85021 USA

2:00 PM
Accretion and Dust Formation in Copper Smelting–Thermodynamic Considerations: Douglas R. Swinbourne[1]; *Eva Simak*[1]; Akira Yazawa[2]; [1]RMIT University, Dept. of Chem. & Metall. Eng., GPO Box 2476 V, Melbourne, VIC 3001 Australia; [2]Tohoku University, 16-32 Niizaka, Sendai 981-0934 Japan

Accretions of dust on the walls of the offtakes from furnaces and in the waste heat boiler are a cause of significant operating and maintenance problems in non-ferrous smelters. The present paper discusses the formation of accretions and the condensation of dust in copper smelting from a thermodynamic viewpoint. Changes in gas composition and the amounts and identities of the species in the dust are calculated using computational thermodynamics. The smelting at 1300°C of a typical copper concentrate to produce a 65% Cu matte is first modelled to generate the equilibrium composition of the offgases. The cooling of this gas, together with entrained matte and slag, is then modelled. The results show that the nature of the dust varies greatly, depending on temperature and amount of oxygen present. Control of the amount of oxygen is required if offtake and waste heat boiler accretion problems are to be minimised.

2:25 PM
Sulphation of Cuprous Oxide in SO2-Rich Atmospheres: *Tiina Ranki-Kilpinen*[1]; Esa J. Peuraniemi[1]; Mika Mäkinen[1]; [1]Helsinki University of Technology, Lab. of Matls. Procg. & Powder Metall., PO Box 6200, HUT, Espoo FIN-02015 Finland

In the flash smelting technique, developed by Outokumpu company, off-gases with dust load are directed into the heat recovery boiler (HRB). As temperature decreases metallic sulphates become thermodynamically stable. Sulphation of oxidic particles initiates. Releasing heat causes an increase in particle temperatures, and soft sulphated particles can stick to the heat transfer surfaces. Dust accretion may cause operational problems. Behaviour of synthetic Cu2O particles (37-53 µm) was studied using a fluid-bed reactor of laboratory scale. Experimental parameters in the latest campaign were oxygen and sulphur dioxide contents (2.5-10 vol-% O2, 40-60 vol-% SO2) and temperature (600-660°C). Samples were examined microscopically (light optical microscope, SEM+EDS) and chemical analysis were conducted. Objectives of the project are to study sulphation reaction mechanisms and kinetics of flue-dust particles in the HRB conditions, and get more information for the flue-dust treatment possibilities. Sulphation was found to be mainly dependent on the temperature and oxygen content.

2:50 PM
Dissolution of Particles in the Mitsubishi Smelting Furnace: *Toru Taniguchi*[1]; Nozomu Hasegawa[2]; Osamu Iida[1]; [1]Mitsubishi Materials Corporation, Central Rsrch. Inst., 1-297 Kitabukuro-cho, Saitama 330-8508 Japan; [2]Mitsubishi Materials Corporation, Naoshima Smelter & Refinery, 4049-1 Naoshima-cho, Kagawa-gun, Kagawa 761-3110 Japan

In the Mitsubishi smelting furnace, copper concentrates, silica sands and water granulated slag returned from the converting furnace are injected through process lances together with oxygen-enriched air into the melt. Because of the bath smelting, the rates of particles/melt and gas/melt reactions are high enough and therefore fine grinding of the solid materials is not necessary. Among these reactions, the dissolution rate of silica sands is the slowest and limits the overall reactions in the smelting furnace. The information of dissolution rate of silica sands plays an important role for design of a new furnace. The theoretical calculation on the dissolution rate of silica particles has been reported elsewhere. The authors conducted cold model experiments and could verify the calculation. In this paper, the dependence of dissolution rates of particles on the gas blowing rate, particle size and solid penetration depth in bath is discussed.

3:15 PM
Operational and Maintenance Experience from the First Full Scale Kumera Steam Dryer: *Olli Tiitu*[1]; R. Puurunen[1]; [1]Kumera Corporation, Tech. Ctr., Kumerankatu 2, Riihimaki FIN-11100 Finland

Drying is a unit process that has numerous applications in metallurgical processing. Kumera Technology Center is a worldwide equipment and process supplier, and has expertize in both direct and indirect drying. In conventional direct-heated drying, the required energy is supplied by combustion of fossil fules emitting considerable amounts of off-gases. However, the thermal energy can be readily available in some metallurgical plants, such as modern copper smelters, when steam is produced by waste heat boilers of flash smelting furnaces. The total volume of off-gases, and especially the amount of harmful gases, released into the atmosphere can be significantly reduced when using steam as the indirect energy source for drying. Kumera Technology Center has developed and promoted a new type of steam dryer for drying of, for example, abrasive copper concentrate and silica sand. In the development of the new dryer, Kumera's approach has been to combine the best properties of both the existing steam dryers and the rotating drum dryers. In the design, special attention has been paid to overcome the reported problems of excessive wear and clogging of steam tubes and to reduce the required maintenance operations. The Kumera Steam Dryer, having a patented flexible steam tube arrangement inside the rotating shell, gives a high effective heating area to drum volume ratio without build up and clogging and also gives the possibility of building a concentrate dryer with a bigger capacity so that all feed drying could be accomplished in a single unit. In this paper, the operational and maintenance experiences from two years operation of the full scale Kumera Steam Dryer at Norddeutsche Affinerie AG, in Hamburg, Germany, will be given. Also, an overview on the further development of construction and the biggest feasible unit size in response to future demand is discussed.

3:40 PM Break

3:55 PM
Optimization of the Energy Cost at Tamano Smelter: K. Noda[1]; S. Tanaka[1]; M. Hamamoto[1]; *Kimihiro Shimokawa*[1]; [1]Hibi Kyodo Smelting Co., Ltd., Tamano Smelter, No. 6-1-1 Hibi, Okayama Pref. 706, Tamano City Japan

Tamano Smelter of Hibi Kyodo Smelting Co., Ltd. is carrying out a low cost production using a Tamano Type Flash Smelting Furnace (T-FSF), which is an improved version of Outokumpu Type Flash Smelting Furnace but without slag cleaning furnace and also by developing its own coke combustion technology. Coke combustion technology, which is the basis of the system, has been improved several times and at present has evolved to a stage where it can be reproduced on the Computer Guide System for Coke Combustion in the T-FSF (CCCT) using a general purpose computer. This CCCT includes a trend control and a prediction function and an operator can freely control the operating parameters so as to control the operation flexibly. We have recently developed an overall energy reduction system covering T-FSF to Sulfuric Acid Plant, which we call an Optimum Low Cost Operation System (OLCO) to reconsider the energy costs including coke, heavy fuel oil, and electric power. In this report, we will summarize the recent operation energy operation utilizing OLCO system.

4:20 PM
Development of the Toyo FSF Concentrate Burner by using Computational Fluid Dynamics: Yasumasa Hattori[1]; Katsuhiko Nagai[1]; Shuuji Endou[1]; Keisuke Yamamoto[1]; Yasuo Ojima[1]; [1]Sumitomo Metal Mining Co., Ltd., Toyo Smelter & Refinery, Otu 145-1, Hunaya, Saijo, Ehime 793-0005 Japan

Fluid flow in a FSF concentrate burner was investigated quantitatively in order to improve burner performance. Computational fluid dynamics was utilized for researching fluid flow in a burner port mathematically then its results were reviewed in the commercial FSF burner and its calculation results were found to be in good agreement with the actual measurement results. Based on those results, a mathematical fluid flow model for the Sumitomo FSF concentrate burner has been developed and successfully utilized for improving pre-mixing efficiency in the burner port so as to hasten the combustion speed of concentrate in the reaction shaft.

4:45 PM Cancelled
Modification and Production Practice for Smelting Flash Furnace in Guixi Smelter of Jiangxi Copper Corporation: *Long Ziping*

Water Vapor Effects on Oxidation of High-Temperature Materials: Ceramics and Ceramic Composites

Sponsored by: ASM International: Materials Science Critical Technology Sector, Structural Materials Division, Corrosion and Environmental Effects Committee
Program Organizers: Peter F. Tortorelli, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6156 USA; Karren L. More, Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6064 USA; Elizabeth J. Opila, NASA Glenn Research Center, Cleveland, OH 44135 USA

Tuesday PM Room: 305
February 19, 2002 Location: Washington State Conv. & Trade Center

Session Chairs: K. L. More, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA; E. J. Opila, NASA Glenn Research Center, Cleveland, OH 44135 USA

2:00 PM
Additive Effects on Si_3N_4 Oxidation/Volatilization in Combustion Environments: *E. J. Opila*[1]; D. S. Fox[1]; R. C. Robinson[1]; R. A. Wenglarz[2]; [1]NASA Glenn Research Center, MS 106-1, 21000 Brookpark Rd., Cleveland, OH 44135 USA; [2]South Carolina Institute for Energy Studies, 386-2 College Ave., Clemson, SC 29634 USA

During oxidation, sintering additives in AS800 Si_3N_4 result in the formation of a lanthanum silicate phase on the scale surface in addition to silica. Increased recession rates of Si_3N_4 have been observed in combustion environments due to silica scale volatility in water vapor. Increasing enrichment of the oxide scale in lanthanum silicates is shown to occur as the silica volatility increases. This is demonstrated in a series of tests at 1300°C in an oxygen-containing furnace, a water vapor-containing furnace and a high pressure burner rig. At the very high silica volatility rate found in a turbine, the lanthanum silicate is reduced to pure lanthanum. This study examines the variations of the oxide scale morphology formed on AS800 as silica volatility increases. In addition, the potential for the silicate-enriched scales to slow volatilization is discussed. To date, lanthanum silicate enrichment has not shown any effect on recession rates of AS800 Si_3N_4 as compared to pure silica scales formed on CVD Si_3N_4.

2:30 PM
Synergistic Effects of Water Vapor and Alkali Chloride Vapors on the High Temperature Corrosion of Si-Based Ceramics: *M. J. McNallan*[1]; Y. S. Park[1]; P. P. Hsu[1]; S. Y. Lee[1]; [1]University of Illinois at Chicago, CME Dept., M/C 246, 842 W. Taylor St., Chicago, IL 60607 USA

Alkali chloride vapor contaminants such as NaCl and KCl in air accelerate the oxidation of SiC and other silicon-based ceramics by reacting with the usually protective silica scale to form alkali silicate species. The alkali silicates provide less resistance to oxygen transport and can be liquid at temperatures as low as 900°C. In the presence of water vapor, the chlorine can be removed from the reaction as the stable HCl species so that the effective activity of alkali oxide in the Na_2O-SiO_2 or K_2O-SiO_2 melt in equilibrium with a given vapor pressure of alkali chlorides is increased. This results in higher corrosion rates as determined by weight change and microstructural evaluation.

3:00 PM
Effects of High Water Vapor Pressures on the Oxidation of Si-Based Materials at High Temperatures: *P. F. Tortorelli*[1]; K. L. More[1]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831-6156 USA

The oxidation of pure silicon and different silicon carbides at high temperature (1200°C) and elevated total water vapor pressures (up to 1.5 atm) was studied using measurements of the amount of recession and scanning and transmission electron microscopy of reaction products. At sufficiently high water-vapor pressures, the oxidation of SiC was ultimately controlled by the conversion of dense amorphous SiO_2 to porous cristobalite and could be described by a classic paralinear kinetic model in which a compact oxide is formed underneath a defective, nonprotective product layer.

3:30 PM
The High-Temperature Stability of SiC-Based Composites in High-Water-Vapor-Pressure Environments: *K. L. More*[1]; P. F. Tortorelli[1]; L. R. Walker[1]; N. Miriyala[2]; J. R. Price[2]; M. van Roode[2]; H. Eaton[3]; E. Y. Sun[3]; G. D. Linsey[3]; [1]Oak Ridge National Laboratory, Metals & Cer. Div., PO Box 2008, Oak Ridge, TN 37831-6064 USA; [2]Solar Turbines, Inc., San Diego, CA USA; [3]United Technologies Research Center, E. Hartford, CT USA

A high-temperature, high-pressure, tube furnace has been used to evaluate the long-term stability of SiC/SiC composite materials at high water-vapor pressures. Composites were exposed in this rig so as to determine their viability as components for combustion applications (in this particular case, combustor liners in a Solar Turbines engine). Exposures were conducted to determine surface recession rates for different SiC/SiC composites at 1200°C and to understand controlling degradation mechanisms at elevated H2O pressures (up to 1.5 atm). Similar composite materials (manufactured as combustor liners with and without protective coatings) were also exposed in at least five different engine tests for times up >13,000 h. Results from both laboratory- and recent field-tests will be compared and discussed.

4:00 PM
Effect of Moisture Exposure on the Fatigue Behavior of Several Ceramic Matrix Composites: *L. Zawada*[1]; S. Steel[1]; J. Staehler[2]; [1]Air Force Research Laboratory, Matls. & Mfg. Direct., Wright-Patterson AFB, OH 45433-7817 USA; [2]1Systran Corporation, Dayton, OH USA

The fatigue behavior of several ceramic matrix composites (CMC) was evaluated at temperatures ranging from 1000°C to 1200°C to establish fatigue diagrams of stress versus cycles to failure for each CMC system. Additional fatigue specimens were cycled for set blocks of cycles, removed from the machine, and exposed to water fog for 24 hours. For several experiments the water contained 0.05 weight percent sodium chloride. After exposure, the specimens were dried and then placed back into the fatigue machine for the next block of fatigue cycles. The results of these fatigue plus water fog tests were compared to the original fatigue experiments. Those CMCs manufactured with boron nitride as the interphase between the fiber and matrix experience decreased lives, while those CMCs without a boron nitride interphase showed no loss in fatigue performance or retained strength after fatigue loading. These results will be used to rationalize the degradation observed on aerospace turbine engine exhaust nozzle CMC hardware flown on a F-16 aircraft. Examples of the degradation observed on the flight hardware will be presented, and related to the observed lifetimes.

4:30 PM
High Temperature Air and Water Vapor Interaction of a Nextel-720/Alumina Ceramic Matrix Composites (CMC): A Combined Spectroscopic and Thermodynamic Study: *S. Seal*[1]; S. Wannaparhun[2]; V. Desai[1]; [1]University of Central Florida, AMPAC, 12443 Research Pkwy., Ste. 404, Orlando, FL 32826 USA; [2]University of Florida, Matls. Sci. & Eng., 1001 S.W. 16th Ave., Apt.# V46, Gainesville, FL 32601 USA

The water vapor interaction with CMCs is found to be a critical issue in high temperature oxidation behavior of most CMCs. A Nextel-720 fiber/alumina CMC designed for land-based gas turbine engines was tested in a high temperature rig equipped with water vapor flow. X-ray photoelectron spectroscopy (XPS) and X-ray powder diffraction (XRD) indicated that mullite phase in the Nextel-720 fiber decomposed into SiO(g) and Al_2O_3(s) due to the low oxygen partial pressure. Formation of volatile silicon hydroxide species was responsible for degradation of the Nextel-720 fiber. Presence of an aluminosilicate layer on the composite surface is believed to delay the water vapor interaction. This was also confirmed through TEM images of Keiser Rig (Oak Ridge National Laboratory)-tested samples.